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Book of Abstracts

ITM Istituto per la Tecnologia delle Membrane
Consiglio Nazionale delle Ricerche

EMS 1982

ALMA MATER STUDIORUM
UNIVERSITÀ DI BOLOGNA

Plenary Lectures

Keynote/Oral presentations

Day	November 21, 2022 (Monday)
Morning/Afternoon	Morning
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Day	November 21, 2022 (Monday)
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18:00- 18:20	Development of nanofiltration membranes incorporating artificial water channel Samhari Omar	341
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09:10-09:20	Benzimidazole-dichlorimidazole zeolitic imidazolate frameworks based on ZIF-7 and their application in mixed matrix membranes for CO_2/N_2 separation Lasseuguette Elsa	346
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09:30-09:40	Dendritic polymer functionalized carbon nanodiscs as additives for water treatment membranes Tournis Ioannis	348
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09:50-10:00	Development of electrospun G-C3N4 NT/CDs polyacrylonitrile (PAN) nanocomposite membrane for improved microfiltration performance Oğuz Orhun Teber	350
10:00-10:10	Removal of heavy metals from surface water using chitosan/ZIF-8 mixed-matrix membranes Vercus Lumami Kapepula	351
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09:10-09:20	Forward osmosis-membrane distillation hybrid technology: opportunities, challenges, and benchmarking Suleman Muhammad	355
09:20-09:30	Gas separation polymeric membranes for xenon recovery Zhmakin Vyacheslav	356
09:30-09:40	Controlling L-serine antisolvent crystallization using hydrophobic polymeric membranes Chergaoui Sara	357

09:40- 09:50	Investigation of innovative hybrid membrane bioreactor (If-Mbr) treatment technology for domestic wastewater Turken Turker	358
09:50- 10:00	Novel trends in membrane contactor processes: Behavioral and structural studies of therapeutic activity of micelles of emerging green solvents Mondal Suchintan	359
10:00- 10:10	Pilot-scale synthesis of novel membrane chemistries: case studies and lessons learned Davenport Douglas M.	360
10:10- 10:20	Potential reuse of old RO membrane as NF membrane by surface regeneration Madduri Srija	361

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Session **66**	Modelling, Simulation, Artificial Intelligence in Membranes

	Keynote lecture	
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	Oral Synopsis Presentations	
09:00- 09:10	Zeolite membranes for gas separation: possible reconciliation between experiments, modelling, and molecular simulation? Kuznetsova Margarita	363
09:10- 09:20	Intermolecular interaction estimation in polymer solutions Kim Dooli	364
09:20- 09:30	Flux estimation of dynamic ultrafiltration for wastewater treatment using combinations of machine learning methods Prado Rubio Oscar Andrés	365
09:30- 09:40	Performance optimisation of curly hollow membrane modules using CFD Horvat Tomislav	366
09:40- 09:50	Polyphenols - polyethersulfone interactions: impact of ions on adsorption on ultrafiltration polymer membranes Certiat Marie	367
09:50- 10:00	Towards the generation of machine learned potentials for coarse grained molecular simulations of polymer membranes for gas separations Ricci Eleonora	368
10:00- 10:10	A mechanistic bridge between pulmonary epithelial membrane architecture, inhaled drug dosage and localized disposal, and permeation following dissolution: a fully parameterized predictive approach Deb Tanmoy	369
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Session **67**	Membranes in Water Treatment and Desalination

	Keynote lecture	
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09:10-09:20	Removal of nanoplastics by microfiltration and ultrafiltration membranes Landaburu-Aguirre Junkal	373
09:20-09:30	Pore-filling microfiltration membrane with zwitterionic polymer brush provides ultrahigh flux with an excellent virus removal from water Moshe Herzberg	374
09:30-09:40	Cr(III) recovery and water reclamation from electroplating industry effluents Moutafis Ipek	375
09:40-09:50	TEMPO-oxidized high surface charge cellulosic membranes for removal of charged compounds from water Nieminen Joona	376
09:50-10:00	Combined application of coagulation/flocculation and membrane filtration technology for the treatment of soap factory wastewater Ghofrane Louhichi	377
10:00-10:10	Role of the membrane's properties on bubble formation, mass transfer coefficients and efficiencies during the aeration process Rizzardi Ilaria	378
10:10-10:20	Implementation of Quorum Quenching to limit bacterial communication in membrane bioreactors Mideksa Ermias	379

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Session **68**	Polymer, Biopolymer Membranes

	Keynote lecture	
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	Oral Synopsis Presentations	
09:00-09:10	Preparation of natural polyelectrolyte complex membranes through sustainable aqueous phase separation Li Lijie	381
09:10-09:20	Preparation of porous PET membranes using recycled polymer in a phase inversion process Breite Daniel	382
09:20-09:30	Polymerizable ionic liquid-based membranes for transport in aqueous media Alcalde Berta	383
09:30-09:40	The use of 1,2-diaminocyclohexane as the new key in solvent-resistant nanofiltration membranes Lenaerts Marie	384
09:40-09:50	Preparation and characterization of chitosan-based membranes for Na-air batteries Casado-Coterillo Clara	385
09:50-10:00	A facile approach to modify cellulose nanocrystal "CNC "with PTMAEMA for the adsorption of perfluoroalkyl substances "PFAS" Gomri Chaimaa	386
10:00-10:10	Progress and challenges on dicarboxymethyl cellulose adsorptive membranes Gago Diana	387
10:10-10:20	Esters of nature-identical engineered polysaccharides as new materials for packaging applications Papchenko Kseniya	388

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Session **69**	Membranes in Biotechnology, Food, Biorefinery, Biosensors

	Keynote lecture	
08:30-09:00	Toward a Catalytic DNA-Membrane Reactor Schaefer Thomas	389
	Oral Synopsis Presentations	
09:00-09:10	Membrane modification methods for high performance enzymatic membrane reactors Su Ziran	390
09:10-09:20	Functionalised food packaging films: greener routes to synthesise and exploit novel agro-based hydrophobic deep eutectic solvents Alke Bhavna	391
09:20-09:30	Long-term stability of cellulose membranes used in the treatment of spent Deep Eutectic Solvent for solvent purification and lignin recovery Vadim Ippolitov	392
09:30-09:40	Fractionation of biologically active compounds extracted from onion skin bio-waste Tylkowski Bartosz	393
09:40-09:50	Osmotic evaporation of red fruit juice and impact on quality and biological properties Conidi Carmela	394
09:50-10:00	Reuse of permeate stream from the dewatering of microalgae biomass using ceramic membrane filtration Malaguti Marco	395
10:00-10:10	Recycling of end-of-life membrane: fouling resistance NF multilayer polyelectrolyte membrane as a new platform in pulp and paper industries Esmaeili Mohammadamin	396
10:10-10:20	Biosensors development by tuning in situ gold nanoparticles formation in PDMS matrix Poerio Teresa	397

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Session **70**	Session: Electromembrane processes (ED, RED, MCDI, Integrated systems)

	Keynote lecture	
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09:00-09:10	Effect of particle size on the desalination performance and flowing properties of activated carbon flow electrodes in capacitive deionization Tauk Myriam	399
09:10-09:20	A new process for resource recovery from surface water RO brines for permeate remineralization Philibert Marc	400
09:20-09:30	Application of bipolar membrane electrodialysis in skim milk acidification Merkel Arthur	401
09:30-09:40	From hollow fibre to flat sheet – How to develop membranes with similar properties and different shapes Bohr Sven Johann	402

09:40-09:50	Application of bipolar membrane electrodialysis for sodium hydroxide production from saltwork brines Tamara León Oviedo	403
09:50-10:00	Anion exchange membranes on block copolymers for use in alkaline electrolysers Žitka Jan	404
10:00-10:10	GREEN BRINE: agri-food brine valorisation through integration of sustainable membrane-based technologies Garcia Jorge	405
10:10-10:20	Development of a polymeric diaphragm for alkaline water electrolysis Fiorentini Diego	406

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Session **71**	Mixed Matrix Membranes

	Oral Presentations	
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11:10-11:30	Semi-interpenetrating poly(ionic liquid) networks: towards the development of mixed matrix iongel membranes for CO_2 separation Nabais Rita	408
11:30-11:50	Microstructured PVDF-based microporous layer for polymer electrolyte fuel cells by phase inversion technique and Cyrene as a green solvent Comite Antonio	409
11:50-12:10	Bactericide effect of silver nanoparticles and silver ion-exchanged zeolites on composite cellulose acetate nanofiltration membranes de Pinho Maria Norberta	410
12:10-12:30	Up-grade mixed matrix hollow fiber membrane fabrication system: accurate production control and data management Etxeberria-Benavides Miren	411
12:30-12:50	Application of Mg(Al)O mixed metal oxides (MMO) nanoparticles in the preparation of sustainable polymeric membranes Jabkhiro Hajar	412

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Session **72**	Integrated Membrane Processes, Hybrid Proc., New trends

	Oral Presentations	
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11:10-11:30	Visible light responsive nanofiltration membranes development to mitigate biofouling in water and wastewater treatment Das Pritam	414
11:30-11:50	An eco-friendly integrated membrane process for the concentration and purification of functional bioactive compounds from citrus hybrid Tacle® extract Conidi Carmela	415
11:50-12:10	Hybrid FO-RO/MD desalination processes: system-scale analysis at the water-energy nexus Morciano Matteo	416

12:10-12:30	Semi-closed reverse osmosis (SCRO): a concise, flexible, and energy-efficient desalination process Mo Zijing	417
12:30-12:50	Positron annihilation lifetime spectroscopy of membranes – probing the free volume with antimatter Dickmann Marcel	418

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Session **73**	Membranes in Biomed and Pharma

	Oral Presentations	
10:50-11:10	Current status and challenges in the artificial lung membrane technology Kim F. Jeong	419
11:10-11:30	Synthesis of silica derivatives and their incorporation in cellulose acetate-based membranes for blood purification Faria Monica	420
11:30-11:50	Improved gas transport of polyurethane-based membranes for blood oxygenation Pires Rita F.	421
11:50-12:10	Biomimetic membrane platform for anti-cancer therapeutic strategies Morelli Sabrina	422
12:10-12:30	Application of 2D material membrane for health care technology Suran Swathi	423
12:30-12:50	Membranes loaded with 18-β-glycyrrhetic acid for skin regeneration, repair and protection Salerno Simona	424

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Session **74**	Membranes in Water Treatment and Desalination

	Oral Presentations	
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11:10-11:30	Advanced wastewater treatment using a novel hybrid anaerobic membrane bioreactor with immobilized biomass foam Baransi-Karkaby Katie	426
11:30-11:50	Gravity-driven membrane bioreactors for decentralized wastewater treatment: uderstanding the impact of fouling control strategies on treatment performance Ranieri Luigi	427
11:50-12:10	Treatment of wastewater effluents using nanofiltration and low pressure UV treatment to produce high quality water that can be reused for irrigation for food production Cristóvão Maria Beatriz	428
12:10-12:30	Treatment of pickling and passivation solutions by diffusion dialysis and membrane distillation at a pilot scale Ruiz-Aguirre Alba	429
12:30-12:50	Natural organic matter membrane fractionation of surface water – a new approach Dejaeger Karlien	430

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Session **75**	Polymer, Biopolymer membranes

	Oral Presentations	
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11:10-11:30	Alkyl levulinates: a new class of green solvents for PVDF membrane preparation Figoli Alberto	432
11:30-11:50	Influence of crosslinking reaction time and crosslinker amount on separation performance and stability of polyvinyl alcohol/pectin membranes Regina Serena	433
11:50-12:10	Cellulose acetate hollow fiber membranes for forward osmosis applications Götz Tobias	434
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Session **76**	Membranes in Gas and Vapor Separation, Hydrogen Production

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11:10-11:30	CO_2 sorption and diffusion in fluorinated polymers for carbon capture and transport application Signorini Virginia	438
11:30-11:50	Simultaneous GHG removal from wet CO_2 streams using novel composite membranes Janakiram Saravanan	439
11:50-12:10	Mixed gas diffusion and permeation of ternary and quaternary gas mixtures in glassy membranes for CO_2/CO separation in CO_2 utilization routes De Angelis Maria Grazia	440
12:10-12:30	Bioinspired ionic liquid-graphene-based smart membranes with electrical tunable channels for gas separation Widakdo Januar	441
12:30-12:50	Asymmetric polybenzimidazole membranes with high performance and durability for anion-exchange membrane water electrolysis Lee Ju Yeon	442

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Session **77**	Nanostructured Membranes

	Oral Presentations	
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11:10-11:30	Novel CO_2 membranes fabrication- an integrated material approach Sandru Marius	444
11:30-11:50	Anti-scaling thin-film composite membranes decorated with pseudo-bottle-brush polyzwitterions for high-recovery desalination of brackish water Bernstein Roy	445
11:50-12:10	Nanostructured pervaporation membranes exclusively made of amphiphilic diblock copolymers: polystyrene-block-poly(vinyl alcohol) Angelini Alessandro	446
12:10-12:30	Development of Ti2AlN MAX phase/cellulose acetate nanocomposite membrane for removal of dye, protein and lead ions Vahid Vatanpour	447
12:30-12:50	Synergistic integration of photocatalytic and thermocatalytic nanoparticles with graphene oxide in nanofiltration membranes for water depollution Boffa Vittorio	448

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Session **78**	Mixed Matrix Membranes

	Keynote Lecture	
14:00-14:30	Mixed Matrix Membranes for target ion separation and resource recovery Yang Xing	449
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14:30-14:40	Oily wastewater treatment using titanium carbide composite membrane Isam Aljundi	450
14:40-14:50	MOF-based mixed matrix membranes for olefin/paraffin separation Hajivand Pegah	451

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Session **79**	Integrated Membrane Processes, Hybrid proc., New trends

	Keynote Lecture	
14:00-14:30	Operation and scale-up conditions optimization of ultra-permeable block copolymer-coated hollow fibers for air dehydration Gebreyohannes Abaynesh Y.	452
	Oral Synopsis Presentations	
14:30-14:40	Integrated membrane distillation and solvent extraction for water, sulfuric acid, and copper recovery from gold mining wastewater Moreira Victor	453
14:40-14:50	Optimization of design and operation for FO-MD-MCr hybrid system in desalination Choi June-Seok	454

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Session **80**	Membranes in Biomed and Pharma

	Keynote Lecture	
14:00-14:30	Multifunctional Membranes for the Extracellular Vesicles Capture: New insights into Regenerative Medicine De Bartolo Loredana	455
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14:30-14:40	Fabrication of composite hollow fiber membrane made of fluorinated polyimide for blood oxygenation Teber Oğuz Orhun	456
14:40-14:50	PLGA membranes with different topographical cues: effect of micropatterns on cell behaviour Piscioneri Antonella	457

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Session **81**	Membranes in Water Treatment and Desalination

	Keynote Lecture	
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	Oral Synopsis Presentations	
14:30-14:40	Solute transport modeling tools for the application of nanofiltration membranes in minerals recovery processes from seawater desalination brines Lopez Julio	459
14:40-14:50	Photothermal air gap membrane distillation for the removal of heavy metal ions from wastewater Alaa Shaheen	460

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Session **82**	Polymer, Biopolymer membranes

	Keynote Lecture	
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14:30-14:40	Stability of biobased polyhydroxyalkanoate (PHA) membranes in cleaning solutions Loulergue Patrick	462
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Session **83**	Membranes in Gas and Vapor Separation, Hydrogen Production

	Keynote Lecture	
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	Oral Synopsis Presentations	
14:30-14:40	A new method to reveal the mechanism of CO inhibition on H_2 flux through ultra-thin Pd/Ag membrane Yu Junbo	465
14:40-14:50	Innovative membrane systems for CO_2 capture and storage at sea (MemCCSea ACT-ERANET project) Asimakopoulou Akrivi	466

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Session **84**	Nanostructured Membranes

	Keynote Lecture	
14:00-14:30	Ultraselective and ultrapermeable tunable nanostructured stainless-steel coated membranes for oil/water emulsion separation Tung Kuo-lun	467
	Oral Synopsis Presentations	
14:30-14:40	PVDF-HFP polypyrrole-decorated nanocomposite fibrous membranes for the electrochemical detection of lactic acid Keirouz Antonios	468
14:40-14:50	Carbon molecular sieve membranes for water separation in CO_2 hydrogenation reactions: effect of the carbonization temperature Poto Serena	469

Poster Presentations **470**

Visualizing Transport Phenomena at Membrane Surfaces

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Extensive research has been carried out for the synthesis of new membrane materials and membrane structures. Understanding transport phenomena inside the membrane has also progressing well over the past decennia. Understanding the physical phenomena at the membrane/fluid interface has been limited so far and is often simply accounted for by estimating a fluid phase mass transfer resistance. Experimental access to and detailed insights into the orgin of such mass transfer resistance is difficult. This presentation shows recent simulation and experimental approaches to analyze two relevant fluid phase resistances

(1) Fouling and filtration physics
(2) Concentration polarization at electrodialysis membranes

For the fouling and filtration physics we have developed a suit of visualization methods to analyse flow dynamics using magnetic resonance imaging (flowMRI) [1] and microfluidics to analyze cake build up and rearrangements [2]. Such experimental data are complemented with Computational Fluid Dynamic – Discrete Element Methods (CFD-DEM) [3] incorporating particle and membrane surface properties. A novel 3D-printing method enables us to print any shape particle with different degrees of softness and study their assembly under flow, i.e. their filtration behaviour [4].

For the challenge of overcoming concentration polarization in electrodialysis, the presentation introduces the electrokinetic phenomenon of electroconvection and presents the electroconvection - based vortex architectures at the membrane interface through particle image velocimetry (PIV). Furthermore, new experiments visualize how such transport enhancing vortices behave under flow or in confinement such as spacers.

The presentation entertains the strategy of integrating sophisticated fabrication, imaging, and simulation methods (Make, Measure, Model) to comprehensively elucidate the origin of mass transport resistances outside the membrane at the membrane/fluid interface.

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Membranes in Space Systems

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Manned space exploration is entering a new exciting age, where mankind will step again beyond Low Earth Orbit, while commercial space stations will start appearing. Our next concrete stop is the Moon orbit, and both the Lunar surface and the first mission to Mars are in the space agencies' plans. Recovery of wastewater as freshwater, liquid-gas phase separation, atmosphere cleaning, as well as energy production are among the most urgent needs in human spacecrafts. The main requirements, particularly in long-duration manned missions, include the minimization of the hardware mass, volume, power consumption and crew time, while maximizing safety and reliability. Some of the associated challenges can be tackled by membrane technologies, some already currently employed onboard the International Space Station. Space environment constraints offer new opportunities for science and technology, leading often to innovative solutions that could be also useful for our daily life. This talk discusses the membrane share in the Space engineering, focusing on water treatment and liquid-gas phase separation, but touching also gas separation, energy production as well as In Situ Resource Utilization (ISRU) applications in extra-terrestrial environments.

Membrane and porous scaffold for bioartificial organs

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For a long period, the major application for membranes was in the field of technology hemodialysis, i.e. renal support system. This is still true with about 3 millions patients worldwide who benefit from 3 dialysis session a week, when transplantation is not possible.

Nowadays, the use membranes or porous scaffolds in health technology is not only dedicated to kidney support and is extended to other organs' support and tissue engineering, thanks to their good biocompatibility and capacity for efficient mass transfer. In this talk, we will review the progress in this field, starting form the pioneer works in the 90's to the current application in bioartificial liver, pancreas, lung, but also in the musculo-skeletal system or in the field of organ on chip for toxicology studies.

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Membrane Emulsification Process: An Overview on its Applications

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My presentation addresses the membrane emulsification technology, a low energy input process which can be applied for the precision manufacture of particulate systems. The main advantage of this technology is the encapsulation of sensitive (bio) active ingredients due to its gentle emulsification process which is carried out under low shear. Some examples include the formulation of bacteria, fungi, enzymes, among others.

Many efforts have been required to develop further these worthwhile technologies for industrial scale production. One of the biggest challenges is the development of novel surface properties of the membranes, needed to control their surface energy and avoid changes in wetting properties over time. Another important aspect concerns the reduction of membrane fouling during processing. Increase in volume production can make these technologies competitive to conventional mechanical emulsification processes, allowing their process intensification and a sustainable production.

If the technical challenges are overcome, membrane emulsification could replace large-scale emulsification processes which require high energy input for the formulation of active molecules.

Rethinking Membrane Process for Food: From particle behaviour to innovative membrane cascades

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When considering sustainable processes for the production of food and food ingredient production, there are a lot of opportunities to do so using membranes! However, the way we develop membrane processes may need to be adjusted so that they are e.g., more flexible to the use of starting materials of different composition, and tailored to specific component behaviour. This will be illustrated using behaviour as observed through microfluidic techniques, and allows amongst others making use of particle migration, and taking flexibility of components into account when designing processes. Away from this, I will show options that are currently underused but would be instrumental in carrying out membrane separation process at reduced energy input through cascaded approach, or the use of e.g., forward osmosis using a current waste stream.

Intensified Membrane Distillation – Concentrated Solar Power/Photovoltaic System for Self-Sustained Inland Desalination

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Management of concentrate streams in inland applications has an uncertain long-term environmental impact. This study investigates the efficiency of thermal energy management within an intensified air-gap membrane distillation (AGMD)-concentrated solar power (CSP)/photovoltaic (PV) collector. The demonstration-scale system has the potential to realize self-sustained zero-waste discharge of concentrate streams in inland and off-grid applications and can produce up to 225 kWh of thermal energy directly supplied to the AGMD system to produce up to 360 L/day. Experiments were performed on the hybrid system to evaluate thermal performance for various operating conditions including MD and CSP flow rates, AGMD operating temperature, and AGMD vacuum pressure. Experimental results indicate that doubling the MD flowrate results in 116% increase in thermal energy utilization and nearly doubles the distillate production. Compared to the winter months, operating the hybrid system in summer months when direct normal irradiance (DNI) is at its peak results in a six-fold increase in average distillate production. Furthermore, the relative specific thermal energy consumption decreases by 30% when allowing the thermal storage reservoir to preheat in the winter. To increase thermal storage capacity, energy utilization, and total distillate production the hybrid system was retrofitted to include an additional thermal storage tank and smart controls to adapt operational configurations for different environmental conditions. Compared to results obtained on the original hybrid system, the updated thermal storage configuration increased MD distillate production by approximately nearly 50% compared to the original operational configuration in the summer. A techno-economic assessment was performed, and compared to conventional thermal desalination processes, the proposed system has lower specific thermal and electric energy demands and can produce high quality water for less than 1.50 \$/m^3. Results from this study highlight important design considerations for integrating thermal desalination with solar energy resources in an operational environment.

Novel Membrane Cascade to Manufacture Skim Milk Concentrate Under Mild Processing Conditions

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Skim milk powder is a frequent ingredient of various food products. The concentration of skim milk to a dry matter (DM) of 50-60% is a prerequisite step during skim milk powder production. The common method for skim milk concentration is evaporation at 55-65°C, which is a thermal energy-consuming process. We developed an evaporation-free three-stage membrane cascade comprising reverse osmosis (RO), nanofiltration (NF), and dynamic ultrafiltration (UF) to produce a concentrated skim milk with a DM of > 45%. Processing temperatures during RO and NF were 10-15°C, which ensures milder processing conditions accompanied by less thermal energy consumption. During the process, NF and UF filtration permeates were recycled back into the cascade, with only RO-permeate leaving the system. Produced skim milk concentrates had a protein dry matter of 33-38%, which was comparable to concentrated skim milk from evaporation. The protein content affected flux and solute retention during filtration. An increase in the UF-permeate recirculation ratio increased the flux during the NF and UF stages while decreasing solute retention during the NF stage. In summary, our RO-NF-UF-membrane cascade is a novel thermal energy saving process to produce skim milk concentrate under mild processing conditions.

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Integration of Ion Exchange and Diffusion Dialysis for the Recovery of Critical Raw Materials from Seawater Saltworks Bitterns

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The European Union is looking for new element and minerals sources, especially for those included in the Critical Raw Materials (CRMs) list. Sea mining has been considered as a new source for obtaining them from seawater. Brines generated after halite crystallization in solar sea saltworks, called bitterns, would be a promising option due to their higher concentration on CRMs compared to seawater (up to 30 times). Then the recovery of certain elements may be economically feasible, such as Mg and Trace Elements (TEs), such as alkaline/alkaline-earth metals (e.g. Li, Rb, Cs, Sr) and transition/post-transition elements (e.g. Co, Ga, Ge, B). Only major components (kg/m^3) as Na, Ca, Mg, K and Br are feasibly mined from seawater and brines. The only economically feasible option to mine TEs (≤mg/m^3) from bitterns is using ion exchange and sorption processes and, to recover these TEs from the sorbents, a desorption stage is needed with the application of 1 M acids (e.g. HCl or H_2SO_4). As the content of acids used in this phase is more than the one needed for regeneration, a subsequent stage is suggested regarding these acids recovery to diminish later alkali consumption and avoid the precipitation of undesired salts. The objective of this work is to evaluate this acid recovery using diffusion dialysis processes with a 8-sheet membrane stack (Fumasep® FAD-PET-75) with 929 cm^2 contact area. Diffusion dialysis tests were carried out with a solution containing HCl and H_2SO_4 acids, B and several metals (e.g. Co, Ga, Ge, Sr), which was circulated counter-current to a demineralized water flow. The effect of pumping both liquids (acid solution and water) at different flow rates (Qacid=Qwater: 0.4-5.0 mL/min) and flow ratios (Qwater/Qacid: 0.2-1.8) showed that maximum acid recovery was achieved for the lowest acid flow rate (>99%) and the highest Qwater/Qacid ratio (77%).

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Supercritical CO_2 Permeation Through Dense Commercial Polymeric Membranes: Permeability Measurements and Influence of Pressure

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Supercritical CO_2 (sc-CO_2) is widely used as an extraction solvent in food industry, especially for the extraction of lipids from seeds. After extraction, products are usually recovered by decompression. Then, recompression for CO_2 recycling is highly energy consuming and this handicaps industrial development when medium to low added-value post-extraction products are considered. Recovery of solutes using membrane technology is an interesting alternative for the regeneration of CO_2 loaded with oil. The suitable membrane must have both high oil selectivity, and should own a high permeability towards sc-CO_2 as reasonable filtration area is needed, while showing a good durability. In this work, we have studied the CO_2 membrane performances (permeability and oil retention) of two different dense polymeric membranes, AG aromatic polyamide reverse osmosis membrane and PuraMemTM silicone nanofiltration membrane. It was considered that the pure sc-CO_2 permeation is governed by the solution-diffusion model which explains the observed permeability changes as a function of CO_2 viscosity and fugacity (i.e. pressure) and the polymer properties as chemical composition and structure. Indeed, the permeation experiments of pure sc-CO_2 showed that the permeability increased with pressure to reach a maximum value at a permeate pressure of 180 bar (17.3±0.9 kg/(h.m^2.bar)), and then decreased in the case of a crosslinked polymer (AG membrane). While, for a slightly crosslinked polymer (PuraMemTM Flux membrane), the permeability was found much higher and it is about 751.6±26.4 kg/(h.m^2.bar) at a permeate pressure of 160 bar. The transmembrane pressure measured using these two membranes do not exceed 10 bar. In the context of solvent recycling, this would lead to a more than 80% energy consumption reduction of the process. The retention rates for sunflower oil measured obtained by these two membranes at a constant permeate pressure of 150 bar, were found to be higher than 85% using the both membranes.

Surgical Masks Durability: Real and Simulated Variation of their Performance Over Time of Usage

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Surgical masks are disposable medical devices that should not be used for more than 4 hours according to the World Health Organization (WHO). Since the early 2020s, due to the COVID-19 outbreak, universal masking has been recommended as an effective non-pharmacological intervention to limit the spread of the virus. The unprecedented demand and the consequent severe shortage of face masks has led to prolonged use and re-use of disposable surgical masks by frontline health care workers. This behavior was even more pronounced in the community. Prolonged use and re-use, after decontamination, has also been recommended by WHO as temporary strategy to address shortages. Several studies have examined potential disinfection techniques while few investigated how the protection offered varies with time of usage and, indeed, if their re-use is recommended. In this work, the maximum durability of two type II surgical masks was evaluated, by regularly measuring the BFE and the differential pressure of specimens worn by 4 operators for an entire work week, 8 hours per day. The results showed that BFE decreased slightly with time of use while differential pressure remained constant. To investigate the cause of the decrease in BFE, some samples were treated under different conditions using a BFE apparatus to simulate the effects on masks performance of the main parameters affecting respiration: humidity of the air, liquid droplets and aerosols, bacteria and airflow. The BFE apparatus was also used to simulate aging during actual use by subjecting samples to constant airflow and measuring BFE and the differential pressure on a regular basis. Different flowrates were investigated and the results were compared with those obtained by real operators. The results showed that the developed protocol allows the durability of surgical masks to be evaluated with a common BFE apparatus, which is now available in several laboratories.

Anion exchanger membranes for fuel cells and water electrolysis

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Anion-exchange membranes (AEMs) have attracted tremendous research interest in alkaline energy devices such as AEM fuel cells (AEMFCs), AEM water electrolyzers (AEMWEs), CO2 reduction reaction (CO2RR), and redox flow batteries due to the feasible utilization of earth-abundant non-noble catalysts. AEM-based energy devices are intended to supersede costly acidic energy systems and provide a low-cost pathway for sustainable energy conversion and utilization. Specifically, AEMs and ionomers are crucial components for alkaline energy devices to conduct anions (typically OH−) and separate fuels between the anode and cathode. Highly ion-conductive, and durable AEMs are an interesting topic in alkaline energy devices. Currently, the ion conductivity and durability of AEMs have been substantially improved in the past few years due to the discovery of efficient aryl ether-free polymers and stable cationic groups, resulting in significant progress in several energy devices. Here, the advances in AEMs and AEM-based energy devices will be presented by understanding their structure-function relationship, covering durable and promising cationic groups, polymer backbones, and AEMs. This will highlight the importance of AEMs and guide future research.

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Ion-Sieving Membranes Based on Polymers of Intrinsic Microporosity for Aqueous Organic Redox Flow Batteries

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Redox flow batteries (RFBs) using aqueous organic-based electrolytes are ideal candidates for developing cost-effective grid-scale energy storage devices [1]. Membrane separators is the key component determining RFB performance, requiring high ionic permeability for charge carrier ions but avoiding cross-over contamination of redox-active molecules. However, it remains challenging to design RFB membrane separators that perform efficient ion and molecular sieving [2]. Here we report the development of new easily-processed ion-sieving membranes based on Polymers of Intrinsic Microporosity (PIMs) and demonstrate their promising performance in aqueous organic RFBs. Our approach design combines 1) the use of PIMs with defining features of rigidity and contorted backbones generates large amounts of microporosity that shows narrow pore size distribution [3]; 2) the introduction of hydrophilic functionalities facilitates the formation of sub-nanometre-sized water channels for ion conduction. The micropore architecture of PIMs can be easily regulated by applying various structural units (i.e., spirocyclic and bridged bicyclic moieties) to produce 2D and 3D polymer topology with different rigidity; the micropore chemistry can be controlled by introducing different types of functionalities (i.e., amidoxime and sulfonate groups) and the degree of functionalization. These PIMs can be solution-processed into robust self-standing membranes, and demonstrate that exquisite control over pore architecture and pore chemistry both play important roles in achieving fast ion transport combined with high molecular selectivity. Two emerging aqueous organic cell chemistries for grid RFBs are applied to uncover the relationship between the membrane properties (stability, selectivity and conductivity) of relevance to cell efficiency and cycle life. Importantly, the new PIM membranes significantly boost battery energy efficiency and peak power density while enable stable operations in benign near neutral-pH electrolytes, promise long-lasting, crossover-free RFBs. The confined ion transport at sub-nanometre scale in PIM membranes suggests their potential utility for a variety of devices for energy generation and storage and water purification.

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Insight into the poly(aryl-co-terphenyl piperidinium) membranes for alkaline membrane fuel cells

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The development of environmental-friendly and sustainable energy has become more urgent due to the deteriorating living environment and the contradictory relationship between the increasing global energy needs and the limited storage of fossil energy. Anion exchange membrane fuel cells (AEMFCs) have attracted increasing attention in the last decade due to their low-cost and high electrode reaction activity under alkaline conditions. However, the development of AEMFCs were hindered by their insufficient durability and power density due to the absence of physically and chemically durable and conductive anion exchange membranes (AEMs) and ionomers. Here, we present a series of ether-free poly(aryl-co-aryl piperidinium)s membranes and ionomers, where the piperidinium group and ether-free backbone endows its excellent stability and conductivity. Specifically, poly(dibenzyl-co-terphenyl piperdinium) (PDTP) and poly(fluorenyl-co-terphenyl piperidinium) (PFTP) based membranes possess high OH^- conductivities of over 160 mS cm^{-1} at 80 °C, durable ex-situ stability in 1 M NaOH solution at 80 °C for over 1600 h, excellent mechanical properties (storage modulus > 1500 MPa, tensile strength > 70 MPa), and limited hydrogen permeability (< 10 Barrer, 1 Barrer = 10^{-10} cm^3 (STP) cm cm^{-2} s^{-1} $cmHg^{-1}$). Moreover, PDTP and poly(fluorenyl-co-biphenyl piperidinium) (PFBP) based ionomers exhibit high water vapor permeability and low phenyl adsorption onto the catalyst surface, which greatly promotes the electrode reaction activity. Based on our rational design of poly(aryl-co-aryl piperidinium) membranes and ionomers, the PDTP-AEM and PFTP-AEM based fuel cells reach outstanding peak power densities of 2.58 and 2.34 W cm^{-2} in H_2/O_2 at 80 °C , respectively. Meanwhile, the PFTP-AEM based fuel cell can be operated stably under a 0.2 A cm^{-2} current density at 70 °C for 200 h with 3.6% voltage loss, suggesting its great application potential in the future.

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Continuous electroreduction of CO_2 for formate in gas-phase at high current densities: comparative study of the influence of the ion exchange membrane

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The electroreduction of carbon dioxide (CO_2) to formate is nowadays considered a promising approach to convert CO_2 into chemicals with value-added, and, simultaneously, store energy from intermittent and renewable sources of energy. However, most of the studies work with a current density lower than the one that is considered to be required for future implementation of the electrochemical process at the industrial scale (i.e. at least 100 mA cm^{-2}, and even 200 mA cm^{-2}) [1]. Besides, the combination of this issue with a continuous operation, and simultaneously, the employ of a CO_2 humidified input stream at the cathode side of the electrochemical reactor, is rarely found in this kind of studies. In this sense, the type of ionic exchange membrane employed to separate cathodic and anodic compartments in the electrochemical reactor can play an important role in the performance of the process. Therefore, this work aims at studying the CO_2 electrocatalytic reduction to formate using a filter press reactor in a continuous mode, with a single pass of the reactants through the cell, employing two different types of ion exchange membranes: (i) Nafion cation exchange membranes [2], and (ii) Sustainion anion exchange membranes [3], using the same experimental setup. Excellent combinations of Faradaic Efficiencies (93 %), energy consumptions (196 $kWh \cdot kmol^{-1}$), and rates (23 $mmol \cdot m^{-2} \cdot s^{-1}$) can be achieved at current densities of 200 $mA \cdot cm^{-2}$ with Sustainion membranes, although at the expense of obtaining a more diluted product than when using Nafion membranes. In this way, under the same operating conditions, Nafion membranes allow reaching formate concentrations of up 312 $g \cdot L^{-1}$, but worsening the value of other key metrics. In spite of achieving promising results, there are still many challenges to be addressed for the implementation of the electrochemical CO_2 conversion to formate process at an industrial scale.

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Micro-structured Ceramic Hollow Fiber Membrane for High-performance Solid Oxide Fuel Cell and Electrolyser

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Europe is dedicated to achieving Carbon Neutrality by 2050, and also COP26 points out the serious environmental issues caused by CH_4 and CO_2. All of them make the research related to energy and environment more stringent and urgent. Solid oxide fuel cell (SOFC) and electrolyser cell (SOEC) has been regarded as the key enabling technologies to mitigate these issues. Many distributed methane sources, either natural or artificial, suffer from low concentration methane (<20%), which are difficult and expensive to separate CH4 from N2 and/or CO_2 and are often flared nowadays. Herein, we designed a multi-channel hollow fiber SOFC to take full use of waste methane to generate electricity and the power can reach >2 W/cm^2. In the meantime, the SOFC is able to suppress coking which is often encoutered in methane-based fuel cell and have very good stability (>500 hours). Besides, the SOEC is also designed based on hollow fiber membrane and is used to split CO_2 (and H_2O) to CO (and H_2) which can be used as the feedstock for Fischer-Tropsch and methanol synthesis. It shows great potential to solve the intermittency issues of renewable electricity by circulating carbon and producing green hydrogen. Beyond the exploration of applications of micro-tubular hollow fiber membrane, structure-performance relation is also revealed by systematic study. Several multi-channel hollow fiber membrane is designed and the effect of morphology on performance is studied.

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Highly Durable N-Heterocyclic Ammonium Polystyrene polyelectrolytes for Next-generation Alkaline Energy Devices

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Aryl ether-free anion exchange membranes (AEMs) and ionomers have recently received much interest for applications in cost-effective anion exchange membrane (AEM) fuel cells (AEMFCs) and AEM water electrolyzers (AEMWEs). Although a few AEMs and ionomers such as ammonium-type polyethylene, ammonium-type polynorbornene (PNB), and poly(aryl piperidinium) (PAP) exhibit promising properties, there is still a lack of durable AEMs and ionomers for long-term AEMFC and AEMWE operations. Moreover, the compatibility between AEMs and ionomers has not been elucidated. Here, we present a series of highly durable dimethyl piperidinium (DMP) and 6-azaspiro[5.5]undecan-6-ium (ASU)-functionalized polystyrene (PS) ionomers for AEMFCs and AEMWEs. Model DMP and ASU cations can endure highly concentrated NaOH/D2O/CD3OD (hydration number = 10) at 80 °C with a half-life time greater than 10,000 hours. Optimized DMP and ASU-type PS polyelectrolytes have alkaline stability over 3,000 h without chemical degradation in 1 M NaOH at 80 °C, which exceeds state-of-the-art anion exchange polyelectrolytes. Moreover, our study shows that DMP or ASU-PS ionomers with flexible polymer backbones are more applicable for flexible PNB-based AEMs, giving a peak power density (PPD) of 1.63 W cm^{-2} at 80 °C without backpressure for a proto-type AEMFC. Rigid PAP ionomers gave a PPD of 1.1 W cm^{-2} under the same conditions. On the other hand, rigid PAP ionomers gave higher power density in rigid PAP-based AEMs, while flexible PS-based ionomers gave limited PPDs under these conditions. This work demonstrates the importance of the compatibility between ionomers and AEMs for energy devices.

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Investigating solvent / membrane interactions by means of molecular simulations

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Despite years of intense research on transport of water and other solvents through polymer membranes, the physical phenomena that control transport through these membranes are not yet fully understood, particularly at the molecular level. This lack of understanding largely comes from the complex structure of polymer membranes and the subtle interplay of intermolecular interactions between the membrane material and the solvent molecules.

In this keynote lecture, we will review a number of recent molecular simulation results that shed light on solvent / membrane interactions and discuss their implications for membrane performance.

First, we will consider the case of pure organic solvents (methanol, toluene, acetone) or solvent mixtures in contact with membranes relevant for organic solvent nanofiltration (OSN) applications such as P84 polyimide membranes and membranes made of polymers with intrinsic microporosity (PIM-1) [1].

We will then focus on the interactions between water and the fully aromatic polyamide membranes that are used on a very large scale for seawater desalination [2].

Finally, we will discuss the crucial "mediating" role played by the solvent in setting up the effective interactions between the solute molecules and the membrane [3].

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Sustainable Fabrication of Biodegradable Cellulose Membranes for Organic Solvent Nanofiltration

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Membrane technology has become an indispensable part of our daily lives. The rapid growth of membrane technology has been breeding an unavoidable yet critical challenge — the unsustainable disposal of used membranes. Commercial polymer membranes are fabricated from fossil-based monomers and polymers that are not biodegradable. Hence, there is an urgent need to develop membranes that are sustainable from cradle to grave, i.e. both bio-derived and biodegradable. Cellulose is one of the most abundant biopolymers that are biodegradable upon disposal. However, it is only soluble in a handful of solvents, limiting its fabrication into membranes at an industrial scale. To circumvent this bottleneck, in this work, we propose a sustainable and scalable method to fabricate cellulose membranes from cellulose acetate with sacrificial acetate group. The proposed method allows cellulose membrane fabrication utilizing green solvents, and the fabrication procedure is sustainable with minimal solvent consumption. One of the most appealing applications of cellulose membranes is organic solvent nanofiltration (OSN). It is an emerging technology to separate solutes in nano-precision in harsh organic solvents, requiring solvent-stable materials. Surprisingly, the cellulose membranes exhibited unique transport behaviors, with solute rejection ranging from 100% to –100% depending on the solvent medium. Such trends were not previously observed in the OSN literature, and the underlying mechanism was thoroughly investigated. Importantly, the membranes were completely biodegradable in a carbon-neutral manner upon disposal. The life cycle of cellulose membranes was compared with that of conventional OSN membranes in a qualitative and comparative study. The proposed methodology can be applied to substitute fossil-based polymers in all aspects of membrane technology, and it has the potential to become a sustainable fabrication platform for membrane materials.

From simple casting polytriazole membrane to fractionation of crude oil

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There is an urgent need for cleaner and sustainable energy conversion, and membrane technology can be an alternative to conventional energy-intensive separation processes [1]. However, there is still a lack of separation materials operating under industrial conditions and complex mixtures. We have demonstrated that polytriazole with OH groups is a versatile polymer that combines high thermal stability and good mechanical stability with easy processability, making it suitable for preparing membranes for separation applications [2]. This paper reports a simple strategy to fabricate polytriazole asymmetric membranes with ultrathin selective layers by combining the non-solvent induced phase separation method and thermal crosslinking [3]. The method allows us to design layers of 10 nm, which contain sub-nanometer channels. In addition, the selectivity of the layer can be tuned to the lower end of the typical nanofiltration range (200 to 1000 g mol^{-1}). The resulting membranes were tested to separate one of the most complex mixtures: crude oil fractionation. The polytriazole membrane can enrich up to 80-95 % of the hydrocarbon content with carbon numbers below C10 (140 g mol^{-1}). These membranes preferentially separate paraffin over aromatic components. Moreover, by controlling the fabrication conditions, we are able to apply a membranes cascade configuration for fractionation procedures, making these membranes suitable for integration in hybrid distillation systems.

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Elevated temperature performance of solvent-resistant nanofiltration membranes and role of solvent activation

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Recently developed one-pot crosslinked poly(vinylidene difluoride) (PVDF) nanofiltration and well-known diamine crosslinked polyimide (PI) were prepared and tested for solvent resistance nanofiltration (SRNF) applications. Experiments were performed in a broad range of solvents from apolar to polar solvents namely butyl acetate, chloroform, xylene, toluene, isopropanol, ethanol, and water. Seven different types of dyes, charged ones in polar solvents namely, Rose Bengal (1036 Da), Acid fuchsin (585 Da), Richard dye (551 Da), and Rhodamin b (479 Da), and hydrophobic ones in apolar solvents namely, Sudan Black B (456 Da), Zinc tetraphenylporphyrin (ZTPP) (678 Da) and 5,10,15,20-Tetrakis (3,5-di-tert-butyl phenyl)porphyrin (TBPP) (1036 Da) were used. Depending on the solvent and the solute, the MWCO of membrane can change. Butyl acetate and chloroform filtrations release the sharpest MWCO for PVDF crosslinked and PI crosslinked membranes respectively. High-temperature solvent resistant nanofiltration of TBPP solute in butyl acetate, chloroform, xylene, and toluene using both membranes illustrated permeance enhancement by increasing the temperature for all four solvents due to the viscosity drop and polymer chain flexibility rise. The results of consecutive room temperature show that the performance of both membranes remains the same for xylene, butyl acetate, and toluene, but is lost in chloroform after the high-temperature filtration. Pre-treatment with solvent, also known as solvent activation or annealing, is an efficient method to have a beneficial influence on both retention and permeance. The effect of solvent activation or annealing of the membrane by butyl acetate, toluene, xylene and chloroform was investigated and revealed that the changes induced by solvent contact are physical rather than chemical in nature.

Fluorinated thin-film composite membranes with twisted monomers for high flux non-polar solvent nanofiltration

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Conventional polyamide thin-film membranes have previously been reported to be chemically stable and capable of separating a wide range of organic solvents with varying polarity. However, tuning the polymer structure and membrane properties remains challenging, particularly to separate nonpolar solvents. For enhancing performance and applicability of the TFC membrane for nonpolar organic solvent, we employed interfacial polymerization using tailor-made twisted building blocks to synthesize defect-free TFC membranes. The TFC membranes were prepared successfully via interfacial polymerization (IP) using hexaflourinated monomer as an organic co-monomer with a twisted structure and trimesoyl chloride (TMC). Hexaflourinated monomer is a twist tetrafunctional monomer with a hydrophobic group ($–CF_3$) has a great influence on the three-dimensional structure of the polyamide nanofilm, which can form large intramolecular free-volume voids. Upon addition of the co-monomer, the membranes allow the transport of nonpolar solvents such as aliphatic and aromatic hydrocarbons. Note that such transport was challenging with the conventional hydrophilic polyamide membranes. The resulted membrane achieved toluene permeance of 15.84 L m^{-2} h^{-1} bar^{-1} and a Sudan Black (350 Da) rejection of 97% under the optimal preparation conditions. Our study demonstrates that twisted monomers can be used in interfacial synthesis to produce nanofilms with improved microporosity without complex processing.

Drastic performance change of NF-membranes after contact with water/solvent-mixtures

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Industrial water/solvent-mixtures must be recycled or treated before discharge. Solvent-tolerant nanofiltration (STNF) is a process between the fields of aqueous NF and solvent-resistant NF (SRNF). Industry has not yet developed STNF to mitigate water/solvent-mixtures streams for e.g., pharma and Oil & Gas industries. This study aims to answer whether aqueous NF and SRNF can treat water/solvent mixtures. One aqueous NF (FilmTec™ NF245), and two SRNF membranes (DURAMEM® 300 (PI) and crosslinked PVDF) were exposed to a mixture of either DMF, DMSO or acetonitrile with water. Depending on the water/solvent mixture ratio, NF-membranes showed a very significant decrease in both retention and permeance.

Characterization of size-fractionated biopolymers in surface water for better understanding of membrane fouling in MF/UF

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In recent researches on microfiltration (MF) and ultrafiltration (UF) for drinking water production, natural organic matter (NOM) in the feed water has been intensively characterized by liquid chromatography with organic carbon detection (LC-OCD), and high molecular weight hydrophilic organics referred to as biopolymers have been identified as the key foulants. Biopolymers are comprised of various components. It has been reported that biopolymers in surface water have a wide range of molecular weights from 10 kDa to 1 MDa [1]. Although properties of biopolymers may be different depending on molecular weights, this point has been rarely investigated. In this study, biopolymers were isolated [2] from surface water used as drinking water source, and the isolated biopolymers were further separated into three fractions (large, medium and small biopolymers) on the basis of molecular sizes. Fouling potentials of the size-fractionated biopolymers were assessed, and further characterization of the fractionated biopolymers was also carried out for understanding the mechanisms of membrane fouling caused by biopolymers. It was found that the large biopolymers had the highest fouling potential. This could be explained by their high affinity to membrane polymers (PVDF) and their soft natures, which were revealed by QCM-D analysis. The high affinity of large biopolymers to PVDF polymers and their soft natures would be attributed to properties of polysaccharides contained in the large biopolymers. Thus, rather than dealing with biopolymers as a whole, the large biopolymers should be focused on for the mitigation of membrane fouling.

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Nitrite removal by catalytic polymeric hollow fiber membrane contactors

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The presence of nitrates is one of the leading causes of groundwater degradation. In this sense, this pollution source is produced by the excessive use of fertilizers in agriculture, as well as other anthropological sources such as sewage or industrial wastewater discharge without adequate treatment. Nitrates can dramatically affect both human health and the aquatic environment. Therefore, the WHO establishes a limit of 50 ppm nitrates to ensure safe water. Currently, the removal of both nitrites and nitrates is mainly performed by reverse osmosis, ion exchange, electrodialysis or biological denitrification. However, the main disadvantage of these technologies is the production of brines with high nitrate concentration or sludge formation in the case of biological denitrification. In this context, this work aims at developing and testing advanced polymeric hollow fibres with alumina-supported palladium nanoparticles assembled in a hollow fiber contactor as an efficient catalytic nitrite removal from polluted water. PVDF fibres with the presence of Pd/Al_2O_3 nanoparticles were fabricated by phase immersion using a spinneret to subsequently assemble a hollow fiber membrane contactor. The experiments were performed in continuous mode, with an initial nitrate concentration of 150 mg/L, flowing the water stream on the shell side (112-200 mL/min), and the gas stream (20-417 mL/min) composed of hydrogen as reagent, CO_2 as pH buffer, and nitrogen through the lumen of the fibres. Nitrite reduction over time has been duly analyzed, achieving 100% nitrite reduction with a nitrogen selectivity of more than 99%, avoiding ammonium production.

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Long-Term Performance of Full/Commercial-Scale Gravity Driven Membrane Filtration with Passive Fouling Control for Drinking Water Treatment

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GDMF with passive fouling control measures, referred to as PGDMF, uses a low hydrostatic pressure for permeation and fouling control, without any mechanical pump and chemical system. Due to its simplicity, PGDMF is emerging as a promising approach to provide drinking water, especially in small communities. With these passive fouling control measures, a higher permeate flux can be sustained than for the traditional GDMF system, which is discussed in a separate abstract submission. However, the frequency and effectiveness of the passive fouling control measures depend on the extent of fouling which is expected to be impacted by raw water characteristics and the operating conditions. A full/commercial-scale PGDMF system was implemented to supply drinking water for a small community in British Columbia, Canada. The system includes an intake, four ultrafiltration membrane modules (SUEZ ZW1500), and a chlorine disinfection system. The intake is from a nearby river which is expected to have seasonal variations in raw water characteristics. A higher decline rate in the permeate flux was observed during the warmer season but the effect was likely reversible. Over 1 year+ of operation which is still ongoing, the permeate flux decreased steadily but very slowly to ~6L/m^2/h (LMH) at end of 13 months (60% of the initial value). The results demonstrate the viability of PGDMF at full/commercial-scale, enabling a stable operation with a permeate flux decreasing at a very slow rate over an extended period, without any extensive cleaning. To promote a greater throughput in the PGDMF system, different operating set-points including hydrostatic pressure for permeation and frequency of passive fouling control, were considered. The presentation will expand on the system design and performance as well as the optimization potentials, presenting 20 months of data (until the end of October 2022).

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Combining ultraviolet light emitting diodes with membrane filtration to develop effective water disinfection systems

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The development of effective disinfection treatment processes will be crucial to help the water industry cope with the inevitable challenges resulting from the increase in human population and climate change. UV treatment using low pressure mercury lamps is widely applied to achieve disinfection of water due to the proven effectiveness of this treatment process against a wide range of waterborne pathogens. In addition to its disinfection effectiveness, UV can also degrade organic compounds by direct photolysis of photolabile compounds as a consequence of light absorption. The use of UV-LEDs for disinfection recently emerged as a disinfection alternative due to their advantages: they are mercury free and therefore don't lead to the production of mercury waste, compact, robust, have longer lifetimes, do not need stabilization time, lead to a low energy consumption and can be constructed with a diversity of wavelengths. So, if proven effective, they can replace low pressure mercury lamps. Three single small ultraviolet-C diodes emitting light at different wavelengths were tested in terms of their efficiency to inactivate water quality indicator bacteria (total coliforms and Escherichia coli) and fungi [1-3]. The small LED system achieved extremely high inactivation levels in real wastewater effluents. LEDs that emit at 265nm led to DNA and phenotypic damages, as well as effects in the membrane permeability, enzymatic activity and proteome response [2-3]. Two different custom-made LED panels were built and tested for surface water disinfection using a submerged hybrid photocatalytic membrane reactor previously developed and tested for the treatment of olive mill wastewaters [4]. The combination of membrane filtration and LED panels achieved an extremely high quality of permeate in terms of water quality indicator bacteria and bacteria resistant to antibiotics while guaranteeing an extremely high treatment of the retentate, one of the issues associated with the membrane processes [5-6].

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Using Natural Virus Markers to Safeguard the Integrity of Membranes in Water Treatment Plants

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Water sources, used for the production of drinking water, are often contaminated with pathogenic micro-organisms e.g. viruses. Their effective removal/inactivation is the main strategy to safeguard our water systems. Unfortunately, virus removal by water treatment processes is difficult to monitor, because the concentration of pathogenic viruses is normally too low to measure. There is an urgent need for a fast, precise and simple method to determine the removal of viruses by water treatment processes. This is particularly relevant for membrane filtration systems, which are increasingly used for the reuse of highly contaminated water sources (e.g. wastewater or effluent) for the direct and indirect production of high quality water, such as drinking water [1]. Membrane integrity is typically monitored by low-sensitive measurements resulting in a maximum of 3 Log removal values (LRV), hampering further implementation of membrane-based treatments. The lack of testing methods for virus removal led to the development of our new natural virus (NV) method [2], which is based on indigenously present virus markers in water sources. This novel NV method is used for the first time to regularly monitor four full scale drinking water treatment plants, including MF/UF/NF and RO membranes. We have monitored up to 5 LRV for UF membranes. End-users can use the data to exactly measure the performance of the membranes, and also determine when performance is decreasing and anticipate on membrane replacement. Furthermore, on pilot scale we deliberately induced membrane damage to membranes to determine the effect on virus rejection. We have investigated impairment, chlorine and abrasive damage on RO membrane modules and the influence of fibre breakage on UF membrane modules. From these deliberate membrane damage tests, we concluded that the NV method is a very promising tool to monitor membrane performance.

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A Facial Polypyrrole Deposition Strategy on Commercial Substrates for Freshwater and Salts Generation from Brine via Photothermal Membrane

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Water-scarce countries need a radical re-think of water resource planning and management including the innovative exploitation of a growing set of viable but unconventional water resources for sector water uses, ecosystems, climate change adaptation, and sustainable development. The generated brine water from seawater desalination plants is considered one of the hindered factors of desalination expansion. Solar-driven evaporation via photothermal membranes is considered a sustainable strategy for clean water production through desalination and wastewater treatment to overcome the global challenge of water scarcity. In this work, different strategies of pyrrole chemical vapor deposition "CVD" polymerization were studied as a function of pyrrole concentration (0, 5, 10, 15, 20, and 25 μL), type of substrate (PVDF, woven and non-woven fabric), the oxidizing agent $(NH_4)2S_2O_8$, $FeCl_3$, $CuCl_3$, $KMnO_4$, and $Na_2Cr_2O_7$), and the concentration of oxidizing agent (0.1, 0.5, and 1.0 M), at 80 °C for 1 h. The optimized facial deposition strategy resulted in a stable deposited dark layer of polypyrrole "ppy" onto various porous substrates, which acts as an efficient light absorber to harvest solar light and achieve excellent heat localization on the thin-layer of water surface for solar-driven interfacial water evaporation. The obtained photothermal membranes achieved light absorption up to 95 % under one sun illumination (1 $kW.m^{-2}$), with accelerated water evaporation up to 1.35 $kg.m^{-2}.h^{-1}$. Furthermore, salts harvesting is achieved with self-cleaning of the photothermal membrane due to the superior hydrophobic deposited ppy layer on the top surface of the non-woven fabric without any salts accumulation after running for one week under natural sunlight.

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Modeling the effect of an applied AC field on colloidal deposition

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Colloidal fouling remains a major challenge in many membrane separation processes, including water purification, as well as bioseparations such as proteins and viruses. Fouling adversely affects system performance and, consequently, cost, leading to many studies aimed at controlling fouling, including passive methods such as various forms of surface modification, and active methods such as manipulating hydrodynamic conditions or applying an external electric field. Of the latter, DC electric field have received much of the attention, while AC electric fields are far less understood. Here, a theoretical model is derived with the goal of examining the effect of an applied AC field on a colloidal particle adjacent to the membrane surface. Specifically, we examine the shown the long-range time-averaged electric field that emerges under AC conditions, as a consequence of ion transport asymmetry. This field persists over length-scales far greater than the electric double layer thickness, and will present a long-range electric repulsion force on a like-charged particle. The developed framework further considers the effect of the permeation. The force balance on a particle is calculated, establishing the parametric phase-space permitting equilibrium positions to exist, whereby a particle, dragged toward the membrane by permeation, is then held fixed, at a finite distance from the membrane, through the action of the time-averaged electric force. The dependence of such equilibria on system parameters is explored, particularly ionic composition and the frequency of the applied AC field, revealing the regime where AC fields may be applied for colloidal fouling mitigation.

Molecular modeling of PIM-2: From structural characteristics to transport behaviour

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The partially fluorinated polymer of intrinsic microporosity, PIM-2, synthesized from 5,5′,6,6′-tetrahydroxy-3,3,3′,3′-tetramethylspirobisindane (TTSBI) and decafluorobiphenyl (DFBP), is a promising candidate as a membrane material for the separation of important gas mixtures. Industrially relevant processes where this material may be successful include carbon capture from flue gas (CO_2/N_2 separation) and biogas upgrading or natural gas sweetening (CO_2/CH_4 separation) [1]. A computational study was performed in order to correlate the morphological analysis with the gas transport proprieties of this polymer [2,3]. The results were compared with the experimental analysis to shed light into the molecular structure/properties relationships. Via molecular modelling, a high fractional free volume (FFV) of about 34% was computed, and a Brumauer-Emmett-Teller (BET) surface area of 642 m^2g^{-1}, in very good agreement with the experimental value of 636 m^2g^{-1}. The relatively low BET surface area compared to other PIMs, in combination with a high FFV, suggests low interconnectivity of the free volume elements. This is the reason for the modest permeability and separation performance, that sees CO_2 being the most permeable gas and N_2 the least permeable gas. Sorption isotherms show dual-mode behaviour for all gases, CO_2, CH_4, O_2 and N_2, except for H_2 that follows Henry’s law. The solubility coefficients decrease as a function of gas critical temperature, indicating that the polymer chains appear to have no particular interaction with the gas molecules, whereas they establish inter- and intra-chains halogen and hydrogen bonds.

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Dynamic modelling of Reverse Osmosis: added value for operation and control

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Most reverse osmosis (RO) models consider steady state operation, hence ignoring the transient character of the system and assuming equilibrium at all times. As a consequence, important effects are not simulated in a steady state model. In contrast, dynamic models take into account this time dependency and simulate the system’s behaviour towards a new equilibrium. On the other hand, solving a dynamic model is more complex and requires more computational capacity. This raises the question of the added value of a dynamic model in improving full-scale operation of RO systems. In this study, a steady state and a dynamic model are compared. Both models were applied to a full-scale installation of a Flemish drinking water company. The steady state RO model, based on the solution-diffusion model, is described in Gaublomme et. al [1]. The dynamic RO model, built on the same physical principles, is constructed from mass balances and partially adopted from Al-Obaidi and Mujtaba [2], and uses online data, such as feed temperature and conductivity, as input. The step response of the output permeate concentration for a step input for several input variables is investigated, since its dynamics are slower compared to other model outputs. This can be explained by concentration polarisation, which is a result of (slow) solute diffusion towards the membrane interface. The concentration dynamics are thus diffusion-limited in rate. At one minute, which is the measurement frequency of the online sensors, the steady state model hides this effect by assuming equilibrium, and deviates by more than 37% from the dynamic model. Equilibrium is only reached after 5 minutes. This is important when making decisions concerning the operation of a RO plant. Indeed, dynamic modelling offers opportunities to real-time control of the installation by incorporating these transient effects, which is not possible with a steady state model.

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Prediction of water uptake and diffusivities in ion-exchange membranes via Molecular Dynamics simulations

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Very recently, the field of process technology has witnessed an exponential increase in the utilization of ion-exchange membranes (IEMs) in light of their pivotal function in green technologies such as electrodialysis (ED), reverse electrodialysis (RED), and fuel cells. Various computational chemistry techniques are currently employed to reliably model the IEMs' properties, as understanding the underlying structure-properties relationships in these materials might ultimately lead to the design of novel devices. In particular, the equilibrium water uptake in IEMs has been shown to have a prominent effect on some of their fundamental properties [1]. In this work, we have elaborated a molecular dynamics-based protocol to reliably predict the water uptake of IEMs by considering a tetramethylammonium-functionalized polysulfone anion-exchange membrane (AEM) as a case study. The procedure led to a favorable agreement with reported experimental data in a wide range of ion-exchange capacities and improved the results with respect to the DFT-based approach developed in our previous work [2]. The thickness of the membrane model was found to be a critical aspect, as if not large enough, it can lead to inaccurate results. The issue was addressed by proposing an alternative simulation setup with respect to those reported in the literature. Finally, we have illustrated how to employ the knowledge of the water uptake, evaluated in molecular scale, to compute chloride counter-ion diffusivities within three different theoretical frameworks. It was revealed that a reasonable agreement with experiments can be achieved, which confirms the potential of the current strategy for the prediction of ion diffusivities in IEMs without resorting to experimental data, thus paving the way toward a computationally driven design of new materials for IEMs.

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Modelling and simulation of HCl and NaOH production by means of photovoltaic solar - electrodialysis with bipolar membranes

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Modelling and simulation are reliable tools for the estimation of the technological performance of processes, which can be employed not only for the selection of operative conditions and optimization, but also for the scale-up analysis of technologies. Hence, the increasing demand and the rise of new applications for electro-membrane processes result in the need for development of accurate models. In this sense, research efforts have been carried out in the development of models for electrodialysis with bipolar membranes (EDBM) [1]. Integration of renewable energies (e.g. solar photovoltaic (PV) energy) with EDBM can contribute to the decarbonization of electro-membrane processes [2], although there is a lack of studies regarding its modelling and simulation. Therefore, the present work aims at the modelling and simulation of an EDBM-PV integrated process. The simulation will be executed using gPROMS software, considering a five-parameter model for PV modules [3] that provides the current-voltage conditions from PV panels datasheet and irradiance and temperature curves from database. Feed and bleed and batch operation modes have been evaluated through the developed EDBM-PV model taking into account the performance of both EDBM and PV solar energy system and aiming at the optimization of the coupling strategy in order to maximize the performance indicators of the integrated system, such as productivity, products quality and reduced energy consumption As a case of study, the model has been used to calculate the required PV panel area for an integrated EDBM-PV plant in Lampedusa (Italy) as an example of a real location within the Mediterranean regions, thus, further demonstrating how the developed EDBM-PV model can be a useful tool to support the scaling-up of integration of renewable energies with electro-membrane technologies.

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Modelling and tuning of structure-performance relationship for rapid increase in proton conductivity of composite PEM membrane for application in fuel cell

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PEMFC or Proton Exchange Membrane Fuel Cell efficiently transform the chemical energy into electrical energy with zero emission. Among many verities, the most efficient one, PEMFCs utilize porous polymeric membrane as electrolyte. One of the major barriers to large-scale commercialization of this technology is its loss of proton conductivity at high temperature and low humidity rendering it ineffective for practical application. The answer to the challenge is proton conductivity enhancement through structural modification. Many researchers have reported highly customizable fabrication of various composite membranes with varied pore structure which is experimentally proven to significantly enhance the efficacy, even in adverse operating conditions. However, a comprehensive theoretical study is not yet reported to identify the most effect additive and polymer and its distribution for maximum proton conductivity at various operating conditions for specific application. In this work, a first principle-based physio-chemical model of proton transport through generic composite PEM membranes has been developed accounting for both the vehicular and hopping proton transport mechanism. Applicable governing equations (continuity, Navier-Stokes, advection diffusion, Poisson and Nernst-Planck equation) and boundary conditions have been solved numerically to study the distribution of water velocity, potential and proton distribution. The proton conductivity subjected to velocity, potential and proton concentration distribution has been predicted based on ohmic relationship. Pore level structural variation with uniform square and hexagonal structure along with random additive distribution enables to evaluate proton conductivity with porosity, additive distribution and potential difference, operating temperature and humidity. The simulated results have been validated the with the experimental values for precise prediction of proton conductivity of composite membrane. Further the conductivity prediction has been extended for structural optimization for practical scenario. This model has the potential to offer a ready-made guideline for choosing futuristic membrane material for intended operating condition which is crucial for large-scale application.

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Removal of steroid hormone micropollutants from water in reactive membranes

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Steroid hormones are endocrine disrupters and micropollutants that reach the aquatic environment mostly from wastewater discharge and pose a high risk to both environmental and human health. Nanofiltration can achieve high but incomplete removal, requires high operating pressure and energy for effective removal and micropollutants accumulate in the concentrate. 'Reactive' membranes, such as photocatalytic membranes; are an alternative that addresses such shortcomings. Such membranes utilize the nanoconfinement of micro- and ultrafiltration membrane pores (1-200 nm) in which reactions occur, while water is being transported through these pores. If successful the micropollutants are degraded in situ within the short residence times in the order of seconds. The phenomena that contribute to photodegradation are presented, followed by some examples of steroid hormone degradation in a flow-through photocatalytic membrane reactor. The membranes used were i) a polyethersulfone–titanium dioxide (PES-TiO_2) membrane produced by collaborators at IOM Leipzig [1, 2], and an organic PVDF membrane with a porphyrin photosensitiser [3, 4]. The TiO_2 nanoparticles (10 nm) and the porphyrin were immobilized in the nanopores (220 nm) of the membrane polymers. Water quality and operational parameters were evaluated in order to determine limiting factors in the degradation of steroid hormones. Steroid hormone concentrations ranged from low environmentally relevant concentrations 50 ng/L to near the solubility limit of 1 mg/L. Flow-through the photocatalytic membrane increased contact between micropollutants and reactive oxygen species in the pores and high removal (80-95%) was achieved. Results show that relatively simple material can achieve a very high removal of micropollutants and further enhancement is achievable through potentially smaller pores (nanoconfinement), modified photocatalytic materials and longer residence times – that is reactive membranes operated at nanofiltration range fluxes with pressures typical for micro- and ultrafiltration. As such the ambitious water quality guidelines (1ng/L for drinking water) are reachable with drastically enhanced water permeability.

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Improved Performance of a Slurry Photocatalytic Membrane Reactor with a Vertical Filter and an External Membrane

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The research in the field of photocatalytic membrane reactors has evidenced, in almost 20 years, that slurry photocatalyst and submerged membrane is a configuration that seems interesting in the mineralization of recalcitrant pollutants in wastewaters [1]. However, submerged membranes are only able to retain the photocatalyst particles and not many types of pollutants [2], so a RO membrane was used in series to the submerged membrane to improve the quality of the treated water [3]. Two membrane systems in series are quite complicated to operate as well as they have high capital and operating costs. A new configuration, based on a vertical filter working under hydrostatic pressure (able to retain the photocatalyst) and an external nanofiltration (NF) membrane (able to retain pollutants in the photoreaction zone), has been designed, built and tested. The behavior of the vertical filter in presence of the photocatalyst and the photocatalytic performance of the overall system, in a batch configuration, in the photodegradation of gemfibrozil as a model pollutant have been studied. Some parameters (e.g., TiO_2 amount, pH, light intensity and wavelength, air and oxygen, membrane rejection) that influence the performance of this integrated system (photoreactor, filter and membrane) have been considered. The filter was able to retain the TiO_2 particles while assuring higher flux than a submerged membrane (200 vs. 40 L m^{-2} h^{-1} average values). A pH 8.0 (giving 40-50% rejection of the Fortilife NF membrane) was chosen against pH 9.7 (giving 90% rejection), to avoid filter plugging. The external membrane was not exposed to light irradiation, permitted the recycle in the photoreactor of the not-photodegraded pollutant and produced a treated water (permeate) quality better than a submerged membrane. The obtained overall performance was 80% reduction of gemfibrozil concentration but it can be further improved by a fine tuning of the various parameters, as well as the operation can be of continuous type as required by many industrial applications.

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Membrane assisted methanol synthesis reactor to enhance a methanol production from CO_2 and H_2 in biomass to biodiesel route

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Liquid biofuels have a significant role to play in meeting the climate change targets. Presently approximately 0.1 percent of all biomass is used to produce biodiesel, owing to an inefficient and complex production process. The EU CONVERGE project demonstrates a new innovative process, that will make biodiesel competitive with fossil fuels in terms of both effectiveness and price. One of the technologies developed in this project, is an enhanced methanol membrane reactor. In-situ methanol and/or water separation greatly enhances per pass production of methanol, thereby reducing equipment size significantly. Additionally, selective removal of reaction products will decrease the energy demand in downstream processing. This project aims to increase the CO_2 and H_2 conversion per pass to 33% and to reduce CAPEX by 10%. To demonstrate membrane reactor performance the multi-tubular membrane reactor system was constructed and tested. This multi tubular system that consists of: packed bed reactor and, cooler and multi-tubular membrane reactor in series. The membrane reactor has 7 membrane tubes ceramic supported polyimide membranes with total effective area of 0.25 m^2. The isothermal conditions are maintained by circulating of the thermal oil. Membrane reactor showed stable performance over 2664 h of testing. In all experiments, achieved CO_2 conversion and methanol yield in the multi-tubular membrane reactor were beyond equilibrium measured in the packed bed. For the stoichiometric H_2/CO_2 ratio, the highest CO_2 conversion and MeOH yield were achieved at GHSV = 449 h^{-1} at temperature of 230 °C: CO_2 conversion =30% and MeOH yield= 17%. Feed pressure was 35 bara, and permeate pressure 1.5 bara. This represents 36% and 21% increase of the CO_2 conversion and MeOH yield, respectively, compared to the equilibrium conditions.

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Polymeric membrane engineering for catalytic membrane reactor: from materials design to fine chemistry production and CO_2 valorization

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Polymeric membrane filtration can be efficiently coupled with catalytic chemical reactions to obtain an optimized and intensified process where the reaction and the purification can be realized in one-step. These systems, called catalytic membrane reactor (CMR) are particularly efficient when the catalyst is directly included in the membrane. The incorporation of the catalyst needs a prior modification of the membrane in order to avoid future catalyst leaching during the process. We will present here with the example of metallic catalytic nanoparticles (MNP) mixed membranes that the pertinent choice of the strategy to achieve this goal permits to enhance the performance of the process in term of productivity and quality of the final product. We have study the chemical UV-photografting of charged or neutral reticulated polymeric networks at the membrane surface. We found that the structure of the gel (charge, porosity, swelling) affects the properties of the catalysts and its efficiency (conversion, selectivity) for catalytic reactions (nitroaromatic reduction, Suzuki-Miyaura coupling, Hydrogenation) [1-2]. The main key-point of the high efficiency of our system is the high local concentration of nanoparticles that enhance the kinetics of reaction despite short residence times of the reagents in the active layer of the membrane. Coupled with the high compactness of hollow fibre modules, CMR permit to achieve production compatible with the fine-chemical industry standards (100 ton/years). In this way and for the reduction of the environmental impact of processes, we have developed a pioneering catalytic membrane system with dual properties: CO_2 capture (thanks to the functionalization of membranes by amines) and the catalytic reactivity of CO_2 with organic substrates to produce oxazolidinone-derived compounds for the pharmaceutical industry.

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Photocatalytic membrane reactor based on photocatalytic nanoparticles immobilized on Thin Film Composite (TFC) membrane for removal of Methyl Orange

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Wastewater from textile industry contains different kind of dyes difficult to degraded and chemical additives, such as salts, detergents, organic acids a part of which is discharged into the sewer system, what causes an important environmental impact [1]. Especially huge amount of inorganic or organic dyes/colors released by the industries wastewater causes water pollution. As consequence, searching an effective advanced method of treating and reusing wastewater from the textile industries is highly challenging [2]. Integrated membrane process and photocatalytic membrane reactor (PMR) represent an interesting and effective solution. PMR is a continuous process that combine a membrane separation and heterogeneous photocatalysis and can be realized in different configurations [3]. Aim of this research is to study the effectiveness of photocatalytical nanoparticles immobilized on a thin-film composite (TFC) membrane for removal of Methyl Orange (MO), an azo dye largely used in textile industry that represents a strong problems for wastewater treatment. The commercial polyamide TFC (150-300 Da) and PES (5 kDa) membrane was immobilized with photocatalytic titanium di-oxide stable suspension, in order to realize a photocatalytic membrane reactor able to degradate MO solution. Preliminary study on stabilization of photocatalytic titanium di-oxide suspension has been carried out by testing effect of sonication and concentration on suspension stability. Membrane properties and permeability before and after immobilization have been evaluated too. Photodegradation has been studied as in batch photocatalytic reactor as in the PMR: effectiveness of the PMR has been evaluated on the basis of the quantity of immobilized catalyst for both the polymeric membranes in order to achieve the process optimization.

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Dispersion synthesis of photocatalysis-Fenton nanocatalysts for organics degradation: Proposed coupling mechanism in membrane reactor

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Micro-morphologic control synthesis of Cu_2O, Co_3O_4 and $CuCo_2O_4$ nanocatalysts was achieved with the membrane dispersion solution method, largely reducing the need of complex surfactant agent. Micro-octahedron enclosed with (111) crystal facets was proved with both Fenton activity and visible light photo-activity. Degradation of organic pollutants in water was enhanced by a combination of photocatalysis-Fenton process upon the bi-functional nanocatalyst. The removal rate of methyl blue increased from 68% in the Cu_2O-based photocatalysis process to 92% in the combined process as adding the oxidant reagent H_2O_2 in 120 min. Furthermore, the degradation reaction rate constant had a 10-fold increase to $k=0.200$ min^{-1} when H_2O_2 solution was feeding through a mesoporous ceramic membrane instead of direct addition. To follow up, the industrial paper-mill wastewater with an initial Chemical Oxygen Demands (CODs) of 813 mg/L was treated, which was the effluent concentrate from reverse osmosis process at the wastewater plant having a treatment capacity of 40,000 t/day. CODs in the effluent was reduced by 79% using the visible/Cu_2O NPs/H_2O_2 system in membrane reactor. Synergistic merge of photo-redox and Fenton reactions could occur in a domain where a high concentration of reactant micro-droplets existed at the membrane surface. The (111) facet dependent reaction coupling mechanism is proposed to explain the mutual stimulating effect. In parallel, the industrial organic wastewater containing N, N-dimethylacetamide as the main pollutant with a total CODs 19,080 mg/L was treated with $CuCo_2O$ NPs/H_2O_2 system in membrane reactor. N, N-dimethylacetamide was mineralized by 99.9% in a fast transformation to inorganic ion NO^{3-}. In a conclusion, the membrane advanced oxidation processes (MAOPs) were proved being able to intensify activation and coupling effect for persistent organics degradation in water and wastewater.

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40 years Polymeric Membrane Development: Highlights and Dead Ends

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The 2022 Euromembrane celebrates the 40th EMS anniversary. This lecture will highlight milestones and dead ends in polymeric membrane developments during the last 40 years. Thousands of new membranes have been introduced in the patent and scientific literature, but the way to commercialization is long and full of unpredictable challenges. As a result, less than 1% of those have become commercially competitive.

Among these are interfacially polymerized TFC membranes for desalination and nanofiltration, Monsanto's multilayer membrane for gas separation, crosslinked polyimide membranes for organic solvent nanofiltration, MTR's composite membranes for organic vapor separation and carbon dioxide capture or GMT/Borsig's silicone-based ONF membrane. More winners will be identified. But what about hot topics like carbon nanotube membranes for desalination, membranes made from polymers with intrinsic porosity (PIMs) for gas separation, Aquaporin containing membranes for water purification, isoporous self-assembled block copolymer membranes for liquid separation, polymer-derived microporous carbon membranes, or mixed matrix membranes containing selective nanoparticles like zeolites or MOFs for gas separation? Winners or losers? The answer is not easy, and I will give my personal academic and industrial view, hopefully provoking opposition. To start the discussion: I invested precious time of my professional life as a membrane researcher in developing mixed matrix membranes containing size-selective absorbents (e.g., zeolites, MOFs) for gas separation. The worldwide research has led to numerous exciting publications in high-ranking journals, and impressive performance data have been published, e.g., for olefin/paraffin separation or carbon dioxide capture, but despite tremendous efforts, we see no real-world applications. Will it ever come? I will give my perspective.

In summarizing, I will identify highlights in polymeric membrane development during the last 40 years, but I will also remind researchers not to jump on the bandwagon and continue to work with "novel" concepts long after the promise has worn off.

Preparation and Characterisation of Dual and Multi-Layer composite ultrafiltration membranes functionalised with photoactive nanoparticles.

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Advanced oxidation is a widely used process for mineralizing organic water pollutants. However, chemical-based oxidation can lead to the risk of unwanted side reactions forming hazardous by-products. Oxidation by means of photocatalysis is an emerging technology showing great promise for the removal of various organic and inorganic water pollutants [1]. Membrane filtration is another popular technique in the field of water purification. Ultrafiltration membranes have pore sizes ranging from 10 nm to 0.1 µm and can effectively reject enzymes, proteins, bacteria, viruses, iron, manganese and total organic carbon [2]. In this study, bismuth-based photoactive nanoparticles were synthesized and incorporated in polyether sulfone (PES) and carboxylated polyether sulfone (CPES) ultrafiltration membranes. Layer by Layer technique was employed using cationic (PDADMAC) [1] and anionic (PAA) [2] polyelectrolytes to coat the feed side of CPES membranes with photoactive nanoparticles. Particle embedded membranes were cast by introducing photoactive nanoparticles into the polymer dope solution. Multi-layer composite membranes were prepared by dual layer casting technique with particles embedded in the thin top layer for a higher particle concentration at the surface and better light exposure. These membranes are characterized using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) techniques. A photocatalytic batch test with suspended nanoparticles in bisphenol A (BPA) solution under VS light irradiation was performed to evaluate the photocatalytic activity of synthesized nanoparticles and found that 90-100 % degradation of BPA was possible. A comparison of photocatalytic performance of particle functionalized membranes is presented in this contribution. [1]Poly-(diallyl-dimethylammoniumchloride) [2]Polyacrylic Acid

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Kinetic model for coke formation on Pd-based membranes in propane dehydrogenation processes

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Palladium-based membrane technology has gained significant attention for its potential application in propane dehydrogenation processes [1], [2]. However, their applicability is limited by the presence of carbon side-products, which may negatively affect membrane stability over time. Carbon-based components tend to adsorb and dissociate on the membrane surface, leading to membrane coking, which inhibits the hydrogen adsorption and dissociation, thus reducing its flux [3]. Even though the membrane coking has been demonstrated to be reversible through regeneration, the large coke formation rate during dehydrogenation processes limits continuous operation in a membrane reactor and hampers the detailed design of this technology. This work aims to investigate the kinetics of coke formation on Pd-based membranes, to gain a deeper understanding into the membranes deactivation mechanism under propane dehydrogenation. We propose a detailed analysis of the mechanistic monolayer-multilayer coke growth models. The mechanism resulting from the best performing model matches with the coking behaviour observed by thermogravimetric analysis (TGA) on conventional thin layer (TL) and double-skinned (DS) Pd-based membranes: monolayer coke forming directly on the surface of the membranes is the main responsible for deactivation and it is more evident in the DS than in the TL membrane. The fittable order h representative of the number of sites involved in monolayer coke formation step, is found to be 5.10 in the DS sample and to 4.28 in the TL sample. The DS membrane offers more available active sites with a higher value of the maximum amount of carbon (C_max) that can be formed on it than the conventional membrane, being equal to 0.0963 and 0.049 mgcoke/cm2sample respectively. The DS membrane deactivates with a faster reaction rate than the TL, and this is in agreement with the faster decay in hydrogen flux observed in the DS than in the TL membrane, during hydrogen permeation tests.

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Solvent-Free Production of Hollow Fiber Membranes

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The recent sustainability drive in the membrane industry has steered the research towards finding greener alternatives for the production of polymeric membranes. One such approach is the Aqueous Phase Separation (APS) technique where an aqueous polymeric solution is transformed into a solid polymeric membrane either via changing the pH [1] or the salinity [2-3] of the coagulation medium. Accumulated research on the APS membranes has already demonstrated its versatility by producing flat sheet membranes with tunable morphologies and performances. Here, we demonstrate that the APS technique can be taken one step further towards scalability by successfully producing hollow fiber (HF) membranes in a dry-jet wet-spinning process. To obtain the HF geometry, a polyelectrolyte dope solution along with the bore fluid is pushed through a single orifice spinneret into a coagulation bath. Depending upon the type of polyelectrolyte, the coagulation bath can be a low pH acidic buffer or just demineralized water. In the case of pH-shift induced APS, the pH and concentration of the acidic coagulation bath determine the rate of precipitation and hence dictate the structure and morphology of the resultant fiber. Using this method, HF membranes with water permeabilities in the range of 12–800 $Lm^{-2}h^{-1}bar^{-1}$ were successfully fabricated which showed excellent performance in micro-and ultrafiltration applications. On the other hand, the HF membranes obtained via salinity-change induced APS showed mild swelling in water and therefore, were not used for traditional water filtration applications. Instead, such membranes showed excellent stability in organic solvents and had ethanol/propanol permeabilities in the range of 20–200 $Lm^{-2}h^{-1}bar^{-1}$. The presented research demonstrates that the APS technique is certainly a scalable and sustainable alternative to the traditional NIPS method where stable membranes for various applications, including organic solvent filtration, can successfully be produced at a large scale.

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Unblocking Ion-Occluded Pore Channels in Poly(triazine imide) Framework

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Nanoporous materials with tunable properties have attracted significant attention in important applications such as gas separation, ions, and water transport. Among these, two-dimensional crystalline, nanoporous materials are poised as the ultimate membrane design. Nanoporous poly(triazine imide) (PTI), belonging to the family of graphitic carbon nitrides, is composed of imide-bridging of triazine rings intercalated with metal halides through the ionothermal synthesis. PTI hosts high density (1.6×1014 cm^{-2}) of 3.4 Å-sized nanopores with outstanding chemical and thermal stability attractive for developing high-performance gas separation membrane either by layer stacking or as a nanoporous two-dimensional filler in mixed matrix membrane [1]. However, the intercalated ions sitting in the gallery spacing block the desired molecule diffusion path. Thus, removal of the intercalated ions is crucial to access the porous framework of PTI. The possible layer displacement of PTI after depletion of ions from AA' stacking to AB stacking [2], resulting in the pore occlusion by the triazine ring in the adjacent layer; however, so far, there has not been conclusive evidence to identify the atom position. Herein, we demonstrate the approach to easily tune the concentration of intercalated Li+ and Cl- ions in PTI framework using acid treatment while preserving the crystallinity and the morphology of PTI nanosheets. The direct visualization of ion-depleted PTI using integrated differential phase-contrast scanning transmission electron microscopy (iDPC-STEM) fueled by X-ray scattering techniques and solid-state nuclear magnetic resonance spectroscopy confirms the coexistence of open channels and occluded domains for the first time. The extent of open channels with AA' stacks increases with higher depletion level. An increasing population of open channels vastly improves the proton conductance through PTI from 1.1 mS/cm to 5.4 mS/cm, giving a prospect of PTI for application in molecular transport.

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Molecular-Level Engineering of Highly Selective Composite MOF-CNT-PDMS Membranes for Effective Gas Separations

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The essence of the Molecular-Level Engineering (MLE) concept is to control the structure and morphology of prepared materials at the molecular level. Besides the targeted design of nanomaterials for specific purposes, MLE can also control anchoring nanoparticles by chemical bonding between surface groups and the support (polymer or carbon-based materials). The main goal of this work was the development of innovative MOF-CNT-PDMS composite materials for targeted gas separations (e.g. CO_2/N_2, CO_2/H_2 and C_3 olefin/paraffin) [1]. A successful synthesis of the ZIF-8 gutter-layer on the free-standing chemically modified carbon nanotube (CNT) platforms was conducted via the ZnO linkages. The formation of aligned ZIF-8 arrays onto the CNT surface acted as an effective buffering layer that boosted the molecular gas transport through the membrane. The interstitial pores at the surface-bulk of the CNT-ZIF-8 formations were sealed by thin polydimethylsiloxane (PDMS) layer deposited via the spray-coating. The resulting non-defect morphology was confirmed by the SEM and the 3D optical profilometer. Furthermore, the PDMS deposition improved the composite's mechanical stability (stress vs. strain) with a twelvefold increased elongation and thermal stability, leading to a 46% mass reduction. Moreover, composite membranes exhibited outstanding gas separation performance with high CO2 and C3H6 permeabilities of 8705 and 4965 Barrer, respectively and selectivities overcoming the appropriate 2008 Robeson upper bounds for various gas pairs CO_2/N_2 (ideal selectivity α=45.6) and CO_2/H_2 (α = 23.9) gas pairs. Similarly, the results of the other tested gas pairs, e.g. C_3H_6/N_2 (α=26.0), and C_3H_6/C_3H_8 (α=4.9), were further remarkable compared to the literature. Hence, the MOF-CNT-PDMS composite membrane concept has the excellent potential for fabricating efficient membranes suitable for targeted gas separation with high commercial and environmental relevance (CO_2 capturing, hydrocarbons recovery for energy harvesting, and olefin/paraffin separation).

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Environmentally friendly extraction of c-phycocyanin from Arthrospira maxima and its purification by ultrafiltration

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Phycocyanins (PCs) are photosynthetic pigments produced by cyanobacteria with great potential for industrial and commercial development due to their antioxidant, anti-inflammatory, anti-cancer and hepatoprotective properties. Their market value is estimated to be around 10-50 million US$ per year [1]. Pressure-driven membrane operations represent an useful approach in fractionation, purification and concentration steps of PCs as alternative to expensive sequential purification techniques such as ammonium sulphate precipitation and gel filtration chromatography [2,3]. In our approach a combination of aqueous extraction and ultrafiltration (UF) was investigated to produce PC with food-grade purity from Spirulina (Arthrospira maxima), a blue-green coil shaped cyanobacterium. The biomass-solvent ratio was optimized in order to maximize the yield of PC in the aqueous extract. 20 kDa flat-sheet membranes and 20 kDa hollow-fiber membranes, operating in dead-end and cross-flow configuration, respectively, were studied to remove non-protein molecules (mainly DNA) from PC in the centrifuged extract. Experiments were performed in batch concentration mode up to a volume concentration factor (VCF) of 5 and the UF retentate was diafiltrated with distilled water in order to enhance the PC purification. Membrane performance was assessed in terms of PC and DNA retention, productivity, fouling index and cleaning efficiency. PC rejections measured in dead-end and cross-flow filtrations with selected membranes were of about 96% and 98%, respectively. According to the mass balance of the process more that 97% of PC was recovered in the UF retentate. Diafiltration allowed to remove more than 90% of the DNA from the crude extract, thereby improving the purity of the PC in the retentate fraction. In both processes the purity degree of the final product resulted higher that 1.1, making it suitable for human food use. Purified PCs have been separated by SDS-PAGE, sequenced by means of mass spectrometry and identified by bioinformatics.

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Solvent-exchange drying as a conditioning tool for solvent-resistant membranes

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Membrane conditioning is defined as the set of practices which aims to preserve the membrane in its original state after synthesis. It most commonly involves drying of the membrane to reduce transportation costs, prevent microbial degradation, reduce ageing effects and facilitate welding of membranes in a module. While being an indispensable step in the membrane production process, little scientific literature has been dedicated to the topic of membrane conditioning. With an increasing number of research groups involved in membrane scale-up, validation and optimization of these conditioning protocols becomes ever more important. Successful membrane drying requires a solvent-exchange. The latter relies on the displacement of pore liquid in the membrane with liquids of lower surface tension to minimize capillary pressures which would otherwise cause collapse of the smallest membrane pores. In this research, different solvent-exchange sequences are compared for their ability to preserve the pores of (crosslinked-)polyimide (PI) integrally skinned asymmetric (ISA) membranes upon drying. In the case of non-crosslinked polyimide membranes, a severe decrease in ethanol permeance with respect to the undried membrane was observed, when directly drying from water (>99% and 94% for ultrafiltration (UF) and nanofiltration (NF) membranes respectively). By performing solvent-exchange prior to drying, the permeance drop could not be reduced more than 76% and 27% for respectively non-crosslinked UF and NF PI membranes. In the cases of crosslinked-PI membranes, identical solvent-exchange procedures did prevent a permeance drop, maintaining an identic permeance compared to a pristine, non-dried membrane. In search for a deeper, theorethical understanding of the drying process, the Young's-modulus is proposed as a diagnostic parameter to quantify the physical strength of a membrane matrix to resist pore collapse. Additional physicochemical characterization techniques (TGA, contact angle measurements, gas-liquid porometry) are employed to develop a framework of principles for rationalizing the drying process of ISA membranes.

Elucidating the impact of natural organic matter composition in feedwater on the chemical cleaning performance of aged PVDF based ultrafiltration membranes

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Chemical cleaning is essential to maintain a sustainable permeate flux in membrane systems. However, long-term exposure to chemical cleaning agents, accumulation of irreversible foulants and/or other rigors of full-scale operation have been documented to cause irreversible changes in membrane performance and characteristics. These changes are typically referred to ageing and eventually trigger replacement of membranes in a system [1]. Anecdotal evidence and recent research suggest that the effectiveness of chemical cleaning decreases as membrane age due to changes in membrane affinity to typical foulants such as natural organic matter (NOM) [2]. However, the extent to which NOM fractions in feedwater impact cleaning effectiveness of aged membranes, and the mechanisms governing this impact, are unknown. The present research investigated the impact of NOM fractions on the chemical cleaning performance of aged PVDF based ultrafiltration membranes. Membranes, aged in the laboratory with equivalent cleaning agent exposure doses to membranes operated at full-scale were considered. Cleaning performance was quantified based on rate and extent of recovery of permeability during chemical cleaning of fouled membranes. The membranes were fouled using a bench scale system that repeatedly cycled membranes between filtration and hydraulic backwash (air-assisted backwash in this study), mimicking full-scale operation. Filtration was performed with a synthetic feedwater of model foulants containing bovine serum albumin (BSA) and/or humic acid (HA) as surrogates for biopolymers and humic fractions respectively, in NOM. Chemical cleaning was initiated when the permeability of the membrane dropped to 35-40% of the permeability of the unfouled membrane. NOM fractions were observed to impact the cleaning performance of aged membranes. For HA, the cleaning performance was unimpacted by membrane age. However, experimental runs with BSA, demonstrated changes in cleaning performance with membrane age. These results indicate the need for devising age-appropriate chemical cleaning protocols in conjunction with relevant feedwater pre-treatment approaches.

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Steroid Hormone Micropollutant Removal with Single Walled Carbon Nanotube – Ultrafiltration Composite Membranes

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Micropollutants (MPs) including steroid hormones occur in wastewater and surface water at sub-µg/L concentrations, costing Europe of hundreds of billion dollars annually in healthcare [1]. Nanofiltration (NF) with significant energy costs fails to achieve the European guideline for hormone removal (~99%). Ultrafiltration membranes (UF) can remove hormones once effective adsorbents are incorporated in the UF structure [2]. Composite membranes with single-walled carbon nanotubes (SWCNTs) were studied at operational UF conditions to examine mass transfer limitation [2,3]. To obtain the SWCNT–UF, SWCNTs were deposited in the support structure of UF with the MWCO range of 3–100 kDa. A micro-crossflow system with 2 cm^2 effective membrane area was used in filtration experiments with 100 ng/L hormone solutions. Parameters selected to examine mass transfer include SWCNT loading, residence time, solution pH, hormone type (estrone, 17β-estradiol (E2), testosterone and progesterone), and organic matter (OM) type. Particularly, the OM interference with adsorption was examined with nine OM types with different characteristics. SWCNT–UF 10 and 100 kDa partially removed steroid hormones (35–75%) at a SWCNT loading of 2 g/m^2. Hormone removal increased with loading from 0.1 to 4 g/m^2, and but was independent of residence time in the range of 0.08–7.1 s. These results imply that adsorption occurred only at the most accessible adsorption sites of SWCNTs, where adsorption was fast and not limited by the residence time. When certain OM types were introduced in the feed solution at 10 mg C/L, E2 removal decreased from 35% to 5–20%. The strongest interferant is tannic acid (TA), a polyphenol. However, OM shielding was possible with a top UF membrane of compatible pore diameter and the OM interference was mitigated. In summary, partial steroid hormone removal is attained with the permeate-side incorporation of adsorbents, although the European guideline was not achieved, and the nanoparticles raise toxicity concerns.

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Thin film composite membranes for desalination and osmotic power generation: Rheologically modified supports and revised interfacial polymerization.

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Thin film composite (TFC) membranes show huge potential in terms of low reverse solute flux and high water flux for desalination via forward osmosis (FO) and renewable osmotic power generation via pressure retarded osmosis (PRO), however, they still require innovative modifications to achieve these goals. In this work, high performance TFC membranes have been successfully designed for FO and osmotic power generation. The newly developed TFC-FO membranes not only exhibit an excellent water permeability (A = 5.5 - 7.5 L m^{-2} h^{-1} bar^{-1}), but also overcome the bottlenecks of internal and external concentration polarization (ICP/ECP). This was achieved first by modifying the supports during their phase inversion either by creating surface patterns (up to 250 um high) or via non-solvent (NS) spraying. Patterned supports provided additional surface area to increase the water flux while NS spraying resulted in highly porous supports which significantly reduced the ICP. Secondly, the interfacial polymerization (IP) method to deposit a polyamide selective layer was also modified by using spin-coating to create a thin, less dense and hydrophilic top layer. Under lab-scale PRO power generation tests, the membranes could withstand trans-membrane hydraulic pressures up to 14 bar thanks to the unique backing support and exhibit a power density ranging from 7 to 12 W m^{-2} using seawater and 1 M NaCl as draw solutions. The support layers showed high bulk and surface porosity with a small structure parameter and excellent mechanical robustness, while the polyamide selective layer was physically and chemically modified using spin-assisted IP, choice of monomers, and post modification to achieve desired water permeability. The impressive water flux, mechanical stability, and attractive power density suggest significant potential of the newly developed composite membranes for effective desalination via FO and harvesting osmotic energy via PRO process.

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Achievement of stable PRO performance of Toyobo hollow fiber membrane under ultra-high salinity by controlling ICP effect

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In Pressure Retarded Osmosis (PRO) process, membrane active layer faces high salinity side, while support layer faces fresh water or low salinity side, in order to reduce internal concentration polarization (ICP) effect to ensure high net driving force across membrane for high water migration [1]. Here, ICP is in normally considered caused by either asymmetric structure of membrane wall or the non-perfection of active layer, which means the salt (NaCl) rejection does not reach 100 % so that salt may reversely diffuse through active layer to support region, as a result, salt continuously accumulates in porous substrate to build up ICP. In lab scale researches, ICP effect has been experimentally simulated by adding NaCl in feed side, and indicated by decrease of water flux and increase of reverse salt flux [2]. But ICP concentration has never been measured directly. Besides, such finite element analysis is limited by simplification of boundary condition, where only single salt is considered and at relative low pressure. In reality, water contains versatile ions, and their diffusion tendency across active layer increases under high applied pressure. Most of them are further stuck in porous layer due to porous tortuosity, together with those rejected by active layer from feed side. Accumulation of multi-electrolytes in porous support may become unneglectable impact on ICP in long term running. In a pilot scale PRO study, SaltPower achieves a stable performance of 10 inch commercialized membrane module under 250 g/L for 150 h, by controlling ICP effect. In this long term experiment, multi-electrolytes besides NaCl are considered. In addition, a simple method is built up to detect ICP effect directly utilizing the reversible properties of ion absorption. By this way, accumulation of more ions than NaCl in porous substrate is verified and evaluated.

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Synthesis of Ce-based Metal-Organic Framework Thin-Film Composite Membrane for Pressure Retarded Osmosis

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Pressure retarded osmosis (PRO) is an emerging platform technology to generate electricity through osmotically-driven membrane processes [1]. One of the major technical barriers to the development of PRO is the lack of membranes specifically designed to exhibit high permselectivity and resultant high power density [2]. Of the membranes available, polyamide thin film composite (TFC) membranes are the ones most used in these applications [3]. In this work, by incorporating amine-functionalized Cerium-based metal-organic framework (Ce-MOF) into the TFC selective layer, we synthesized PRO TFC membranes that have improved transport properties. The surface chemistry of Ce-MOF was modified to fabricate a novel amine-functionalized Ce-based MOF whose dispersion in the aqueous solution is better than those of pristine Ce-based MOF, leading to improved compatibility with the polyamide membranes. Amine-functionalized Ce-MOF was incorporated at different loadings (250, 500, 1000 ppm) into the selective layer of TFC membranes. The successful incorporation of amine-functionalized Ce-based MOF into the modified membranes was investigated by using XPS, TEM, and FT-IR spectra. When tested in PRO mode, the modified TFC membrane shows improved perm-selectivity because of the high compatibility of amine-functionalized Ce-MOF with polyamide chains. For example, the amine-functionalized Ce-MOF membrane (with 500 ppm amine-functionalized Ce-MOF) exhibits a water flux of 40 $l/(m^2{\cdot}h)$, higher than 28 $l/(m^2{\cdot}h)$ for the pristine TFC membrane, using a deionized water feed solution and a 1.0 M NaCl draw solution. Moreover, the reverse solute flux of the modified membrane decreases to about 6.7 $g/(m^2{\cdot}h)$ representing a 42% improvement in the reverse solute flux of this membrane in comparison to the pristine TFC membrane, which significantly outperformed compared to ones reported in the literature. This study provides a new way of designing membranes for the PRO process, showing great potential for the application of MOFs in the PRO TFC membranes to harvest energy from saline water.

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Forward Osmosis Coupled with Enzymatic Membrane Reactor for the Removal of Emerging Contaminants

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With the increasing population, demand for global freshwater resources is increasing at an unsustainable scale, and therefore cost-effective and feasible technological applications for the combined upcycling and reuse of water are essential. Significant amounts of wastewater are already discharged by agricultural, urban, and industrial sectors, causing pollution to the natural environment, which is of primary and increasing concern for human health. These reasons bring sustainable requirements for robust and efficient degradation of contaminants. Municipal wastewater treatment plants are using existing systems based on activated sludge, and removing the contaminant of emerging concerns (CECs) remains challenging. In order to deal with these challenges, this study proposes a two-step solution based on i) applying forward osmosis (FO) to concentrate the CECs, which is a promising technology for pre-treatment thanks to its low fouling propensity and operational and energy costs; and, ii) carrying out an enzyme-immobilized membrane application in order to degrade the specific CECs. The study presented here develops a general structure for wastewater treatment that can be used in municipal wastewater and resource recovery applications. To this end, an eco-friendly degradation of the CECs stream can be obtained, which has a promising potential compared to existing techniques.

Separation and purification of plastic waste pyrolysis oils using organic solvent nanofiltration

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For decades, mankind has been living in a ‘make, take, dispose’ economic model. Plastics, with their unrivaled properties and low cost, are an exponent of this way of living. While plastics production reached 311 million tons in 2014, it is estimated that today nearly 50% of the plastic waste in the EU is landfilled, roughly 25% incinerated and only 25% recycled. Since plastics persist in the environment for prolonged periods of time and incineration adds up to greenhouse gas emissions, recent European legislation calls for far higher recovery and recycling rates. Plastics recycling processes can be categorized under four main types, i.e. in-plant recycling, mechanical recycling, chemical recycling and energy recovery. In view of carbon circularity, mechanical recycling is in principle the preferred option, however this route is only applicable for clean plastics streams and retaining high product quality is often difficult. Therefore, chemical recycling back to chemical feedstocks is coming more and more into the picture [1-2]. The leading technology here is pyrolysis, where waste plastics are thermochemically cracked and a viscous pyrolysis oil comprising a plethora of hydrocarbons is obtained. This crude plastic oil offers a potential source of platform chemicals that have significantly more value than the crude oil itself. To recover these compounds from the complex plastic oil, cost-effective downstream separation and purification processes are of utmost importance. Within this study, OSN has been explored, using model test mixtures mimicking pyrolysis oils obtained from relevant waste plastic types such as mixed polyolefins and polystyrene. Various separation challenges were identified, including fractionation of paraffins and purification of styrene monomer. Commercial polymeric as well as (in-house modified) ceramic membranes have been systematically tested. This presentation will highlight recent results from this work, showing the potential of OSN for downstream separations within tomorrow’s chemical waste plastics recycling plants.

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Carbon molecular sieve membranes with integrally skinned asymmetric structure for organic solvent nanofiltration (OSN) and organic solvent reverse osmosis (OSRO)

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Organic solvent nanofiltration is an energy-efficient separation process that requires robust membranes with high stability in harsh environments. In this study, we developed free-standing membrane with integrally skinned asymmetric structure based on carbon molecular sieve (CMS) that synergize the advantages of stable carbon materials and porous polymer membranes [1]. The membranes were prepared using a polyimide of intrinsic microporosity (PIM), known as 4,4′-(hexafluoroisopropylidene)diphthalic anhydride (6FDA)-3,3′-dimethylnaphthidine (DMN), via a phase inversion technique. The prepared membranes were followed by carbonization, which preserved the surface porosity and finger-like porous morphology of the membranes after structural rearrangement. The effects of dope solution concentration, pyrolysis temperature, polymer porosity, and membrane thickness on separation performance were investigated. The membranes showed no swelling and high stability in strong acids, bases, and organic solvents, as well as an excellent rejection profile and reasonable permeance. The membrane pore size, molecular-weight cutoff, and performance were fine-tuned by controlling the pyrolysis temperature, dope solution concentration, and polymer porosity. In comparison to carbonizing nonporous 6FDA-m-phenylenediamine (mPDA), carbonizing porous 6FDA-DMN afforded a 10-fold higher permeance. We also extended the work by investigating the effect of various fillers on the structure, morphology, and OSN properties of the CMS membranes. Upon carbonization, we found that the presence of fillers can control the membrane's porosity, which further can be used to fine-tune the OSN performance. To the best of our knowledge, the developed membrane fabrication platform yielded one of the tightest, most robust, and highly solvent-resistant nanofiltration membranes reported thus far. The next step is to improve the permeance of CMS membranes for OSN.

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On the OSN transfer mechanisms of a set of linear organic solutes only differing in the polar groups at the end of the carbon chain

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Organic Solvent Nanofiltration still suffer from a lack of understanding of the transfer mechanisms of small organics. To tackle this issue, a set of 7 linear solutes of close molecular weight and structure, except the existence of one or two polar groups at the chain end side, have been selected. Two solutes have one polar group located at each chain end side: diethyl sebacate (258 g.mol^{-1}) and 12-oxo-dodecanenitrile (195 g.mol^{-1}) while the 5 others have only one polar group: 10-undecenal (168 g.mol^{-1}) , 10-undecenitrile (165 g.mol^{-1}), dodecanenitrile (181 g.mol^{-1}), methyl 10-undecenoate (198 g.mol^{-1}) and 10-undecenoic acid (184 g.mol^{-1}). 4 of these solutes are present in a hydroformylation media we have extensively studied aiming at the Rh catalyst recycling and the selective product extraction [1, 2]. Each solute was filtered (10-40 bar) in single solution in toluene (0.1 mol.L^{-1}) with a PDMS dense membrane (Pervap 4060, Sulzer). By fitting the experimental rejections with the solution-diffusion model and film model accounting for polarisation concentration (if any), the real rejection and the diffusion coefficient were calculated. A nice insight is given, at 10 bar, by plotting the real rejection versus the diffusion coefficient in the membrane. The solutes can be divided into 3 groups according to their structure: (i) those with 1 polar group (except 10-undecenoic acid), (ii) those with 2 polar groups and (iii) 10-undecenoic acid. While at TMP=30 bar, all solutes (except 10-undecenoic acid) merged in a single distribution. The discussion proposes an explanation of transfers based on the desolvated and unfolded solute orientation at the membrane entrance, supporting that the solute local properties play a significant role. Accordingly, the polar surface area of both end side groups is evidenced as a relevant descriptor to model the 6 solutes through a single linear trend (at a given pressure).

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Anti-fouling membranes for organic solvent nanofiltration (OSN): graft modified polybenzimidazole (PBI)

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Fouling is one of the major obstacles to successful membrane-based separations. In aqueous systems, fouling is well characterised and fouling reduction techniques are established. However, in organic solvents, membrane fouling is not well understood. In this work we manufactured organic solvent stable anti-fouling membranes by graft modifying polybenzimidazole membranes with linear poly(ethylene glycol) (PEG) and poly(propylene glycol) (PPG). The anti-fouling characteristics of these organic solvent nanofiltration (OSN) membranes were tested with colloidal silica particles in a range of organic solvents. PEG and PPG graft modification provided an effective method of reducing fouling. However, the anti-fouling properties were dependent on the interactions between the graft modifier and solvent, and were closely correlated to the hydrodynamic diameter of the grafting polymer in solution. In water, modification of PBI with hydrophilic PEG provided significant anti-fouling properties due to an anti-fouling steric layer on the surface of the membrane. However, hydrophobic PPG graft modifiers provided no anti-fouling properties due to the unfavourable interactions of water with PPG. Conversely, in polar organic solvents, hydrophobic PPG modifiers showed improved anti-fouling properties over an equivalent molecular weight PEG modifier due to preferential PPG-solvent interactions. In non-polar solvents, both the PEG and PPG modifiers had poor interactions with the solvent and provided minimal anti-fouling properties. Therefore, the choice of graft modifier for anti-fouling OSN membranes needs to be carefully selected based on the system solvent.

Modular concept for high saline river water desalination by LPRO and MCDI: Results of a pilot plant in Vietnam

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Membrane capacitive deionisation (MCDI) has shown many advances in different applications, however, a performance evaluation of combination between MCDI and other treatment technologies has not been widely reported. In this study, the performance of post-treatment MCDI with low-pressure reverse osmosis (LPRO) as a promising, combined technology for desalination was designed, tested and evaluated. The pilot tests were conducted in Can Gio, a mangrove region in a river delta affected by seawater intrusion and progressive salinization (total dissolved solids TDS = 1-30 g/L). The pilot plant consisted of a ultrafiltration (UF) pre-treatment (2x AQQA4, Weise Water), a LPRO (3x 2540-XLE) and an MCDI (C17, Voltea®). Results showed an SEC of 6.5 and 7.1 kWh/m³ for the production of two water qualities (TDS <1.5 g/L and <0.45 g/L) by use of the LPRO or in combination with LPRO+MCDI, respectively. Additionally, the combined system was compared with seawater RO (SWRO, 3x 2540-SW30) which showed a slightly lower SEC of 6.9 kWh/m³. However, an optimization of the LPRO+MCDI is still possible by adjusting operational parameters of the MCDI, better dimensioning of the pressure pump for LPRO as well as targeting lower feed salinities, which allows lower pressures for the upstream desalination. The salient advantages of the modular systems were the improved environmental footprint of the product water by supplying two different qualities, and adaptability to salinity changes in the river water showing the feasibility of an application of the MCDI at pilot scale.

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Characterizing low-salt-rejection RO membranes with the Kedem-Katchalsky model

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Low-salt-rejection reverse osmosis (LSRRO) is a newly developed membrane process for brine management. Recent studies demonstrated that LSRRO is capable of producing hypersaline brines with a moderate energy consumption compared to thermal brine concentrators. Understanding the salt-water separation mechanisms in LSRRO membranes is critical to optimize and further improve LSRRO technology. In this study, the Kedem-Katchalsky (KK) model is employed to describe salt transport in LSRRO. In contrast to the solution-diffusion model that has been widely used for membrane desalination processes, the KK model considers both advective and diffusive salt transport [1]. Additionally, the KK model allows the assessment of LSRRO membrane performance from experimental data through the determination of the phenomenological reflection coefficient and salt permeability. Specifically, by varying the exposure time of RO membranes to a chlorine solution, LSRRO membranes with different transport properties and salt rejections were obtained. The LSRRO membranes were then tested in filtration experiments using different feed salt concentrations (0.1 to 0.5 M NaCl) and various hydraulic pressures (14 to 33 bar). The phenomenological coefficients were extracted by fitting the KK model to the experimental data of salt rejection and water flux. Our results reveal that the reflection coefficient decreases and salt permeability increases as the exposure time of the membrane to the chlorine solution increases. Overall, our study demonstrates that the KK model can adequately describe salt transport in LSRRO membranes and thus can be used for the design of LSRRO systems.

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Fouling behavior and cleaning strategies of ceramic ultrafiltration membranes for the treatment and reuse of laundry wastewater

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One of the most promising strategies for maintaining stable water sources for on-site wastewater reuse is greywater reclamation, particularly laundry wastewater reclamation. This study proposes an efficient strategy for the pre-treatment of laundry wastewater, which reduces membrane fouling and improves flux recovery after membrane cleaning. The fouling behavior, organic retention, and flux recovery rates of ceramic ultrafiltration (UF) membranes were comprehensively investigated using synthetic laundry wastewater. Under identical applied pressure and temperature conditions, a fouling resistance that corresponded to the water permeate flux and normalized flux was explored. Total organic carbon (TOC) retention was also tested to investigate the feasibility of using ceramic UF membranes as an effective pre-treatment for laundry wastewater reclamation. Furthermore, four different cleaning strategies for the fouled ceramic membranes were systematically compared, including deionized (DI) water, alkaline, acidic, and combined alkaline and acidic chemical agents to provide an in-depth understanding of the potential recovery rates of the membranes relative to the initial state. The filtration and treatment performance of real laundry wastewater samples collected from a university student dormitory was compared with synthetic laundry wastewater. This work provided valuable information on fouling behavior and cleaning strategies that could advance ceramic UF membrane pre-treatment technology for sustainable laundry wastewater reuse. Despite the challenges associated with the organic fouling and the potential of incomplete flux recovery in engineered systems, our findings provide insights into fouling mechanisms and cleaning strategies that could enable the optimization of engineered wastewater reuse systems.

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Development and Application of Functionalized Boron Nitride Ceramic Nanofiltration Membrane for Semiconductor Wastewater Treatment

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Ceramic membranes with high chemical stability and durability can be used properly in the treatment of industrial wastewater with complexity and toxicity. In this study, we demonstrate the fabrication of nanofiltration (NF) membrane using functionalized boron nitride (BN). Functionalized BN under tannic acid treatment showed more hydroxyl groups and thinner sheet than those under acid treatment. Based on this, BN ceramic NF membranes were prepared through the surface modification of ceramic ultrafiltration (UF) membrane using BN treated with tannic acid. BN ceramic NF membrane was confirmed to be successful through physiochemical and structural characteristics analysis, and an average pore radius was found to be 1.6 kDa, which was significantly reduced compared to that of the pristine membrane of 6kDa. Whereas the performance of pristine ceramic UF membrane were investigated with 97% of total silica and 3% of dissolved silica rejection, the BN ceramic NF membrane showed 20% of additional rejection with pristine ceramic UF membrane permeate reaching total 23% rejection of dissolved silica in hybrid ceramic UF and NF membrane process. Organic matter were rejected 56.6% with pristine ceramic UF membrane, and totally 82.5% were removed in hybrid ceramic UF and NF membrane process. Through comparative evaluation of the removal potential of the UF or NF alone process and the hybrid process, our results indicate the promising removal potential of the hybrid process superior to the single process in soluble substances in industrial wastewater.

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Rigorous simulations of high performance membrane gas separations for hydrogen purification and carbon dioxide capture through Computational Fluid Dynamics (CFD)

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Membrane gas separations are considered today as one of the key process technologies for different industrial applications. The rigorous evaluation of the process performances with a given membrane material is based on a design methodology which has been validated for decades [1]. Basically, a 1D modelling approach where the membrane is assumed to be the unique mass transfer resistance, fits a large number of situations. Pressure drop effects and, in some cases, non isothermal conditions can be included. With the development of advanced materials showing breakthrough performances in terms of permeance and/or selectivity, accounting for the gas phase mass transfer resistance (i.e. concentration polarization) becomes absolutely necessary in order to achieve a rigorous computation of the process performances [2]. Simulation of membrane gas separation processes with concentration polarization effects has been already reported by some authors, most often through fluid phase correlations, and more recently through Computational Fluid Dynamics (CFD) [3]. We report here a CFD study (ANSYS Fluent software) of gas permeation, which offers a generic and rigourous simulation of pressure drop, mass transfer and non isothermal effects. A systematic comparison to the classical 1D approach is achieved, with two experimental set of data: hydrogen purification by a Pd membrane and natural gas upgrading by a zeolite membrane. A series of perspective offered by this strategy will be shown: - The precision and limitations of analytical expressions of pressure drop evaluation in tubular or hollow fiber membrane will be analysed - The set conditions for which concentration polarization effects can be neglected (comparison to the 1D approach) are identified - Based on the exact CFD evaluation of the lumen side Sherwood number, correction factors are provided for improving the precision of available mass transfer correlations under significant concentration polarization conditions.

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Assessment of Anisotropy and Connectivity of Microporous Membranes by Digital Reconstruction of Real 3D Structures by Computational Fluid Dynamics

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The progressive development of new porous materials with specific topological properties requires a precise characterization of the influence of system anisotropy. As pointed out in Kim et al. (1987) [1], anisotropy is not an absolute properties of a material, but strictly depends on the specific transport phenomenon considered. For example, a material isotropic with respect to molecular diffusion could be – and in general is – anisotropic with respect to other transport phenomena. In this work, an original and self-consistent systematic assessment of anisotropy and connectivity of 3D structures of real microporous membranes is presented. For this purpose, we introduce an Average Effective Diffusivity Tensor, whose components are evaluated by an appropriate set of computational fluid dynamic simulations in pure-diffusion conditions once reconstructed digital models of the desired real 3D structures. In particular, after a detailed explanation of the methodology and of the way to apply it systematically, we validate our approach by reproducing the values of tortuosity of an already well known isotropic structure, i.e., the face-centred cubic one (FCC). Afterwards, we analyse two artificial membrane structures of different orientations and two case-studies of digitally-reconstructed 3D real structures of zeolite membranes: the LTL zeolite and the CIT-5 one. As main results, we show the ability of the presented approach in characterising precisely the distribution of the structural internal voids in terms of tortuosity and connectivity tensor, anisotropy factor and the here-called preferential-direction factors, introduced to quantify the preference of diffusional directions expressed in terms of percentage. Our methodology represents an original and relatively easy way for diffusional anisotropy assessment that can be effectively applied not only to existing materials of interest, but also to design novel materials – in form of membranes, catalysts and adsorbents – with desired characteristics in terms of internal void distribution and facilitated mass transport.

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A parametric CFD study of hollow fiber membrane modules for hemodialysis

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Hemodialysis is a membrane-based process in which solute transport from the patient's blood to a rinsing solution (dialysate) occurs by diffusion and ultrafiltration. Devices used in hemodialysis are cylindrical modules filled with hollow-fiber membranes which allow the removal of toxic substances and metabolic wastes from the blood, but inhibit the passage of proteins and cells to the dialysate [1]. A predictive porous-media model of hemodialysis was developed and validated against experimental data [2]. Unlike previous literature models, it requires only basic membrane properties (hydraulic and diffusive permeabilities and reflection coefficients) instead of relying on empirically adjusted global mass transfer coefficients. The necessary porous-media characteristics, notably Darcy permeabilities and shell-side mass transfer coefficients, were obtained by combining theoretical results, CFD predictions for regular fiber arrays [3] and experimental data for commercial modules. A parametric analysis was conducted to assess the influence of different physical quantities and operating conditions. Simulation results for different solutes showed that clearance is affected, in decreasing order of significance, by the membrane's diffusive permeability (kM), the dialysate flow rate (QD) and the ultrafiltration flow rate (QUF). Doubling kM yields an enhancement in clearance of ~7% for urea and ~20% for B12 vitamin, while halving kM yields a decrease of ~14% for urea and ~32% for B12 vitamin. Changes in the flow rates affect clearance to a lesser extent: a 50% increase of QD yields a clearance increase of ~4−5%, while a ±10 mL/min variation of QUF with respect to the reference value of 10 mL/min leads to a clearance change of ±1.3% for urea and ±3.2% for B12 vitamin. The oncotic pressure in blood has almost no influence. Therefore, possible performance improvements, at least in terms of clearance, strictly rely on the development of a novel generation of membranes characterized by a significantly higher solute diffusive permeability.

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The role of complexity in the application of artificial intelligence for organic solvent nanofiltration

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Large, available data and the rapid improvement in computational power have alleviated the recent progress in numerical optimization models. This improvement is allowing significant enhancements in process and material design. Machine learning and deep learning – two subsets of artificial intelligence – allow optimization in hyperdimensional spaces where analytical solutions are too expensive. We recently showed how rational data aggregation and curation could drastically boost process parameter prediction even in niche cases, such as organic solvent nanofiltration (OSN). As a potentially sustainable technology, OSN has a promising future in the pharmaceutical and fine chemical industry for solvent and catalyst recovery or purification. This presentation shows how to use machine learning to predict inherent membrane parameters (for example, rejection or flux) using only molecular structural information. We directly use this molecular structural information of the solutes, solvents, and membranes. Our current models use more than 500 different chemical compounds in 10 different solvents and four different membranes. Using structural information for the machine learning models allows us to interpret which features of the solute, solvent, and membranes affect the rejection and flux. Using graph neural networks, we explain the modifying effect of functional groups, rings, or even single bonds and atoms in a molecule. As of today, our models have the lowest root mean squared error and highest fitting scores for predicting solute rejection in OSN. We are also working toward making our work accessible to everyone; therefore, we are currently hosting a free and open-access database (www.osndatabase.com). Today, this OSN Database contains the most extensive data collection related to OSN with more than 15000 datapoints. Moreover, some of our models are also available at no cost on the website.

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Effect of microfibres on membrane biological reactor (MBR): separation and membrane fouling

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The concern about the occurrence of natural fibres such as cotton and wool in wastewater has increased newly. While microplastics are a booming field of study, natural fibres coming mainly from the clothes washing are really present as well [1]. Above 95% of microfibres (µFB) are removed in wastewater treatment plants, but the huge volumes processed cause that they could still appear in effluents [2]. Membranes technology has been proved to separate them, but several troubles such as membrane fouling and microfibres appearance in the permeate have been reported [3]. The current study was carried out introducing denim cotton µFB into a synthetic wastewater. The laboratory plant consisted of a 35 L membrane bioreactor treating a constant feed with a F/M ratio of 0.2 g COD·g $SS^{-1}\cdot d^{-1}$. The system included one compartment where an immersed A4-size flat sheet Kubota membrane was introduced (nominal pore size: 0.4 µm; effective membrane area: 0.11 m^2). The influence of 2 $mg\cdot L^{-1}$ of µFB in the feed on both biological process and membrane performance were studied. µFB distribution between permeate and mixed liquor and membrane fouling have been analyzed. Results showed the presence of a range of 105 and 250 $\mu FB\cdot L^{-1}$ in the permeate when denim cotton was added to the synthetic wastewater. Additionally, both cleanings in place and external cleanings suggested by the manufacturer were needed to solve operational difficulties, but inadequate permeability recoveries were obtained. FESEM images of the membranes were acquired as well to better understanding of the existing fouling. Additionally, organic matter elimination was characterized. Biological degradation reached 90.04±1.31% and the whole process achieved 94.33±1.67% COD elimination before µFB addition, whereas those percentages decreased to 84.53±6.83% and 91.51±1.80% respectively after cotton µFB were introduced in the system.

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A novel granular anaerobic osmotic membrane bioreactor for high quality wastewater reuse and energy production: proof of concept, potential and challenges.

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Anaerobic membrane bioreactor (AnMBR) is rising attention for domestic wastewater treatment thanks to the production of high quality permeate and biogas production towards energy positive treatment [1]. Granular sludge based anaerobic membrane bioreactor (G-AnMBR) has gained emphasis in the last decade since granular biotechnology boosts the biomass activity and reduces membrane fouling simultaneously [2]. With the aim to further reduce energy costs, produce higher quality effluent for water reuse applications and improve system efficiency, forward osmosis (FO) system was integrated to a G-AnMBR. Kubota microfiltration modules were step by step replaced by FO Submerged FO plate and frame modules. Process stability, impact of salinity on biomass, produced water quality and organic matter removal efficiency were assess and compared for system working in 100% MF, 70% MF/ 30% FO, 30% MF/ 70% FO and 10% MF/ 90% FO respectively. In this study, 3 plate and frame FO&MF 0.1 m^2 submerged modules were used in a 15 L anaerobic reactor containing about 85 g of dry granular biomass per liter. Synthetic wastewater was used as feed (COD content 500 mg/L), with hydraulic retention time of 10h and operated at 25 ºC. The system was operated neither with gas scouring nor relaxation at around 5 L/m^2/h permeation flux during at least 10 days. Above 90% COD degradation was observed for all configurations and with a remaining COD content below 50 mg/L and below detection limit for MF and FO permeates respectively. Interestingly also, thanks to the use of TFC FO membrane and novel module designed developed, FO was operated with a low salinity draw solution (13 g/L sea salts) and allowing for FO draw recovery at lower energy than former studies with the potential to produce excellent water quality suitable for all water reuse applications and which could be easily post treated for drinking water production.

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Membrane Bioreactors to Produce Polyhydroxyalkaonate (PHA) from Gaseous Feedstocks

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Polyhydroxyalkanoate (PHA) is a promising alternative to petroleum-derived plastics due to their comparable physical and chemical properties and biodegradability. Many microorganisms can produce PHA as an intracellular energy and carbon storage material. Microorganisms such as Cupriavidus necator can metabolize CO_2 as a carbon source and produce PHA when a mixture of H_2, CO_2 and O_2 gas is supplied. Thus, it is possible to produce PHA directly from CO_2 which would reduce greenhouse gas emissions. However, the optimum gas composition ratio for cell growth is 7:2:1 for H_2:O_2:CO_2 which is within the gas-explosion range [1]. To eliminate the explosion risk, the oxygen concentration should be maintained below the lower explosion limit however this limits the growth and productivity due to oxygen limitation. Furthermore, gas fermentation faces substrate limitation due to the low solubility of gases in the culture medium. Membranes have the potential to achieve high gas transfer efficiencies at low gas supply rates due to the high specific surface area available for transfer. Thus, membrane bioreactors are promising reactor systems for gas fermentation processes. This project demonstrates the applicability of membrane bioreactors to deliver gaseous substrates to produce PHA. To accomplish this, membrane bioreactors were constructed using 50 ml centrifugal tubes and gas-permeable polydimethylsiloxane membrane fibres with 760 µm outer diamater. The obtained membrane bioreactors are operated in batch mode where membranes are used for supplying gasesous substrates to maximize yield. The specific surface area for gas transfer is adjusted by optimizing the number of fibers used. The effect of operational conditions such as recirculation and gas flowrate is studied. Later, a fed-batch strategy is used to maximize PHA production by applying nitrogen limitation. PHA content within the biomass is increased from 8 ± 2% to 22 ± 2%. This is expected to be increase further by optimizing the conditions.

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The Temperature Effect on Fouling in Anaerobic Membrane Bioreactor: Roles of Extracellular Polymeric Substances and Soluble Microbial Products

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Biofouling is the main challenge of anerobic membrane bioreactors (AnMBR) operation, affected by various environmental conditions including temperature. Here, we explored the effect of two operational temperatures, 35 °C and 25 °C, on the AnMBR performance, delineating the interactions of soluble microbial products (SMP) and extracellular polymeric substances (EPS) with the membrane surface and their associated viscoelastic properties. We hypothesized that membrane performance at different temperatures (with similar biological performance) will be differentially affected due to different SMP and EPS properties, causing membrane biofouling. AnMBR operation showed a drastic membrane blockage after 11±2 and 18±2 days at 25 and 35 °C, respectively. Although the AnMBR biologically performed similarly at these temperatures (DOC removal, ORP biogas composition), by changing the hydraulic retention time and maintaining similar permeate flux (changing membrane surface area), more significant biofouling indicatives were detected at 25 °C versus 35 °C: (i) confocal laser scanning microscopy (CLSM) analysis showed significantly higher biofilm amount; (ii) quartz crystal microbalance with dissipation monitoring (QCM-D) showed more adhesive, viscous and elastic EPS layer, and (iii) Ex-Situ filtration experiments with SMP and EPS extracted from both the mixed liquor suspended solids (MLSS) and the membrane surface showed faster membrane fouling with EPS rather than with SMP. Our main conclusion, corroborate with our previous aerobic MBR studies: The cohesion of EPS originated from MLSS and its viscoelastic characteristics are critical for AnMBR fouling behavior, likely due to effects on flocs integrity at the AnMBR shear induced environment. As such, EPS layer produced at 25 °C, was more fluidic and accessible to the UF membrane pores in the AnMBR than EPS produced at 35 °C. Future analysis of the microbial communities structure composing the AnMBR flocs and the membrane biofilm will clarify the above differences of EPS properties and the consequent membrane fouling at different temperatures.

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A Reverse-Selective Ion Exchange Membrane for the Selective Transport of Phosphate

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Specific ion selectivity is a highly desirable feature for the next generation of membranes. However, existing membranes rely on differences in charge, size, and hydration energy, which limits their ability to target individual ion species. Here, we demonstrate a nanocomposite ion exchange membrane material that enables a reverse-selective transport mechanism that can selectively pass a single ion species. We demonstrate this transport mechanism with phosphate ions selectively transporting across negatively charged cation exchange membranes (CEM). Selective transport is enabled by the in-situ growth of hydrous manganese oxide (HMO) nanoparticles (NPs) throughout a CEM that provide a specific diffusion pathway via phosphate-specific, reversible outer-sphere interactions. Upon incorporating the HMO NPs, the membrane's phosphate flux increased by a factor of 27 over an unmodified CEM, and the selectivity of phosphorous over sulfate, nitrate, and chloride reaches 47, 100 and 20, respectively. By pairing ion-specific outer-sphere interactions between target ions and appropriate NPs, these nanocomposite ion exchange materials can in principle achieve selective transport for a range of ions.

Evaluation of the Photostability of Polymeric-Based Photocatalytic Membranes Using Accelerated Ageing

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The limited robustness of photocatalytic membranes (PM) to light exposition requires reliable methodologies for PM photostability evaluation. Long-term application can be simulated in a laboratory environment using accelerated ageing and, combined with suitable characterization techniques, gives parameters to address the PM photostability through the support membrane and photocatalyst evaluation [1]. Three combinations of support membrane (polyethersulphone–PES; polyvinylidene fluoride–PVDF) and photocatalysts (titanium dioxide–TiO_2 [2]; palladium-porphyrin–PS [3]) were aged in an irradiation chamber. The simulation of six months of PM use was obtained with the exposition up to 250 h under intense light irradiation (365 nm-981 W m^{-2}, 405 nm-1242 W m^{-2}). Changes in aged membranes were evaluated by several methods, such as visual observation, scanning electron microscopy (SEM), and UV-Vis spectroscopy. Methylene blue (MB) was employed as a model contaminant to evaluate the photocatalytic performance of aged PM in a photocatalytic membrane reactor [3]. UV-Vis spectroscopy results of PS-PVDF exhibited a significant reduction of characteristic porphyrins absorption bands with irradiation time, evidencing the photobleaching. SEM surface images of aged TiO_2-PES revealed damage to morphology with pore structure collapse. Photocatalytic experiments with MB showed that aged TiO_2-PVDF preserved the MB degradation efficiency, unlike TiO_2-PES and PS-PVDF membranes. In summary, the results revealed that accelerated ageing could simulate real applications of PM. The inorganic photocatalyst evaluated (TiO_2) had superior stability compared to porphyrin, and PVDF membrane support was less affected by ageing than PES. Overall, the adequate assessment of PM photostability must evaluate the modifications of each PM component (support membrane and photocatalyst) as well as the general photocatalytic performance.

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pH-Responsive Complexation of Polyelectrolytes: A Strong Promise Towards Green Synthesis of Membranes

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Hollow fiber (HF) membrane geometry is the preferred choice for most commercial membrane operations. Such fibers are conventionally prepared via the non-solvent-induced phase separation (NIPS) technique, which heavily relies on hazardous and reprotoxic organic solvents such as N-methyl pyrrolidone (NMP). A more sustainable alternative, i.e., aqueous phase separation (APS), was introduced recently that utilizes water as a solvent and non-solvent; this technique was deployed to prepare polymeric HF membranes for the first time via a pH gradient. The dope solution comprising poly(sodium 4-styrenesulfonate) (PSS) and polyethyleneimine (PEI) at high pH along with an aqueous bore liquid is pushed through a single orifice spinneret into a low pH acetate buffer coagulation bath. This results in PEI becoming charged resulting in complexation with PSS. The compositions of the bore liquid and coagulation bath were influential on the structure and performance of the HF membranes. Tampering with the acidic bath concentration led to different precipitation rates and final products; microfiltration membranes (permeability ~500 to 800 $L \cdot m^{-2} \cdot h^{-1} bar^{-1}$) with complete retention of emulsion droplets for slower rates and ultrafiltration membranes (permeability ~12 to 15 $L \cdot m^{-2} \cdot h^{-1} bar^{-1}$) having molecular weight cut-offs in the range of ~7.8–11.6 kDa. The research follows previous work on flat sheet polyelectrolyte membranes using APS, and is one further step towards commercialization of the green and efficient membrane production process.

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Polyelectrolyte Complex Hollow Fiber Membranes Prepared via Aqueous Phase Separation
Muhammad Irshad Baig, Mehdi Pejman, Joshua D. Willott, Alberto Tiraferri, and Wiebe M. de Vos
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Study of ammonia transport in Perfluorosulphonated Aquivion® mambranes

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Ammonia is a key chemical component for various relevant applications, but over the last few years it is attracting even more interest due its promising prospects as an energy carrier. Many studies, indeed, have recently attempted to find more efficient and environmentally benign processes for its synthesys starting from sustainable raw materials such as green hydrogen [1]. In this context, perfluorosulphonated membranes can play a role both as base materials for polymer electrolite membranes for low temperature electrochemical ammonia synthesis and as membrane for effective product separation. The characterization of Ammonia transport properties in such materials is therefore of great importance to deeply undisclose the real potential of their use in such processes, and truly understand the gas transport behavior. Unfortunately, only little experimental data exists of ammonia in perfluorosulphonated membranes, mainly related in Nafion [2], while other PSFA have been somewhat ignored for such application. Aiming to partially fill this gap, the present study reports the results of a series of permeation tests carried out on Aquivion (short side chain perfluorosulphonic acid) with pure ammonia and its mixtures with nitrogen and hydrogen. Test were performed at different temperatures and at relative humidities ranging from 0 to 80% in order to assess the influence of these parameters on the different gas permeabilities. Due to its highly hydrophyilic character, indeed, Aquivion switches from a diffusion based to a solution based selectivity at increasing RH [3].

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Optimization of Coagulant Dosage and Pre-treatment Configuration to Minimize Ultrafiltration (UF) Fouling

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Coagulation/flocculation prior to ultrafiltration (UF) may be optimized for the reduction of fouling or removal of natural organic matter (NOM). However, the selection of optimal coagulant dose and pre-treatment configuration applied prior to UF remains a major knowledge gap. Three main coagulation mechanisms may be defined as: i) adsorption destabilization, ii) combined adsorption and sweep, and iii) sweep coagulation. Coagulation, which includes coagulant addition and rapid mixing, may be followed by flocculation to increase particle aggregation and floc size. While evidence suggests that both coagulation mechanism and pre-treatment configuration (coagulation only, coagulation + flocculation) impact floc properties, their combined impact on floc properties and subsequent UF performance has not been clearly defined. This study evaluated the impacts of coagulation mechanism and pre-treatment configuration on UF performance for two source waters using a bench-scale continuous-flow coagulation/flocculation-UF system. Jar tests were performed to identify optimal pH and alum dosages with respect to each coagulation mechanism in terms of NOM removal. During UF trials, coagulation (G = 350 /s, HRT = 2 min) and coagulation + flocculation (G = 20 /s, HRT = 12 min) were utilized to continuously produce feed water. Results to-date indicate that the combination of adsorption destabilization (pH 5.5, 2.5 mg/L alum) and coagulation + flocculation affects the properties of floc ≤1 μm in size, increasing hydraulically reversible/irreversible fouling resistance, when compared to no coagulant addition, while providing similar NOM reduction. Enhanced particle aggregation and floc growth for a combined coagulation mechanism and coagulation + flocculation is anticipated to result in optimal UF performance by simultaneously increasing cake layer permeability and NOM removal. Guidance regarding coagulant dose selection (favouring a specific coagulation mechanism) and pre-treatment configuration could be readily adopted by the water treatment industry. Quantification of floc properties provides evidence regarding the mechanisms which dictate optimal performance.

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Optimization of phosphate/kaolinite microfiltration membrane using Box-Behnken design for treatment of industrial wastewater

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The present work reports the preparation and characterization of flat ceramic membrane via dry pressing method made from natural phosphate as based material and kaolinite as additive to enhance membrane characteristics. Response surface methodology based on Box–Behnken design was used to optimize the preparation conditions notably amount of kaolinite (5-15 wt.%), sintering temperature (900-1000 °C) and time of sintering (2-4 h). The optimized membrane has 40.2 MPa of flexural strength, 41.3% of porosity, 1045 L h^{-1} m^{-2} bar^{-1} of permeability and 0.35 μm of average pore size. The phosphate/kaolinite was subjected to the filtration of industrial textile wastewater and it showed excellent performance as microfiltration membrane. It is able to remove 98.99% of turbidity, 69.39% of total organic carbon, 74% of chemical oxygen demand and 77.11% of biological oxygen demand.

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Removal of glyphosate and AMPA by ultrafiltration membrane integrated polymer-based activated carbon

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Glyphosate (GLY) and its main metabolite aminomethylphosphonic acid (AMPA) have been reported to cause human diseases, including cancer. The detected concentration of GLY and AMPA in ground and surface water ranged from 0.2 to 400 µg/L [1]. Meanwhile, the current limit of GLY (as a pesticide) for drinking water is 0.1 µg/L (according to EU guidelines for pesticides). Conventional water and wastewater technologies have not shown promising removal of GLY and AMPA [2]. Therefore, this study focuses on evaluating ultrafiltration membrane incorporated with polymer-based activated carbon (UF-PBSAC) to remove GLY and AMPA in the low-pressure filtration process. A composite of UF membrane (10 kDa PLHGC, Millipore Corp., USA) with PBSAC (Blücher Co., Germany) placed in the permeate side is prepared in a stirred cell system [3]. PBSAC was evaluated for GLY and AMPA removal by static adsorption experiment. The experiments were performed with feed solution GLY and AMPA at concentrations of 1 µg/L. The filtration was performed through the UF-PBSAC in dead-end mode. 95 % of GLY and 57 % of AMPA were removed from a 1 µg/L (each) mixture of GLY and AMPA by PBSAC 0.5 g/L. Different PBSAC parameters and water quality were determined for adsorption characterization. Higher GLY and AMPA adsorption capacities were observed with PBSAC at higher activation levels, and lower oxygen contents, and at lower pH conditions. 75 % of GLY and 50 % of AMPA were removed from 1 µg/L feed by a thin layer of UF-PBSAC. The saturation was not achieved due to the tremendous adsorption capacity of PBSAC. Higher GLY adsorption capacities were observed with lower fluxes (<200 L m^{-2} h^{-1}). In conclusion, UF-PBSAC could remove GLY and AMPA but could not reach EU guidelines for pesticides. Further research is being proceeded to improve the adsorption performance of UF-PBSAC towards GLY and AMPA.

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Modification of Polyvinylchloride membrane by Poly(Terephthalic-co-Glycerol-g-Fumaric Acid) copolymer nanoparticles as pore formers

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Current study evaluates a poly(terephthalic-co-glycerol-g-fumaric acid) (TGF) as a water-soluble pore former nano-additive for the modification of a polyvinylchloride (PVC) ultrafiltration membrane. Membranes incorporated with hydrophilic (TGF) with loadings of 1 to 6 wt.% in the dope solution were fabricated aiming to improve the (PVC) membrane surface porosity and hydrophilicity. Modified membranes were characterized for detection the influence of varying the TGF content on their hydrophilicity, porosity, morphological structure, and composition, as well as subjected to ultrafiltration tests using feeds of pure water and bovine serum albumin (BSA) solution. The obtained results indicate that the porosity and hydrophilicity of the modified membrane prepared with optimum TGF content showed good enhancement. Lower contact angle was mainly a reflection of the improved membrane porosity. Furthermore, upon increasing the TGF content in the polymeric matrix, a more porous structure with longer finger-like micropores was formed, beside a sponge-like layer clearly. For optimum TGF content, a noticeable increment by 2-folds for the pure water flux, accompanied by an increase in BSA rejection up to 98 %. The findings demonstrate the efficacy of TGF as pore former for PVC membrane fabrication and modification; moreover the obtained separation behavior of the PVC/TGF membrane could stand as a promising choice for wastewater treatment applications.

Methods of manufacturing hollow fiber modules

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Depending on the diameter of the fiber, two module manufacturing techniques are commonly practiced: centrifugal potting and stationary potting. In stationary potting, the module is positioned vertically and the resin is injected into the bottom potting form where it hardens. Gel time of the resins used in this manufacturing technique is relatively long to guarantee proper infiltration of the adhesive between the fibers. Due to the presence of voids between the fibers, the capillary rising of the resin in stationary potted modules occurs, reducing the active length of the membrane fibers. The severeness of the capillary rising drastically increases for fibers with small diameter (e.g. dialysis, gas separation). To counteract the capillary rising effect, the centrifugal potting process was implemented. Here module is positioned vertically or horizontally in a centrifuge and is rotated during the potting process. After injection of the resin into the ends of the module, centrifugal force enforces flow of the resin between the fibers securing complete infiltration of the voids between the fibers. The centrifugal force can be adjusted with rotation speed and module length; thus, penetration of the glue is possible even for very small capillaries. Moreover, centrifugal force with opposite vector to the capillary force limits the severeness of the capillary rising. An application of the centrifugal force and quick potting process allows precise definition of the potting line and active length of the fibers. Me-Sep, under umbrella of INNOMEM projects developed a pilot line for centrifugal potting of hollow fiber modules of the industrial size (8”x100 cm). INNOMEM (Open Innovation Test Bed for nano-enabled Membranes) aims at developing and organizing a sustainable Open Innovation Test Bed (OITB) on membranes for different applications. The OITB will offer a network of facilities and services through a Singl-Entry Point (SEP) to companies.

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INNOMEM (Open Innovation Test Bed for nano-enabled Membranes) project under umbrella of H2020 framework

Development of an innovative PVDF based hollow fiber membrane with incorporated graphene oxide to combine ultrafiltration and adsorption

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In the frame of Graphil project, European project aiming to develop innovative graphene related material, Polymem has developed a PVDF hollow fiber composite membranes incorporating Graphene Oxide (GO) nanoparticles. This innovative membrane allows to combine suspended solids and virus removal with a UF cut-off and at the same time an adsorption of micropollutants thanks to the graphene oxide. This membrane could be used advantageously inside domestic and outdoor ultrafiltration cartridges. As the contact time of water and graphene oxide would be increased drastically if the GO flakes are small and perfectly dispersed in the porous structure of the membranes, a better adsorption performance is expected with this all-in-one composite membrane compared with the coupling of an adsorption bed followed by a filtration on membrane. To obtain such membrane, the dispersion and quantity of GO has been studied. A PVDF/GO hollow fiber has been prepared by a Nonsolvent Induced Phase Separation (NIPS) technic. GO nanoparticles dispersion has been studied and the combine effects of the stirring and the sonication of the dope solution investigated. Dope composition and membrane spinning parameters has been finely tuned to optimize the membrane characteristics and to fabricate membranes at industrial scale. Geometry (thickness, macro voids), filtration performances (pure water permeability, virus retention), adsorption performances (ciprofloxacin, PFAS and heavy metals removal) and stability (mechanical resistance, ageing tests) have been measured and the performances of these membranes evaluated.

Interfacial synthesis of PVBC-DABCO anion exchange membrane for vanadium redox flow batteries

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Generally, in redox flow batteries (RFB), membranes are developed as a porous or dense film through casting and subsequent solvent evaporation or phase inversion, respectively. Other common options are grafting or filling existing commercial membranes. Thinner films can be obtained by synthesizing the membrane interfacially, which is a fairly new concept in membranes for redox flow batteries. In this presentation, the interfacial reaction of a PVBC-DABCO membrane is presented, more in specific the synthesis conditions and its resulting performance. The membranes are optimized for their area resistance, their permeability, and their energy efficiency in the RFB set-up.

Efficient adsorption and conversion capability of bimetallic MOF-Modified Separator towards lithium polysulfides for Lithium-Sulfur Battery

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Lithium-sulfur (Li-S) batteries are one of the most promising next-generation battery systems due to their energy density of 2600 Wh kg-1, and low cost. However, one of their major technical problems is the shuttling of soluble polysulfides between electrodes, resulting in rapid capacity fading. An efficient separator which inhibits shuttling and preserves active material can vastly improve the performance of Li-S cells. Among many other materials, MOFs with micropore structure, open metal sites, and pore apertures (window) less than 6.9 Å are studied to be attractive to be used as separators. In this study, we present a bimetallic metal-organic framework (MOF) modified separator to alleviate the shuttling of polysulfide. The bimetallic MOF with two metal sites not only efficiently blocks/confines the sulfur species by Lewis's acid-base interaction but also effectively reutilized them to mitigate the polysulfides migration towards the anode side. When a sulfur-containing carbon material is used as a cathode material with a bimetallic MOF-modified separator, the Li-S battery exhibits a high-rate capability (1218 and 579 mA h g^{-1} are obtained at 0.1 C and 4 C, respectively), excellent cyclic performance with ∼76% capacity retention and 99% Coulombic efficiency at 0.5 after 300 cycles, placing bimetallic MOF modified separator one of the best polysulfides adsorption and conversion. Our approach demonstrates the potential for bimetallic MOF-based materials as separators for metal-sulfur applications. Furthermore, the ultrathin coating of bimetallic MOFs (in the order of a few microns) is under investigation for enhanced Li-S performance.

Acknowledgements

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A high power density and energy efficiency aqueous organic redox flow battery with optimized anion exchange membrane

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To address the ever-increasing greenhouse gas emissions, which are primarily caused by the release of large amounts of carbon dioxide when fossil fuels are used, researchers have been conducting intensive research on replacing those fuels with renewable energy sources, such as solar and wind. Renewable energies, on the other hand, generate intermittent electrical power depending on the time (day/night) and climatic factors. One strategy to address the intermittency is by coupling them with energy storage devices, such as aqueous organic redox flow batteries (AORFBs), which are regarded as promising electrochemical energy storage systems since they exhibit room temperature operation, long charge/discharge cycles and independent energy & power sizing. The membrane is a critical component of the AORFBs since it allows the transport of charge-carrier ions for the electrochemical reactions while providing a physical barrier between the two compartments in order to prevent the mixing of redox-active species and electrical short circuits. Herein, a series of anion exchange membranes (AEMs) were prepared and the correlations between the membrane properties and battery performance were investigated. Based on those findings, a membrane with optimized properties exhibited high capacity retention (94% after over 100 cycles), higher peak power density (293 mW.cm^{-2}) and energy efficiency (80%) than a well-performing commercial reference membrane was fabricated. In conclusion, the findings of this study provide insights guiding the development of advanced membranes to improve the efficiency and power capability of RFBs.

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Anion exchange membranes for the electrochemical reduction of CO_2

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The electrochemical reduction of residual CO_2 towards fuels and chemicals such as ethanol and ethylene is a promising route to lower our carbon footprint, especially when combined with renewable energy to drive the reactions. CO_2 reduction is mainly performed in membrane-containing flow cells consisting of electrodes on opposite sides of an ion exchange membrane in an alkaline electrolyte solution. The ion exchange membrane should facilitate the transport of ions between the anode and cathode while serving as a chemical barrier between the two compartments. When operating in an alkaline environment, anion exchange membranes are preferred because their positively charged exchange groups allow the transport of hydroxide ions from the cathode to the anode. Their main drawback lies in the poor chemical and mechanical stability at high pH and cross-over of negatively charged reaction intermediates. While extensive research has been performed on electrodes for CO_2 reduction, it remains unclear how the membrane affects the reduction and which membrane-type would be ideal. Therefore, we focused on commercially available membranes and analyzed their performance in terms of selectivity and efficiency for the CO_2 reduction reaction to use as a benchmark for further research. We aim to expand the current membrane technology by incorporating functionalized silica nanoparticles to improve hydroxide conductivity without sacrificing membrane selectivity and mechanical stability. By using wire-based electrospinning, a more homogeneous distribution between the organic polymer and silica nanoparticles is achieved, leading to better mechanical strength and conductivity compared to traditional casting methods.

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Sustainable porous membrane based on cellulose and soy protein for lithium-ion battery separator applications

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The food industry produces millions of tons of natural by-products. These by-products are often discarded, rather than revalue for social benefit. Through this study, we followed an environmentally friendly strategy using discards from the food industry, such as soy protein (SPI) and marine cellulose (Cel) from the agar industry for advanced applicatons. In particular, this work focuses on the development of a membranes based on soy protein and cellulose and their validation as a battery separator toward sustainable energy storage systems. The properties of these membranes are important in order to obtain lithium-ion batteries with high cycling performance and stability [1]. The main parameters affecting performance of he battery membrane are permeability, porosity, electrolyte absorption and retention, chemical, mechanical and thermal stability, which have been evaluated in the present work. SPI membranes with Cel show excellent compatibility through physical interactions. The FTIR spectra of the SPI/Cel membranes show absorbances associated with amide I (1628 cm-1) and amide II (1540 cm-1) as well as the absorbance at 1040 cm-1 associated with the C-O of marine cellulose, indicating physical interactions between the membrane components. These physical interactions favor the swelling of the membranes, improving the performance during electrolyte loading and unloading processes due to the good ionic conductivity, reaching swelling values of 1000% after three days in the liquid electrolyte. Analyzing the membranes by FTIR after being subjected to the liquid electrolyte, it is observed that the structure changes but the porous structure is maintained, while materials remain easy to handle. Furthermore, the membranes are thermally stable, showing an onset of thermal degradation at around 180 °C due to cellulosic compounds. The ionic conductivity value is above > 10-3 S.cm-1 and battery performance in cathodic half-cells is > 100 mAh.g-1.

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Purification and fractionation of polyphenols from wet olive pomace by means of organic-solvent nanofiltration

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Wet olive pomace, generated by the two-phase manufacturing process, is a concerning by-product derived from olive oil production. It is a semi-solid residue containing a high organic load as well as phytotoxic compounds, such as polypenols [1]. However, phenolic compounds are highly valued by industry due to their antioxidant and anti-inflammatory properties [2]. These molecules can be almost entirely retrieved from the wet olive pomace by means of an ultrasound-assisted solid-liquid extraction with ethanol/water 50:50 (v,v), during 45 minutes at 40ºC. In this process, some other undesired molecules are also extracted. Thus, a solvent-based ultrafiltration can be performed, obtaining a total solids rejection of 75%, whereas the polyphenols of interest are recovered in the permeate. However, this permeate still includes sugars, organic acids and free fatty acids that should be removed. Thus, a model solution of this ultrafiltration permeate was treated by organic-solvent nanofiltration, in the range of 15-36 bar, until a minimum volume reduction factor (VRF) of 3 was achieved. The NF270 membrane (Dow), as well as several solvent-resistant polymeric membranes, have been tested, including the NFS, NFX (Synder), oNF-1, oNF-2 (Borsig) and DuraMem 150, DuraMem 300, DuraMem 500, PuraMem 600 membranes (all from Evonik). The samples were characterized by liquid chromatography coupled to mass spectrometry. Sugars were determined by liquid chromatography coupled to a refractive index detector. These strategies allowed the evaluation of the individual rejection of each compound, whose variation with VRF was thoughtfully studied. Possible interactions between the solutes and the different membrane materials were also considered. NFX, DuraMem500 and NF270 membranes displayed satisfactory values of permeate flux. Also, high-added value compounds (such as hydroxytyrosol) were barely rejected, whereas high rejection values (50-100%) were observed for the unwanted molecules, such as sugars and organic acids. Thus, the permeate was greatly enriched in highly valued compounds.

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Lignin Oil Fractionation and Extraction of phenolic monomers by commercial OSN membranes: solvent choice and quantification of separation efficiency.

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Lignin oil is a biological source of phenolic compounds that are otherwise synthesized from fossil fuels. Phenolic monomers such as propyl guiacol can be used in the production of popular synthetic polymers, as well as specialty chemical such as food additives. [1] Lignin oil used in this study is depolymerized by reductive catalytic fractionation, using a Ru/C catalyst. The depolymerized lignin oil however contains a plethora of different molecules, making separation and purification of phenolic monomers challenging. An Organic Solvent Nanofiltration method, single pass or diafiltration, is proposed as primary fractionation method for the valorization of this lignin stream. The effect of two solvents was explored, i.e. methanol and ethyl acetate, selected for their physicochemical properties and sustainable character in this specific process. Fractionation efficiency is judged by the ability of separating oligomer, dimer and monomer groups, as well as their capacity to separate particular phenolic monomers, using Separation Factor as main quantifier. Characterization methods include Gel Permeation Chromatography and Gas Chromatography. Many different commercially available nanofiltration membranes were compared, with Borsig membranes GMT-oNF-1, GMT-oNF-2, GMT-oNF-3 and Evonik membrane Puramem s600 showing the highest potential for lignin oil fractionation. To purify these phenolic monomers however, hybrid systems or membrane cascades are advised. Furthermore, nanofiltration of phenolic compounds of near-identical molecular weight confirmed that molecular weight is a severely insufficient predictor of selective permeation.

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Preparation Membrane using Polybenzimidazole and Characterization for Organic Solvent Nanofiltration Application

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Separation and purification of organic solvents in the fine chemical and pharmaceutical industries plays an important role for environmental protection and sustainable development. Products are produced through many synthesis processes using strong organic solvents, and not only consumes a lot of energy but also incurs a lot of cost in disposing or recovering the organic solvents used in this process. As a method to solve this problem, an organic solvent nanofiltration membrane (OSN) is required, and many studies are being conducted. The organic solvent nanofiltration membrane must have mechanical properties that can withstand high driving pressure to separate organic solvents and chemical resistance that can withstand strong organic solvents. Therefore, in this study, an organic solvent nanofiltration membrane was manufactured using a polybenzimidazole (PBI) material with excellent mechanical properties and chemical resistance, and cross-linking was performed to further improve chemical resistance. Thereafter, the prepared membrane was subjected to chemical and physical property evaluation, and its performance as an organic solvent nanofiltration membrane was identified by determining the transmittance and removal rate.

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Utilization of biomass and green solvents for the fabrication of organic solvent nanofiltration membranes

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To address increasing environmental concerns, green and sustainable materials that promise to replace fossil-based separation materials, and reduce waste generation are attracting considerable research attention. Green solvents, biopolymers, and natural monomers are rapidly emerging as potential solutions to make membrane fabrication more sustainable. The challenge of sustainable membrane manufacturing is multifaceted and lies in finding sufficiently robust polymers that withstand harsh environments required during organic solvent nanofiltration (OSN), or finding reactive natural monomers that are soluble in green solvents with opposing polarities. Over the past few years our group has been exploring various green approaches and resources to fabricate nanofiltration membranes with solvent resistance. We have developed reversible crosslinking methodologies [1] and membranes with a closed-loop lifecycle [2]. Date seed biomass [3], abundantly available from the multimillion-metric-ton date industry, and chitosan obtained directly from shrimp-farming waste [4], have been successfully tuned into integrally skinned asymmetric and thin-film composite membranes, respectively. Antipathogenic upcycling of face mask waste into membranes using green solvents was successfully achieved [5]. Bio-sourced fully recyclable polyesters could open new avenues in the field because of the sustainable life cycle of the membranes via organocatalytic polymerization and organocatalytic depolymerization [2]. Overall, this presentation will cover the diverse approaches and materials we have been developing over the past years to make membranes greener.

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Hybrid Adsorption/Coagulation/Ceramic MF for pesticides control in drinking water treatment

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A powdered activated carbon (PAC)/Coagulation/Ceramic microfiltration (MF) pilot was operated in a water treatment plant during 18 Months, with water from different points of the treatment line. The optimization of PAC/MF operational performance was conducted and a new indicator was developed for performance assessment and optimization - the treatment capacity (TCp), i.e., the design flow rate normalized to membrane area and intake pressure [1]. TCp integrates all key-aspects of process productivity and therefore constitutes a useful indicator to balance flux, energy consumption and backwash and chemical cleaning frequencies. As expected, a higher TCp was obtained with filtered water, while similar results were obtained with the remaining waters. PAC addition (6-24 mg/L) did not promote membrane fouling and had no or a slight positive effect on TCp. Further, short-term pilot trials with 7.2–10.3 µg/L total-pesticides spiked to the waters were conducted to compare continuous PAC inline dosing with PAC dosing to a 2-h contact tank (~10 mg/L) [2]. Removal differences between the two PAC-dosing options depended on pesticides' amenability to adsorption and NOM characteristics (254 nm absorbance, A254). Waters containing low A254-absorbing NOM and only pesticides amenable to adsorption showed very high removals (all pesticides ≥ 93%) and no significant differences between the two PAC-dosing options. Waters containing higher A254-absorbing NOM and high loads of pesticides less amenable to adsorption (dimethoate, bentazone) required a higher inline PAC dose. Cost functions were developed and, for the operating conditions set and low-turbidity/low-NOM intake waters, yielded a MF cost of 0.04-0.07 €/m^3 for 100 000 m^3/d, including investment and operation costs. Moreover, the cost analysis showed PAC inline dosing is more cost-effective than PAC dosing to the contact tank when identical PAC dose is sufficient or when the doses are low, even if 50% higher for inline dosing, and the plant is small.

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Pore-filling microfiltration membrane with zwitterionic polymer brush provides ultrahigh flux with an excellent virus removal from water

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Membrane technology is one of the most attractive alternatives to water treatment with high virus removal. Here we developed and investigated zwitterionic (ZI) polysulfobetaine (polySPE) brush pore-filling polyethersulfone (PES) microfiltration (MF) membranes for high virus removal and low energy consumption. To this end, a 0.22 μm PES MF was pore-grafted with the polySPE brush with different SPE concentrations (0.1 M, 0.2 M, and 0.5 M) using a two-step atom transfer radical polymerization (ATRP). ATR-FTIR, XPS, CA, and SEM confirmed the successful modification. The virus removal and removal mechanisms were estimated using bacteriophages T4 (80 nm) and NT1 (70 nm), used as surrogates for pathogenic human norovirus. Remarkably, up to 4.7 and 3.3 log reduction values (LRVs) of T4 and NT1 were achieved, respectively, using the pore-filling membranes, while maintaining membrane permeability at few ~103 LMH·bar-1. The virus removal mechanisms by the MF membranes were investigated using a mechanistic mathematical model with the pore size distributions determined by a liquid-liquid porometer (LLP). It was found that the size exclusion is the main removal mechanism for pristine MF membranes, but that adsorption was dominant for the ZI-polySPE pore-filling membranes. μ-X-ray fluorescence mapping (μ-XRF) was used to study the polymer brush effects on location and amount of 100 nm Silica spheres after filtration under similar conditions. Here, to elucidate the effect of the polySPE brush morphology, a model surface with a mushroom-like polyZI grafted (nonbrush) via free radical polymerization was also studied. Quartz crystal microbalance with dissipation (QCM-D) and the μ-XRF results corroborated, showing higher adsorption of T4/NT1 and Silica spheres, respectively, to the brush surfaces than the PES and nonbrush surfaces/membranes. These results correspond to the bacteriophages removal results with the pore-filling brush membranes and support the assumption that the increased removal was due to electrostatic and hydrophobic interactions.

Electrically conductive ultrafiltration membrane for enhanced removal of dissolved organic matter from water

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Ultrafiltration (UF) is a promising technology for treatment of different waters. However, UF membranes are not capable to remove dissolved organic matter (DOM). One of the intriguing possibilities to improve retention properties for charged substances is the modification of UF membranes to make them electrically conductive. This way, an external potential can be applied onto the membrane surface in order to induce electrostatic forces. Depending on the applied potential, electrostatic attractive or repulsive forces towards the membrane surface can be induced. We propose a novel concept for enhanced removal of negatively charged DOM using an electrically conductive (EC) UF membrane. The EC membrane (ECM) was developed by thermal evaporation of Pt (20 nm) on ethylenediamine (EDA) functionalized UF polyacrylonitrile (PAN) membrane. When Pt-coated PAN-EDA was used for DOM removal in dead-end filtration, uncoated PAN-EDA membrane shows a DOC maximum removal of 69% at 25 L/(m^2·h) when feed with 12 mg/L DOM, 7 pH and 1 mmol/L NaCl was applied. This removal is mostly likely due to presence of the positively charge amine group. The anodic cell potential was applied when the intrinsic capacity of the membrane was fully utilized to further enhance the adsorption capacity of the EC UF membrane. The maximum DOC removal was 45% at 2.5 V anodic cell potential and same water flux of 25 L/(m^2·h). The size-exclusion chromatography coupled with organic carbon detection indicates major removal of humic substances. The DOC adsorption capacity due to intrinsic charge was calculated to be 230 mg/m^2. The enhanced DOC adsorption capacity due to external anodic cell potential was 155 mg/m^2, indicating an additional adsorption capacity of 67% due to electric field. Finally, an attempt was made to regenerate the EC membrane revering the cell polarity to negative. Currently, we are working on further optimization of the electro-assistance membrane process.

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Robust Photothermal Membrane via deposited f-MWCNTs on Porous Substrate for Water Recovery from Brine

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Brine is a high-concentrated solution of salts in water, mainly produced from desalination plants. There are limited thermal-based desalination techniques that can recover freshwater from brine with high intensive energy consumption because of the highly concentrated TDS. Herein, the f-MWCNTs were deposited by vacuum-assisted filtration, natural filtration, and Layer-by-Layer (LBL) deposition on common and cheap substrates to develop robust photothermal membranes with a strong ability to resist the hyper-saline water sources such as brine and recover freshwater. Different weights of f-MWCNTs (0, 0.1, 0.3, 0.5, 1.0 and 1.5 mg) were used to optimize the optimum weight of f-MWCNTs deposited on PVDF substrate (0.22 µm) for sun-driven evaporation. In addition, two kinds of commercial filtration fabrics (woven and non-woven) were used to be loaded with f-MWCNTs either by LBL deposition or natural filtration. The color of PVDF membrane using MWCNTs deepened from white (0 mg) to complete black with 1.0 mg MWCNTs uniform layer with thickness 295 nm, achieved light absorption efficiency reach 92 %, and evaporation rate 1.25 and 1.18 Kg.m-2.h-1 for 3.5% and 8.5% NaCl, respectively. The deposition of f-MWCNTs the f-MWCNTs on the top surface of commercial non-woven fabric substrates was investigated by vacuum filtration, natural filtration, and LBL deposition. The vacuum filtration was not effective due to the large pore size of non-woven fabric substrates. In contrast, the natural filtration using 8.0 to 12 mg of f-MWCNTs and LBL deposition technique using 3 mg/mL f-MWCNTs soaking solution for 24 h achieved promising results. The non-woven fabric with loading weights 8.0 and 10 mg of f-MWCNTs achieved 90% efficient light absorption. The woven fabric substrates were tested to deposit f-MWCNTs of the top surface with loading ranging from 0 to 8.0 mg by natural filtration. Moderate surface coverage was achieved due to the large pores of the substrate.

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Next-generation water purification systems using pressure-driven vapor transport across air-trapping membranes

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Current reverse osmosis membranes poorly remove low molecular weight uncharged solutes and are vulnerable to degradation from chemical oxidants used in water treatment. Here, we demonstrate the novel pressure-driven distillation process enabling desalination with ultrahigh selectivity and oxidation tolerance. This process operates similarly to reverse osmosis but uses hydrophobic air-trapping membranes with sub-100 nm pores. Under a hydraulic pressure exceeding osmotic pressure of feed water, a partial vapor pressure gradient is created across the membranes, allowing water molecules to evaporate on the feed side, diffuse through air gap, and condense on the permeate side. To operate the process, we realized a set of Janus membranes with an ultrathin superhydrophobic layer on the top of a thick hydrophilic aluminum oxide support. The membranes showed an exceptional water permeability of up to 10.5 $Lm^{-2}h^{-1}bar^{-1}$ and more than 99.8% rejection of sodium chlorine under hydraulic pressures of 45 bar, breaking the permeability-selectivity tradeoff that constrains polyamide reverse osmosis membranes for decades. The membranes are also highly selective towards problematic solutes permeating through polyamide membranes: boron, urea, and N-nitrosodimethylamine with greater than 98.1% rejections and showed unaffected performance after cleaning with 1000 ppm chlorine for 36 h and 25 ppm ozone for 1 h. Finally, we show that the separation strategy is scalable using large-area polytetrafluoroethylene membranes which exhibit more than 98% salt rejection even after exposing to 1000 ppm chlorine for 3 h to prevent fouling from biofilm-forming *Pseudomonas aeruginosa* bacteria. With high selectivity and oxidation resistance, pressure-driven distillation reduces the need of pre-treatment by microfiltration and post-treatment by advanced oxidation or ion exchange, allowing desalination and water reuse utilities to decrease system footprint and save significantly cost and energy consumption for water purification.

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Exploiting osmotic driving force for concentration and dewatering: Optimization, examples, and challenges

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The number of scientific publications around forward osmosis (FO) has been increasing exponentially for nearly 30 years. More recently, another osmotically-assisted process, namely, osmotic membrane distillation (OMD), has been growing in terms of interest and research outputs. FO and OMD have potentially unique advantages compared to competing membrane technologies owing to their low energy consumption and presumably low fouling tendency. Yet, these osmotically-assisted processes seem to have a hard time being adopted at the industrial scale and their reputation in the academic circles is passionately mixed. This talk will provide an account of some recent advances around FO and OMD, discuss ways to intensify the related processes, the limits of the technologies, as well as possible future directions of development in the field of water and wastewater management – and of liquid separation in general. Furthermore, it will present examples of applications and recent results obtained in the concentration of industrial liquid streams with the goal of aiding their safe management and with the aim to extract valuable resources. Specifically, results of the deployment of FO in the concentration of wastewaters from the energy production industry and results of the application of OMD in the dewatering of aqueous solution of phycocyanin will be presented and evaluated.

Modelling and Simulation of Membrane-Assisted Processes to Enhance Green Syntheses of Methanol and DME

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The lucrative utilization of CO_2 as chemical feedstocks is a challenging, yet major breakthrough toward the circular carbon economy. In this perspective, methanol and dimethyl-ether (DME), among others, seem attractive targets as energy carriers. The two chemicals are either liquid or possible to be liquefied with reasonable effort, and, with the state-of-the-art catalysts, they can be produced in one single reactor via so-called direct CO_2 hydrogenation. However, the presence of water vapor strongly inhibits the formation of the desired products in both reaction systems. Water-selective membrane reactors (MRs) were studied in the literature focusing on the enhancement of the one-pass conversion. However, their equipment level approach ignores a significant energy loss, at the flowsheet level, due to the undesirable co-permeation of H_2, which could otherwise remain on the reaction side at high pressure. Thus, the MR performance should be evaluated at the system level to monitor the overall efficiency. To that end, a rigorous and customizable model for MRs was developed in Aspen Custom Modeler. The model requires no analytical correlations for thermodynamic properties, which often fail to reflect the system behavior accurately, and is the first model that formulates the transmembrane process via an isenthalpic path while considering two-direction permeation. Using this integrated simulation framework, we identified the minimum required H_2O/H_2 permselectivity for methanol production at 190 and 970 based on the exergy analysis and power requirement, respectively. Therefore, the permselectivity of the most membranes described in the literature as suitable candidates for this application is well below the minimum requirement when the overall methanol synthesis process flowsheet comes into consideration. Moreover, for the direct DME synthesis, we present a comprehensive technical and environmental assessment at the flowsheet level. At 7.5 MPa pressure, a membrane-based process offers 1.5%, 44.5% and 69.4% savings in power, heating and refrigerant utilities, respectively.

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Two-dimensional ion transport modelling of water desalination by reverse osmosis system considering the real roughness membrane effect

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Standard technologies for water desalination, such as ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO), separate high concentrations of ions (e.g., Cl^-, Na^+, K^+, Mg^{2+}, Ca^{2+}) by pressing water through selective membranes. Modern composite membranes have an ultrathin polyamide active layer (20–200 nm) that performs the ion separation and support layers of polysulfone (20–50 µm) and polyester (50–150 µm). Current theoretical models, such as the solution-diffusion or solution-friction, explain the solute and water fluxes by computing concentration and potential gradients developed in one direction, namely perpendicular to the membrane surface. However, several experimental studies indicated that a relation exists between membrane morphology and its permeability and selectivity [1,2]. Only very few studies described the effects of polyamide membrane roughness on the RO process through two-dimensional (2-D) models [3], while also disregarding membrane charge effects, electromigration and possible convective ion fluxes, and the impact of real polyamide layer morphology. We developed a solution-friction 2-D model to describe the transport of multiple ions through an RO membrane with irregular polyamide active layer. The model considers the effect of active layer morphology (i.e., roughness, void fraction, variable thickness), all transport mechanisms (diffusion, convection, electromigration), membrane charge, and the water-membrane partition effects (e.g., Donnan, steric, etc.). We characterised the polyamide membrane structure through transmission electron microscopy (TEM) and scanning electron microscopy (SEM), and from these images characteristic model geometries were constructed. Model results showed the importance of membrane surface irregularity, correlating the salt and water fluxes not only with salt concentration and membrane charge but also with surface roughness descriptors. Furthermore, the model revealed the possible existence of internal ionic currents in the membrane and their effects on the separation process.

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Model development for dehydration of mixtures containing heterogeneous azeotropic

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The study is motivated by the industrial separation problem in the pharmaceutical and fine chemical industry, ethyl acetate and isobutanol removal from process wastewater. The separation of water and alcohol mixtures can be considered a well-known example of hydrophilic pervaporation operation in chemical and related industries. Ethyl acetate and isobutanol form heterogeneous azeotropic mixture with water. This research aims to rigorously model and optimise the separation of the mixtures in a professional flowsheet simulator environment. To complete this goal pervaporation membrane method is investigated and optimised [1]. Pervaporation is a relatively new technology, where the mixture to be treated is vaporised at low pressure on the permeate side of the membranes, and the separation of the mixtures progresses by preferential sorption and diffusion phenomenon of the desired component through the dense membranes. Vacuum pump on the permeate side can maintain the low vapour pressure. Pervaporation is capable of the separation of many organic aqueous systems. The unit operation is mainly used for dehydration of organic compounds from its aqueous mixtures, removal of low concentration organics from water and organic-organic separation. This unit operation has the specialities such as no-pollution and energy-saving, simply actualisation and high separation, which are difficult to obtain by other conventional technologies. The pervaporation measurements can be achieved in continuous and batch configurations [2]. The maximum recovery means the primary product goal in the case of batch pervaporation, and the retentate is considered as low-value (by-)product. These align with our goals and the environmental goals of treating process wastewaters. Many recent papers have analysed and optimised the modelling of pervaporation in continuous mode. Considering the simulation results, it can be determined that the batch configuration of pervaporation is also suitable for separating mixtures above azeotropic composition.

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Digitalization of dynamic membrane filtration systems – from digital shadow towards digital twins

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Digital Shadows/Twins have become synonymous with digitalization in the chemical and biochemical industry. The concept itself is not new and has existed within aeronautics since the 90s. However, it has taken decades for processing industries to incorporate it due to limitations on process monitoring and computing power. A digital twin has been defined as a system of interconnected physical assets and in-silico/digital representations with continuous information exchange, able to determine and implement open or closed-loop actions given by an optimal objective function [1]. In a digital shadow, communication is one-directional. In this regard, a platform has been designed to develop a digital shadow for dynamic membrane filtration systems. Dynamic filtration technologies are interesting due to the enhanced rejection and/or throughput required in challenging separations [2]. However, operation at maximum throughput is challenging to achieve under unmeasured high-variance inlet disturbances, forcing it to operate under suboptimal conditions. Therefore, a virtual representation of the plant could allow predicting the time-variant fouling rate, vital to defining an operation strategy. In previous research, the system dynamics is described using the Autoregressive with Extra Input model (ARX) employing the Forgetting Factor Recursive Least Squared method (FFRLS) for online system identification [3]. The model describes the transmembrane evolution at constant flux operation, and thus predicts the membrane fouling rate. Therefore, a platform for digital shadow has been created where information from the plant (PLC) is stored online in a PostgreSQL relational database server. Data can be retrieved in real-time through vNode as input to the virtual plant for model development and tuning (local machine). The model predictions have around 4% average error, capable of representing fast and slow system dynamics. This contribution is part of bigger efforts as modelling approach for dynamic ultrafiltration towards implementing adaptive digital twins.

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Multistage process for the separation of CO2 and CH4 by using biopolymer-based membranes

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The development of membranes specifically designed for the separation of carbon dioxide/methane mixtures from different sources has gained importance in concordance to the advances in membranes and process designs. Different types of membranes, from polymeric membranes, metal-organic frameworks, mixed-matrix membranes, carbon membranes, silica and zeolite membranes, to bio-based membranes more recently, have been reported for the separation of CO_2 and CH_4.The exploration of polymers derived from natural resources started before two decades ago. Nevertheless, the number of biopolymer membranes applied to CO_2 separation is limited, and further efforts are focused on bio-based materials for a step-forward towards an improvement of the overall technology in terms of sustainability [1]. Despite the efforts to improve performance and reduce the environmental impacts by the use of biopolymers as membrane materials, the previous process engineering tasks concluded that the separation performance in terms of purity and recovery would not be enough to meet the requirements for CO_2 and CH_4 direct use in a single stage [2]. Single-stage processes cannot provide both high product gas purity and high recovery at the same time, but multistage processes do [3]. Therefore, the aim of this work is the simulation and optimization of a multistage process, considering up to three stages, for the separation of CO_2 and CH_4 from different sources, using ionic liquid-chitosan mixed matrix composite membranes. The targets are defined in terms of purity and recovery of CO_2 in the permeate outlet (up to 95%) and CH4 in the retentate outlet stream (95% purity and 98% recovery) of the multistage process. The decision variables include membrane related, as the permeability of a component or the selectivity (CO_2/CH_4), and process related, as the stage-cut of each stage, also determining the membrane area required for a techno-economic evaluation.

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Combining Computational Fluid Dynamics, Machine Learning, and Physics-based Models for Intensifying the Modeling of Membrane Processes

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This work demonstrates a multi-scaled modeling approach that was developed for bridging the gap between fundamental physics-based models [1] and system-scale models for real-world analysis that can be applied to multiple types of membrane systems. Modeling membrane processes often demands a trade-off between simulation speed and fidelity when the focus of these models shifts from micro-scale to system-scale investigations. Computationally investigating fouling, hydrodynamic, and mass transport behaviors that occur at the micro-scale requires intensive and high-fidelity computational simulations that are not suitable for rapid or high-volume investigations. To intensify current system-scale modeling approaches, experimental and computational fluid dynamics (CFD) simulation data have been paired with machine learning software to produce surrogate models for predicting the water recovery and energy consumption of membrane systems. Experimental data was captured from an engineering-scale pressure retarded osmosis system to confirm a new solution-diffusion with defects model [2] and quantify the impact of defects on the membrane surface on water recovery and contaminant rejection. The data captured by CFD simulations focused on how membrane spacer designs impact pressure loss and concentration polarization. Turbulence-promoting spacers are an integral component of most membrane systems for providing both structural integrity and increased mixing for maximizing water recovery. However, existing models don’t account for most of the geometric characteristics of spacers when quantifying the impact they have on pressure loss and concentration polarization. The data collected from CFD simulations was used to train neural networks and produce a surrogate model to replace existing physics-based or empirically derived models. These two surrogate models have been implemented in a multi-scale modeling framework, used for system-scale analysis, to demonstrate how surrogate models derived from high-fidelity investigations can be used to improve the accuracy, speed, and flexibility of more lightweight modeling software [3] for predicting water recovery and energy consumption of membrane processes.

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Photocatalytic membrane reactor vs. membrane distillation unit for treatment of surface water contaminated with ketoprofen

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The aquatic environment is contaminated with a wide range of the so-called contaminants of emerging concern (CECs), such as pharmaceuticals and personal care products (PPCPs), or endocrine disrupting compounds (EDCs). Several studies have reported pharmaceuticals concentrations varying from ng/L to µg/L in surface and ground water worldwide. They are also present in effluents of municipal wastewater treatment plants or even in drinking water. The conventional water and wastewater treatment processes are inefficient in the removal of most of CECs. Since such trace contaminants pose a threat to human health and aquatic ecosystems, new efficient technologies need to be applied. One of the attempts can be the use of membrane distillation (MD), which attractiveness results from the non-requirement of external hydraulic pressure, possibility of application of highly saline feed, and, more significantly, low fouling propensity. Nonetheless, since the retentate contains a concentrated load of the rejected contaminants, including CECs, its further post-treatment is required. An alternative to that can be hybridization of MD with advanced oxidation processes, especially photocatalysis. Such systems are called photocatalytic membrane reactors (PMRs). They offer a continuous operation during which the recovery and reuse of the photocatalyst and the separation of various contaminants (including products and by-products of photodegradation) from the treated solution occur simultaneously. In the presented study, the performance of a submerged photocatalytic membrane reactor (SPMR) coupling direct contact membrane distillation (DCMD) and photocatalysis was investigated. For comparison purpose, single DCMD process was also applied. The aim of the research was the evaluation of the long-term performance of the SPMR and MD during removal of ketoprofen from a synthetic surface water. The changes of the composition of feed and distillate, and the membrane performance were especially analyzed. Moreover, the toxicity of the treated samples was assessed using the bioluminescence inhibition bioassay.

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Algae-sludge membrane bioreactors for wastewater treatment

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Membrane bioreactors (MBRs) have attracted a lot of attention as one of the key wastewater treatment techniques. However, their wide use has been limited by high aeration energy consumption and membrane fouling [1,2]. Algae-bacteria systems have received a lot of interest in recent years, because of their high nutrient reduction efficiencies, low-intensity aeration, and potential for biofuel production. In these systems, algal cells absorb CO_2 created by bacteria strains and emit O_2, which bacteria cells utilize to remove nutrients. The main aim of present study was to study the effects of the algal cells on MBRs efficacy and optimize the parameters. Four plexiglass reactors were designed for the municipal wastewater treatment. Chlorella vulgaris algae cells were inoculated into the algae-sludge membrane bioreactors (algae-MBRs) with different inoculation ratios. The flat sheet membrane modules with an effective surface area of 0.02 m^2 and a mean pore size of 0.2 µm polyvinylidene fluoride (PVDF) were used in the MBRs. The results of this investigation revealed that adjusting the algae-sludge inoculation. Membrane fouling and extracellular substances (EPSs) sharply reduced by introducing algal cells to MBRs. Furthermore, by observing the oxygen consumption trend and estimating the quantity of oxygen produced by algal cells, it was shown that it is possible to reduce the amount of mechanical aeration required in the MBRs without affecting the purification efficiency or increasing the membrane fouling.

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Photoredox-Catalyzed Decomposition of Nitric Oxide over the Photocatalytic MgO@g-C_3N_4/PES and TiO_2@g-C_3N_4/PES Membranes: Discussion on Mechanism and Stability

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Semiconductor photocatalysts are widely used for energy and environmental applications because of their high efficiency, cost, and eco-friendly. However, most photocatalyst in the shape of nanoparticles is a limitation for photocatalysts that can be applied in real work. Nowadays, photocatalytic membranes have shown potential in environmental pollution treatment and overcoming the limitation of nanoparticles. In this study, we take advantage of the photocatalytic membranes for removing Nitrogen dioxides under visible light. The polyethersulfone membrane (PES) has been used the subtract the photocatalytic membranes. The photocatalytic MgO@g-C_3N_4/PES and TiO_2@g-C_3N_4/PES membranes have been synthesized via plasma modification. The photocatalytic membranes achieved approximate 80% NO removal efficiency under visible light after 30 min. Furthermore, the DeNOx index, Apparent quantum efficiency, and NO conversion during the photocatalytic progress are invested in understanding the photocatalytic pathway of the photocatalytic membranes. Besides, the photocatalytic membranes show high stability after serval time recycling. In the scientific view, the characteristic of the photocatalytic members was analyzed by characteristic analysts such as Transmission electron microscopy (TEM), Diffuse reflectance spectroscopy (DRS), Electron spin resonance spectroscopy (ESR), etc. This work promises to enable future studies to be more accessible to apply in the environmental field.

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Modification of PES membrane with Cu@TiO$_2$ photocatalyst and PAA grafted binding layer for antifouling property

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Recent extensive studies on photocatalytic membranes have shown them to be potential antifouling membranes due to the special properties of the photocatalyst layer on the membrane surface. This layer of photocatalysts can reduce the water contact angle of the membrane surface and can degrade foulants under the irradiation of suitable light. However, there is a need to develop high-performance photocatalysts and improve the binding stability between the membrane surface and the photocatalyst. In this study, we developed a high-performance photocatalyst Cu@TiO$_2$. On the other hand, we used a plasma grafted PAA intermediate binding layer on the surface of the PES membrane to create a stable complex bond with the photocatalyst. Multiple -COOH functional groups of PAA will be complexed with titanium and copper metal ions on the catalyst surface. First, the TiO$_2$ catalyst was bandgap tuned by precipitation of copper metal nanoparticles onto its surface. The obtained photocatalyst Cu@TiO$_2$ has been characterized by FTIR, SEM, TEM, EDS, XRD, UV-Vis DRS, XPS, and ESR, demonstrating the successful synthesis of the photocatalyst with a decreased bandgap. The degradation of the dye Acid Blue 260 in the presence of the photocatalyst by photocatalysis and the activation of hydrogen peroxide occurs almost completely. The photocatalytic activity of the Cu@TiO$_2$/PAA/PES membrane for the degradation of Acid Blue 260 is about 91% and is almost stable over five consecutive cycles. The antifouling ability of this membrane is demonstrated by the flux recovery ratio of 98% when performing photocatalytic treatment of the membrane fouled by sodium alginate. Although the pure water flux of the modified membrane is lower than that of the pristine PES membrane, it is acceptable and still superior to other hydrophobic membranes. Therefore, the modified membrane has shown that it can be used as an antifouling membrane in the field of wastewater filtration.

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Modification of reinforced hollow fiber membranes with WO_3 nanosheets for membrane bioreactors

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Due to the rapid population growth in recent years, there has been a serious increase in water demand for domestic consumption and industrial applications. As a result of this increase, it is very important to protect water resources in order to avoid water shortages in the future. The use of membrane technologies has been increasing for the treatment of textile industry wastewater because narrow pore sizes of membranes can eliminate dye compounds and discharge high-quality effluent. The use of membrane bioreactors (MBRs) technology has gained global interest and application in wastewater treatment. MBR system produces less sludge, occupy less space compared to other biological wastewater treatment methods and adapt to more pollutant concentrations. However, reducing permeate flux results from membrane fouling during the wastewater separation is one of the main limitations during the membrane separation process. The aim of the study is to investigate the effects of using WO_3 nanosheets in performance of reinforced hollow fiber membranes to improve antifouling properties for applying in membrane bioreactor system. WO_3 nanoparticles show both hydrophilic and photocatalytic properties, enabling the membrane to decompose organic chemicals present in the environmental liquid waste. To achieve this aim, modified PVDF-UF reinforced hollow fiber membranes were fabricated by adding 0.02 and 0.1 wt% WO_3 nanosheets. The effect on the morphology, hydrophilicity, antifouling and dye removal efficiency of the membranes was investigated. The results showed that the overall porosity, hydrophilicity and permeability of the membranes increased with the inclusion of WO_3 nanosheets. Following long-term operation of tested membranes, two cleaning stages (physical and chemical) were applied to membrane modules in order to determine the contributions of different resistances to total filtration resistance. The WO_3 blended membranes showed good antifouling properties.

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Aqueous Phase Separation of Responsive Polymers for Sustainable Membrane Manufacturing

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Membranes allow us to improve the sustainability of various industrial processes. However, current membrane manufacturing processes heavily depend on the use of aprotic polar organic solvents such as dimethylformamide and N-methyl-2-pyrrolidone. These solvents have long been on a list of harmful toxic chemicals predestined to be banned from use in large scale. Annually over 50 billion liters of waste water contaminated with these solvents is produced from membrane manufacturing [1]. Here we present an innovative and sustainable water-based membrane fabrication process eliminating the need of for organic solvents, called aqueous phase separation (APS). In essence APS is a sustainable new manufacturing platform for membranes using polyelectrolytes as building blocks. This allows for new ways to functionalize membranes and control the membrane formation process. Within the APS framework there are two distinct ways membranes can be prepared [1]. Membranes are prepared by using pH responsive polyelectrolytes which are water soluble in either alkaline or acidic water and precipitated by a simple pH switch. Membranes are prepared from a solution of two oppositely charged polyelectrolytes where a complexation induced phase separation is triggered by a pH or salinity switch. In this work we will focus on two polyelectrolytes, poly(4 vinyl pyridine) (P4VP), a polybase, and the copolymer polystyrene-alt-maleic acid (PSaMA), a polyacid. P4VP membranes are prepared by dissolving it in acidic water and precipitating in a bath of alkaline water, while PSaMA membranes are prepared by using water and precipitation in acidic water. The membrane morphology and resulting separation properties can be tuned by various parameters such as pH, base/acid identity, salt concentration and salt identity. Open microfiltration up to dense nanofiltration membranes were prepared having good separation performances. Aqueous Phase Separation will make the membrane fabrication industry more sustainable by avoiding the use of toxic organic solvents.

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Support synthesis optimisation for supported liquid membrane (SLM) and polymer inclusion membrane (PIM) extraction of chiral amines

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Chemical companies are constantly aiming to improve the efficiency and sustainability of the current processes. Membrane-based solvent extraction (MBSE) is a promising technology, as it combines the strengths of two conventional separation methods, namely membrane technology and liquid-liquid extraction. Two types of MBSE can be distinguished, namely supported liquid membranes (SLM) and polymer inclusion membranes (PIM). Within SLMs, a porous support material is wetted with the extractant, whilst in PIMs, the extractant is immobilised within an entangled polymer matrix. Although both types have been studied significantly in literature, they still suffer from a low membrane stability and decreased fluxes. Furthermore, the current state-of-the-art does not provide comparisons between SLM- and PIM-performance for specific separation applications [1]. MBSE can be particularly useful for equilibrium-limited reactions, such as the synthesis of chiral amines, of which the yield can be improved from only 50 to over 98 %. However, the poor membrane stability hinders the full industrial potential of this technique. In this work, the effect of the support properties on the final membrane stability was investigated. Polysulfone (PSf) and polyvinylidene fluoride (PVDF) were selected as the polymer materials, as they can be used in both SLM- and PIM-configurations. The effect of various synthesis parameters, such as coagulation time and temperature, on the support morphology and hydrophobicity was investigated. It was found that by addition of a plasticiser and a PDMS-coating, the support hydrophobicity could be improved significantly. Furthermore, the wettability behaviour of the support materials for the selected extractant was quantified by contact angle measurements. The synthesised membranes were then compared to commercial membranes in terms of extraction performance and stability. The results obtained from this study enable to finetune the support properties in order to enhance the MBSE-performance, which could aid in the industrialisation of this technique [1,2].

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Interaction effects of non-ionic surfactants and solvents on PES and Cellulose membranes for Virus and Sterile Filtration process steps

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Surfactants are common excipients during plasma processing and play a role for long-term protein stability, adsorption prevention and virus inactivation (e. g. the non-ionic surfactants Triton-X 100 or Polysorbate 80 (PS 80, Tween® 80) for IVIG).[1] The interactions between such surfactants, esp. PS 80, and membrane material in filtration products range from swelling, flow rate reduction and Bubble-Point decrease due to adsorption. The investigation focuses on the adsorption of PS 80 on sterile filtration and virus filtration membranes. Differences in materials (Polyethersulfone (PES), Cellulosic, Polyvinylidene fluoride (PVDF), surface-modified materials) in terms of PS 80 adsorption and adsorption kinetics are observed which are compared the results to literature-known studies for PS 20 [2]. PES membranes are extensively used in the Biopharm industry due to their excellent properties with regard to filtration performance and their very high stability to heat sterilization and to gamma irradiation. However, the PS 80 adsorption capacity of PES is significantly higher compared to PVDF or cellulosic membranes which may require process specific approaches to avoid the decrease of specified concentrations of formulation compounds. Adsorption data are presented for process relevant PS 80 concentrations in aqueous and protein-containing (mainly IVIG) model feed streams. General trends and factors influencing the adsorption (pH, concentration, CMC, membrane specific surface area) are concluded.

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‘Monomer supply’ as parameter to describe the synthesis-morphology-performance relationship of polyamide thin film composite membranes

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The global water crisis requires smart use of water resources. An attractive technology to produce a variety of high quality water is reverse osmosis (RO). RO is a pressure-driven process that exploits a polyamide (PA) thin film composite (TFC) membrane. Despite extensive research on PA TFC membranes synthesized via interfacial polymerization (IP), clear synthesis-structure-performance relationships (SSPs) are lacking. Consequently, tailoring the performance of polyamide TFC membranes to optimally comply with individual process requirements remains largely ineffective. The solvent, a prime element of IP, is known to drastically influence critical parameters of IP, such as monomer diffusion and partitioning, miscibility between both liquid phases and their interfacial tension (IFT). When carefully selected, solvent variation can provide valuable insights into the IP mechanism and its SSPs. This study assesses the influence during IP on PA TFC morphology and performance of varying monomer concentrations and organic phases with widely varying physico-chemical characteristics (IFT with water, viscosity and monomer equilibrium partitioning). These physico-chemical characteristics were used to introduce two new descriptors of the SSPs of PA TFC membranes: ‘MPD supply’ and ‘TMC supply’. Three non-ionic solvents (hexane, isopar G and hexyl acetate) are studied in parallel with three room temperature ionic liquids. It is found that well-performing membranes are prepared until one of the monomers is added in excess. The susceptibility of the system to excess of one of the monomers depends on the interplay of MPD and TMC supply, which determines the system-specific monomer concentration range for high salt rejecting membranes. By combining the results of the current study with literature data, qualitative SSPs are proposed. Overall, the tunability of the SSPs of PA TFC membranes is shown by tying the effect of monomer concentration and organic phase on membrane performance and narrowing it down to MPD and TMC supply.

Polyvinilidene fluoride membranes decorated by magnetite nanoparticles

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Polivinylidene fluoride (PVDF) is a polymer used to make membranes because of its outstanding mechanical strength, thermal stability and chemical resistance. Unfortunately, the hydrophobicity of PVDF leads to surface fouling, which raises the operational costs associated with PVDF filters making their used in water treatment practice unaffordable resource-constrained settings [1]. The goal of the proposed research is to develop a method of modifying the surface of PVDF ultrafiltration membranes with magnetite (Fe3O4) nanoparticles to preparate magnetic membranes capable of reactive removal of foulatants over the long-term operation of the membrane [2]. Surface modification of the membrane was carried out in several stages: firstly, PVDF membrane was activated with a carbonate buffer with the aim of double bond formation; secondly, polyethyleneimine (PEI) was attached to the surface of PVDF membrane as a flexible spacer to fix Fe3O4 nanoparticles. However, polymer is grafted not only to the surface, but also into the pores of the membranes, reducing their effective radius. Taking everything into account, this investigation is devoted to the development of a technique for surface modification of PVDF membranes with PEI (with pre-filling of pores) to improve their transport properties, at the same time preserving the morphology of the membrane and its characteristics. The process of modifying the membrane surface was confirmed by IR spectroscopy, scanning electron microscopy. The transport properties of the modified membrane have been studied in the process of ultrafiltration of water, as well as solutions of proteins. The immobilization of Fe3O4 nanoparticles allowed to improve the transport properties of the membranes (increasing the permeate volume flux and mass transport coefficient) and eliminate the effect of concentration polarization. The magnetically active surface of modified membranes prevents rapid increase of molecule accumulation in the boundary layer due to the movement of MNPs and to the increase of Reynold's number.

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Epoxide-Based Membranes as a Novel Materials Platform for Liquid and Gas Separations

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Epoxide chemistry has been applied for many decades in a variety of industries owing to their excellent chemical, physical, and thermal resistance and the simple tunability of their chemical composition. However, in membrane technology, the use of epoxides for the synthesis of top-layers for thin-film composite (TFC) membranes has only recently been demonstrated by our group for water separations [1,2]. The same principles of polyamide interfacial polymerization (IP) were successfully transferred to the synthesis of epoxide thin films. By tuning the IP conditions in terms of monomer and initiator, their concentration, the support chemistry and pore size, a wide variety of membrane performance can be achieved. Further densification of the interfacially polymerized thin film by inducing additional cross-links allows to improve the rejection of monovalent salts for water desalination and to achieve CO_2/N_2 selectivity for gas separations. Besides these two applications, the epoxide-based membranes could also be applied for solvent-resistant nanofiltration (SRNF) and organic solvent nanofiltration (OSN) as stability tests for 5 days in organic solvents such as toluene, ethanol and isopropanol showed no performance degradation nor changes in the physicochemical properties of the robust membranes. The epoxy-based top-layers are also intrinsically stable in oxidizing, caustic and acid environments as demonstrated by their full stability in sodium hypochlorite (NaOCl, 500 ppm), NaOH (pH 12) and HNO_3 (pH 1), respectively. ue to their intrinsic high robustness and high degree of tunability, the epoxy-based membranes may therefore lay the foundation for a new generation of stable membranes for NF, SRNF/OSN and gas separation.

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Simple and rapid modification of Cation Exchange membrane for improved monovalent selectivity

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Electrodialysis (ED), is a widely used separation technology in which ions are removed by an alternating arrangement of ion-exchange membranes in a direct electrical field. Monovalent selective cation exchange membranes can have an important role in ED used to prepare irrigation water from brackish water while preserving hardness ions in the product, as well as ED used in the processes for producing minerals from seawater and seawater RO brines. The goal of this work is to develop a simple technique for a stable single-side surface-modified Cation Exchange Membrane (CEM) at room temperature (~20°C), which can be scaled up easily for ED application for monovalent permselectivity. Here we have modified CEM (CMX and FKS-75) surfaces using polyaniline (PANI) and L-Valine (LV, doping agent) to improve the efficiency of the separation of Na^+ ions over Mg^{2+} and/or Ca^{2+} by ED. The surface modification were confirmed with XPS (increased nitrogen content), FTIR (presence of C-N bond), SEM (surface morphology), and zeta potential (change in surface charge). Other properties were investigated by testing the water uptake, and ion exchange capacity (IEC) of the unmodified and modified membrane surface. The preliminary results confirm that the modified membranes showed a high resistance ratio towards Mg^{2+}/Ca^{2+} over Na^+ ions compared to unmodified membranes (R_(Mg^{2+}/Na^+)^(unmodif.CMX)=2.7, R_(Ca^{2+}/Na^+)^(unmodif.CMX)=3.5, R_(Mg^{2+}/Na^+)^(modif.CMX)=20, R_(Ca^{2+}/Na^+)^(modif.CMX)=10; R_(Mg^{2+}/Na^+)^(unmodif.FKS75)=2.1, R_(Ca^{2+}/Na^+)^(unmodif.FKS75)=2.6, R_(Mg^{2+}/Na^+)^(modif.FKS75)=7.5, R_(Ca^{2+}/Na^+)^(modif.FKS75)=6.3).

The ED experiments and transport number prove that the modification of these membranes can improve the monovalent selectivity toward Na^+ ions. The present work also shows improved IEC (>3 times) [1] with 10 min of polymerization time compared to the reported literature, which requires at least 4 h [1, 2]. Overall, we have successfully developed a simple membrane surface modification technique that can be scaled up easily and shows better efficiency than the other membranes available to date for such application.

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The facile swelling-diffusion strategy for polyamide membranes to enhance their permselectivity for mono/multivalent cations

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Polyamide (PA) membranes have drawn global attention for securing clean water. However, due to the limited structural and chemical natures of PA, their application to various separation fields has been impeded. Although several strategies have been employed, they resulted in performance trade-off or required expensive processes. Herein, we propose a facile method to modify the structural and chemical properties of PA membranes. The solvent activation (SA) process, where a PA membrane is exposed to a proper organic solvent, can convert a dense reverse osmosis structure into a loose nanofiltration structure via swelling and annealing processes. Moreover, by introducing polyelectrolyte modifiers during the SA process, referred to as the swelling-diffusion process, the modifier will diffuse and be physically interlocked with the PA network, thus altering the structural and chemical properties of the membrane. The resultant membrane exhibits high water permeance, rejection, and superior Li/Mg selectivity, which can broaden the application spectrum of PA membranes.

Interfacial polymerization on PAI hollow fiber membranes featuring a polyelectrolyte intermediate layer for forward osmosis applications

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Forward osmosis applications require thin-walled, robust membranes with dense separation layers to minimize internal concentration polarization and provide low reverse salt fluxes. Indeed, most commercial forward osmosis membranes are flat sheet membranes consisting of a polysulfone support with a selective polyamide layer. This layer forms through the interfacial polymerization of a functional amine, e.g., m-phenylenediamine (MPD), and a functional acyl chloride, e.g., trimesoyl chloride (TMC). However, forward osmosis hollow fiber membranes are highly desired since they allow higher packing densities, backflushing, and are less prone to fouling. Yet, while the interfacial polymerization on flat sheet membranes is a well-established and reproducible process, interfacial polymerization on hollow fiber membranes is notably more difficult. Especially, the occurrence of defects during coating and the delamination of the selective layer during operation has prevented the interfacial polymerization on hollow fibers from achieving larger-scale manufacturing. This work presents the fabrication of robust hollow fiber thin-film composite membranes for forward osmosis applications. The hollow fiber support made of Torlon® polyamide-imide (PAI) with an intermediate polyethyleneimine (PEI) lumen layer forms successfully in a single-step spinning process. The intermediate PEI lumen layer facilitates the subsequent coating with interfacial polymerization with a high success rate. Here, the interfacial polymerization of MPD and TMC creates the dense selective polyamide layer. The resulting thin-film composite hollow fiber membranes show a high NaCl rejection of around 96%. In forward osmosis measurements, the fibers feature water fluxes of about 12 L m^{-2} h^{-1} and low reverse salt fluxes of around 0.6gm^-2h^-1. Moreover, the good mechanical properties of PAI allow the fabrication of thin-walled hollow fiber supports which are necessary for forward osmosis processes. This work marks an essential step towards the large-scale manufacturing of thin-film composite hollow fiber membranes for forward osmosis and potentially reverse osmosis and nanofiltration applications.

Green crosslinking approaches for organic solvent nanofiltration membranes

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Improving the physical, chemical, and mechanical properties of polymer membranes are as crucial as separation performance in organic solvent nanofiltration (OSN). Most polymers used in OSN membrane materials are rigid-rod polymers, containing aromatic groups and heterocyclic rings that possess superior chemical and physical stabilities, and resistance in many organic solvents. Although they have been widely applied in OSN, their stability in extreme conditions can be unsatisfactory. Recently, our group fabricated novel membranes based on ionic crosslinking for OSN [1]. The lifespan of modified membranes was increased up to more than 14 days in strong polar aprotic solvents. Another crosslinker, copper (I) iodide, was also proposed at room temperature, forming a metal-polymer complex throughout the whole membrane structure [2]. The formation of metal-polymer coordination not only improved the solvent resistance but also increased the mechanical properties of crosslinked polymer membranes, and led to the tailoring of the separation performance. These facile yet highly effective approaches could replace burdensome covalent crosslinking methods that employ toxic crosslinkers having halogen atoms, and requiring elevated temperature for the crosslinking reaction. In addition, modified membranes were dissolved in DMAc immediately after treating them in a strong base. This observation indicates that the mechanism of ionic treatment on pristine polymer membranes is reversible. Therefore, it is possible to recycle these modified membranes at the end of their lifetime via simple treatments in base to minimize the number of solid waste generation while providing a more sustainable environment. Apart from that, we investigated the impacts of the stereochemistry of membrane crosslinkers on precise molecular sieving in OSN for the first time [3]. Ortho crosslinked membranes were found to have 50% higher styrene dimer rejection than para crosslinked membranes, while para crosslinked membranes exhibited 13%–33% higher acetonitrile permeance than the tighter ortho crosslinked membranes.

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Atomic layer deposition onto and within polymers for controlling interfaces and nano-structuring ultra and nano filtration membranes

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Atomic layer deposition (ALD) is a powerful technique for thin layer deposition of inorganic materials on tortures or high aspect ratio substrates. With ALD, a wide variety of inorganic compositions can be deposited on organic and inorganic substrates with sub-nm accuracy. Here, I will present our work on ALD onto and within polymers. We show, using high-resolution electron microscopy, that controlling the diffusion time of ALD precursors dictates the metal oxide deposition through the depth of the polymeric, phase inverted, ultrafiltration membranes. With just several cycles of Al_2O_3 ALD, we were able to modify the polymer membranes' interface, enhancing the membrane hydrophilicity and oil anti-fouling behavior.[1] We also show that SnO_2 ALD on nanofiltration membrane can be used to tune the surface charge of NF membranes. We further explore ALD within polymers, also known as sequential infiltration synthesis (SIS). In SIS, long diffusion times and precursor-polymer reactivity enables metal oxide growth within the polymers' volume, creating organic-inorganic composite materials. We performed SIS on self-assembled block copolymer (BCP) layers, where the polymer block chemistry yields selective SIS metal oxide growth in only one block. Following the growth, we removed the BCP template to obtain isoporous metal oxide membranes with enhanced biomolecules selectivity.[2] Finally, we combine both approaches to tune the pore size and surface properties of anisotropic BCP membranes where we selectively grow metal oxides in the polymer block surrounding the pore with SIS and further perform ALD cycles to tune pore size from ultrafiltration to nanofiltration. The metal oxide pore interface allows us to control the filtration selectivity as well as be the basis for new functional moieties addition.[3]

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On the interactions between fouling, cleaning and ageing of skim milk ultrafiltration membranes : back to 20 years of experience

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The main bottleneck of skim milk UF is the flux mastering and the fouling removal during cleaning. Over the last 20 years, the interactions between fouling, cleaning and ageing due to the NaOCl disinfection step was systematically studied by the mean of lab scale experiments specially designed to be representative of industrial behaviour. The selected membrane was a PES/PVP (HFK-131, 5-10 kg.mol-1, Koch) was fouled in a standardized UF filtration of skim milk (3h, 0.5-4 bar, 50°C, 0.3 m.s-1 in presence of feed spacers). Regardless of the fouling pressure, the membrane cleaning (1h, 2 bar, 50°C,) was achieved by formulated alkaline detergents (Ultrasil 10® , Ecolab; Deptal UF L1®, Kersia) or enzymatic detergent (Ultrasil 53®, Ecolab). Two main series of experiments were achieved (1) Filtration of skim milk and cleaning & disinfecting solutions on spiral membranes (6.7 m2), either new or voluntary NaOCl aged membrane (2) Filtration with flat membranes either pristine or voluntary chemically aged by using microwave (MW) activation coupled to NaOCl soaking that is representative for ageing at industrial scale [1,2], A selection of results will be shown: 1- Misleading interpretations can be drawn from water flux measurement when dealing with the efficiency of chemical and enzymatic cleaning after nitric acid treatment for a pristine membrane. 2- Changing UF at limiting flux (commonly used at industrial scale) by UF at critical flux decreased the overall and the irreversible fouling, and moreover increased the cleanability of the pristine membrane. 3-NaOCl disinfection steps used to ensure hygienic safety modifying both the critical and limiting fluxes but also the membrane cleanability. 4- Misleading interpretations can be drawn from water flux measurement for NaOCl aged membranes as opposite phenomenon can compensate: ageing induces a flux increase whereas the irreversible fouling increase induces a flux decrease [3].

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The influence of multiple fouling and cleaning cycles on polyethersulphone tight ultrafiltration membranes during coffee extract decaffeination

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The production of reduced caffeine coffee beverages (so called ‘half-decaf’) has recently gained increased commercial attention. Ultrafiltration (UF) processes have been applied widely in food processing industry over the recent decades and could offer a viable alternative to the use of solvents in the decaffeination process, comprising low operational costs, high selectivities and mild processing conditions [1]. This paper reports the development and evaluation of the fouling occurring in multiple filtration cycles during the selective reduction of caffeine from coffee brews using (i) commercially available synthetic tight ultrafiltration (TUF) polyethersulphone (PES) membranes (GP95PP – Alfa Laval) and (ii) Self-made mixed matrix (MMMs) PES membranes fabricated in-house. The effectiveness and performance of the PES MMMs was benchmarked against the commercial 2 kDa PES membranes. A cross-flow rig was operated at transmembrane pressures of 2-9 bar and cross-flow velocities of 0.04 - 0.1 m/s at 25oC. The process was viable, with the production of a much reduced caffeine concentration retentate still rich in higher molecular weight bioactives. PES MMMs exhibited a higher permeate flux of ca. 10.5 L m-2 h-1 with a lower fouling index (FI) of 42% compared to the commercial PES membrane, which exhibited permeate flux of 6.5 L m-2 h-1 with a FI of 60% over the 29 hr filtration period selected. The rejection ratios of the commercial PES and the PES MMMs were ca. 30% and 50% for caffeine, >90% and ~90% for both polyphenols & proteins, & > 80% for HMWMs, respectively. An effective cleaning protocol was reported, comprised of 0.5 wt% NaOH at 50oC, exhibiting cleaning efficiencies (CE) >100%. Both membranes exhibit similar rejection ratios for three consecutive filtration cycles, however differences in filtration performance and cleanability between the two classes of membranes are identified and linked to variations in surface properties and porosity.

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Stormwater treatment using ultrafiltration – Effect of cleaning chemical and backwash time on membrane efficiency

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Stormwater runoff is known as one of the main contributors of pollutants which is often discharged untreated to the water bodies and causes environmental risk [1]. Membrane treatment may be a good option for treatment of heavily polluted stormwater as it can also remove small particles and colloids that are not normally captured in typical stormwater facilities such as ponds, wetlands, etc. However, fouling in membranes is challenging to control. Application of a pre-treatment prior to the membrane process might help to postpone fouling [2]. In addition, a proper combination of backwash and chemical cleaning for the membrane is essential but this has not yet been studied specifically for stormwater runoff treatment with membranes. In this study, a combination of ultrafiltration PVP/PES membrane with pulsatile fluid flow was used for separation of pollutants from stormwater. Sieving was considered as pre-treatment. In addition, a combination of backwash and chemical cleaning was used to assess reversibility, flux recovery and productivity of the membrane. The results showed that by increasing backwash time from 30 to 60 s, the reversibility and flux recovery increased from 26% to 75% and 55% to 87%, on average and also resulted in 20% increase in productivity. Cleaning chemically with alkaline solution (NaOH, pH=12), followed by acidic solution (HCl, pH=2), had a good impact on removing foulants from membrane surface. According to the experiments, addition of NaOCl to the alkaline solution did not have significant effect on pure water flux recovery. From environmental point of view, using NaOCl is not recommended [3].

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Optical characterization of biofilm growth on different coated membranes by optical coherence tomography (OCT)

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The physicochemical properties of membranes (e.g., hydrophobicity, charge, pore size distribution, surface roughness) strongly determine the removal of bacteria through ultrafiltration membranes. The removal efficiency of membranes against bacteria is affected by fouling, which is, among other factors, caused by pore blocking and abiotic gel layers. Biotic processes on the membrane (e.g. biofilm formation) also influence the removal efficiency of the membrane for bacteria. An promissing method to influence the physical properties of such membranes, and thus biofilm growth on the membrane, is the coating of membranes with different materials. Optical coherence tomography (OCT), as a non-invasive method for characterizing the biofilm growth, is promising and subject of this conbribution. Here, a continuously operated cylindrical cross-flow cell with a relatively small diameter (around 3 cm) is used to generate biofilms of diverse properties on different ultrafiltration membranes. The bacterial strain used is E. coli MG1655 cultivated in a lysogeny broth medium. OCT is used to characterize the fouling layers, which allows the simultaneous determination of the thickness, surface roughness, and porosity of fouling layers (Pratofiorito, Horn & Saravia, 2022), (Im, Fortunato & Jang, 2021). The ultrafiltration membranes used are: an uncoated polyether sulfone (PES) membrane, a PES membrane embedded with Bismuth oxyiodide nanoparticles (BiOX), a Carboxylated Polyether Sulfone (CPES) coated membrane with polyelectrolyte (PE) (in our case we use Poly-(diallyl-dimethylammoniumchlorid): PDADMAC and Polyacrylic Acid: PAA), and a CPES PE-BiOX coated membrane. The results of the optical OCT measurements are presented in this contribution. Different membrane coatings affect the characteristics of the biofilm. These coatings with diverse properties will subsequently be used to determine which membranes have the best conditions to minimize biofouling.

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Spontaneous osmotic backwash for organic fouling control in directly coupled solar energy-powered membrane systems

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Various organic matter (OM) in surface and ground waters can deposit on the membrane surface, causing severe organic fouling issues and often requiring frequent cleaning and/or membrane replacement. Spontaneous osmotic backwash (OB) induced by solar irradiance fluctuations is a promising self-cleaning method to mitigate membrane scaling/fouling in directly coupled solar energy-powered nanofiltration/reverse osmosis (NF/RO) membrane systems [2]. This work aims to validate the feasibility of spontaneous OB on organic fouling control with relevant water chemistries (salinity, pH, and hardness) and to evaluate the suitability of OB cleaning for a wide range of OM types regarding the adhesive interactions with NF/RO membranes. The adhesive interactions were quantified by an asymmetric flow field-flow fractionation coupled with an organic carbon detector (FFFF-OCD). A bench-scale crossflow NF/RO system powered by a solar array simulator (SAS) was used to perform the periodic step-response organic fouling experiments with spontaneous OB. The SAS was used to simulate the control solar irradiance fluctuations and induce the OB process. Humic acid (HA) with calcium was selected as a model organic foulant. Eleven OM types included low-molecular-weight organics (LMWO), humic substances, polyphenolic compounds and biopolymers. The results showed that the spontaneous OB restored 46 to 98% flux and removed 5 to 46% organic foulants from the membrane surface, depending on feedwater chemistry, membrane type, and solar irradiance fluctuations [3]. Specifically, OB was more effective with feedwater containing lower calcium concentration and salinity at neutral pH due to the less sticky fouling layer. Humic acid (a typical humic substance) and tannic acid (a typical polyphenolic compound) induced stronger adhesive interaction with NF/RO membranes than biopolymers and LMWO, causing lower OB cleaning efficiency. Even though spontaneous OB cleaning is promising to control organic fouling, it is ineffective in severe organic fouling conditions (e.g. 12.5 mgC/L HA and 2.5 mM calcium.

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Exploring Wetting in Membrane Distillation

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Access to safe drinking water is a major societal issue, when climate change and resource pollution increase water shortage. Sea water desalination is an alternative but desalination plants contribute significantly to CO_2 emissions. In this context, membrane distillation (MD) is promising, as it reduces water losses and can be powered by solar heating.
Our research focuses on two crucial questions: membrane wetting, which can compromise water quality, and optimization of heat management, for developing solar MD for small communities.This lecture focuses on our achievements on methods for exploring wetting and on the knowledge acquired on parameters influencing wetting in MD.

A key issue for MD is to choose i) an appropriate membrane, able to support the liquid/vapor interface without being wetted and ii)pertinent conditions for sustainable process operation. Some (super)hydrophobic membranes are available. However, the criteria chosen to screen the membranes in their development stages are mainly based on wettability assessment (contact angle and LEP).

New tools to explore wetting it-self, the DTTI method [1] and an in-situ optical method [2] will be introduced. They allow identifying the wetting mechanisms (before total wetting) and quantifying the water intrusion into membrane. The optical method is *in-operando* and measures a wetting indicator and its dynamics during MD. It produces cartographies that show local values of wetting over the membrane surface, as shown on PVDF membranes.

Wetting was studied on a set of PVDF membranes and the possible influence of structural properties will be discussed. The tools are useful to screen and develop membranes, as for nanoparticle-PVDF composite membranes [3] for which the load of NPs affects wetting. Among the operating conditions, feed salinity appears a major influencing parameter with a threshold level. Thus great care should be taken in selecting MD membranes for applications at high salinity such as brine valorization.

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Preparation of supported ECTFE membranes and their application in membrane distillation

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In this work, supported ECTFE flat sheet membranes were prepared at around 160 oC. Halar® Ethylene-Chlorotrifluoroethylene (LMP ECTFE) was used as cast-coating material taking advantage of its low melting point. LMP ECTFE materials have comparable chemical resistance and mechanical properties with standard Halar® but lower crystallinity, lower viscosity and lower melting point [1]. Two different non-woven supports in polypropylene (PP) were used, the first one with pore size of 11 µm, and the second one with pore size of 2 µm. Di-ethyl Adipate (DEA) was used as solvent to dissolve ECTFE powder. Membranes were prepared using various ECTFE concentrations (from 3% to 10%). Di-ethylene Glycol (DEG) was used as coagulation bath and 2-propanol was used for washing the residual solvents [2]. The produced supported membranes were characterized in terms of morphology, thickness, contact angle, porosity and pore size, and their performance were evaluated in direct contact membrane distillation (DCMD) experiments using pure water and salty solution 0.6 M NaCl as feed. Membrane distillation is an innovative operation of growing interest due to its ability to treat highly concentrated solutions and to produce aqueous streams with a high degree of purity. Feed temperatures were set between from 45 to 65 °C. The results achieved until now indicate that the membranes with PP support of 2 µm are characterized by top surface that became more denser with increasing ECTFE concentration. Moreover, 5% ECTFE membranes present stable fluxes in DCMD operations (at around 6 LMH at 55 °C), with salt rejections above 99.9%, indicating their good separation performance in membrane distillation process.

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Using direct observation to decouple heterogeneous and homogeneous scaling mechanisms in membrane distillation: Interfacial energy of the scalant determines the probability for scaling

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Membrane distillation has been proposed for enhanced water recovery from hypersaline water [1]. However, concentration of the feedwater results in the precipitation of mineral scalants which form a layer at the membrane surface, significantly reducing membrane performance [2]. While scaling has been extensively studied, the onset of scaling is primarily detected by using the decline in water vapour flux as a surrogate measure of induction [3]. However, it is acknowledged that induction occurs before this phase. Scaling is also known to occur through growth at the membrane surface (heterogeneous nucleation), or via the deposition of precipitates formed in the bulk solution (homogeneous nucleation). Without techniques to determine the onset of nucleation directly, and that differentiate between heterogeneous and homogeneous scaling, limited advancement has been made in description of the scaling mechanism [2]. In this study, we introduce two non-invasive techniques to provide definitive description of scaling in membrane distillation. Scaling can be described by classical nucleation theory, which emphasises how the interfacial energy of the crystallite determines the critical energy barrier for nucleation (G*crit). Mineral scalants were therefore evaluated based on their interfacial energy which introduce a transition in supersaturation and induction time, directly changing the kinetics of nucleation. Mineral scalants that exhibit low interfacial energy (e.g. NaCl), introduce surface scaling before bulk nucleation occurs. Mineral scalants of higher interfacial energy do not scale the membrane before nucleation (e.g. $CaSO_4$). Scaling instead occurs through a deposition mechanism following bulk induction. The extent of surface coverage before bulk induction is therefore directly correlated to G*crit, where higher supersaturation indices at induction favour a homogeneous scaling mechanism. Nucleation rate and crystal growth also correlated to the relative superaturation achieved at induction. The outcome is a framework to predict scaling kinetics and serves a basis on which to develop scaling mitigation strategies.

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Energy consumption of the permeate recovery step in Osmotic and Vacuum Membrane Distillation for juices concentration

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Fruit/vegetable juices are often concentrated by the food and beverage industry to achieve a better preservation, together with a reduction of transport costs and an easier storage. Thanks to the mild conditions in which they operate, membrane processes were investigated for juice concentration as alternative to the conventional multistage vacuum evaporators, usually working at around 50 °C. In particular, Osmotic Distillation (OD) and Vacuum Membrane Distillation (VMD) were applied. In this work, VMD and OD were compared in terms of energy consumption needed to recover, as liquids at ambient temperature (20 °C), the permeates produced during the juice concentration [1]. The analysis was carried out at parity of operating conditions at the feed side (28 °C and 30 °Bx) and final concentration factor, based on available literature data. In VMD, the vapor permeate was recovered as liquid in a condenser located along the vacuum line (4 mbar), while in OD the vapor permeate was directly condensed in the recirculating brine ($CaCl_2$ 45wt%). Therefore, in VMD the energy requested to recover the permeate was that to condense the vapor permeate under vacuum, while in OD there was the need to evaporate the permeate from the brine and to successively condense it, since the high osmotic pressure of the brine did not allow to use nanofiltration (NF) or reverse osmosis (RO) for the direct removal of the liquid permeate from the brine stream. From the calculations made, it resulted that the evaporation (carried out by boiling the brine at 28°C and 10 mbar) and condensation steps for the brine regeneration in OD led to an energy consumption per produced permeate (4893 J/g) higher than that linked to the condensation of the vapor permeate in VMD (3090 J/g). In both operations, the highest contributors to the energy consumption were those involving a phase change.

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Application of Membrane Distillation-Crystallization for Recovering Fresh-Water from Li-rich hypersaline brines

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Lithium is one of the new metals incorporated to the list of the European Union critical resources, due to its relevance for the fabrication of Li-ion batteries in the context of electromobility supply. Besides, almost 50% Li reserves worldwide are contained in salt-lake brines located in Chile, particularly in the Atacama's salt-lake. However, the current extraction process, based on solar evaporation ponds, requires the evaporation of around 90% of the water contained in the Li-rich brine, losing a valuable resource in one of the most arid place worldwide. This situation has generated tension between Li producers and communities and environmental entities. In this regard, the development of new alternatives for Li-rich brine processing has gained interest by reserachers, technology development companies and Li producers. Thus, the option of using membrane distillation-crystallization (MDCr) to concentrate Li-rich brines, while fresh-water is recovered, was conceptually proposed [1]. This approach was called the LiSa process, which considers the fractionation of main salts contained in Li-rich brines by different stages of MDCr. Results using simplified synthetic brines reached water transmembrane fluxes around of 3.5 kg/m^{2}h at 50 °C of feed temperature of the brine and 23 °C of feed distillate temperature [1]. Under similar operational temperatures and flowrates, several Li-rich brines, mimicking real brines, were tested to assess the effect of main ions typically contained in these sources, such as Na, K and Mg [2]. Results of water flux decresed to a range of 2.0 and 2.5 kg/m^{2}h, due to the use of hypersaline brines. The increase of feed temperature of the brine to 60 °C increased the water flux to 3.5 kg/m^{2}h. Similar result was obtained when a real brine was tested. Fresh-water obtained had high quality for different purposes. This study shows recent progress in the application of MDCr to recover water from Li-rich brines.

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Membrane gas separation processes: yesterday, today… tomorrow

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Membrane processes are nowadays, together with cryogeny, absorption and adsorption, considered as a key technology for gas separation applications. Except the very specific use of mineral membranes based, multistaged process, for uranium enrichment performed since 1942 [1], it took more than 100 years before the visionary application of polymers, proposed by T. Graham in 1866, become an industrial reality [2].

Major issues in terms of materials science and process development needed indeed to be solved for the membrane solution to be considered as liable and cost effective compared to other gas separation technologies. Air separation (nitrogen production), hydrogen purification, natural gas treatment, VOC recovery and gas drying are today the major industrial applications. The number of membranes and modules suppliers nevertheless remains very limited. The commercially available membrane modules (spiral or hollow fiber) almost exclusively make use of ultra-thin dense polymers on a porous support, with a very limited number of polymer types [3].

Beyond the current status, the question of the future of membrane gas separations will be addressed. The impact of nanostructured materials revolution (CMS, MOF, graphene, zeolites…), the possibilities of new materials production technologies, the recent developments of simulation and optimisation tools (Molecular simulation, CFD, Process synthesis) will be discussed through different case studies. The key importance of associating breakthrough materials and process design studies for pushing new developments is highlighted.

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Long-term performance of highly selective carbon hollow fiber membranes for biogas upgrading in the presence of H_2S and water vapor

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In this work, for the first time in literature, the separation performances of as-prepared cellulose-based carbon hollow fiber membranes were monitored for 183 days under continuous exposure to a gas stream also containing H_2S and/or water vapor. After the evaluation of their CO_2 and CH_4 sorption (2.98 mmol g^{-1} and 2.00 mmol g^{-1}, respectively at 298 K and 10 bar), diffusivity (2.45×10^{-7} cm^2 s^{-1} for CO_2), and permeation properties (120.9 and 2.3 barrer, respectively at 308 K) with single gases, long-term tests were carried out by feeding gas mixtures with typical biogas composition. It was found that the exposure of the membrane to the H_2S provoked a reduction of both CO_2 and CH_4 permeability by 43% and 25%, respectively, compared to the mixed gas feeding without H_2S, with a drop in selectivity of about 23%. The exposure to humidified gas streams soon caused a CO_2 permeability reduction of about 67 %, mostly ascribable to strong competitive adsorption between the water vapor and CO_2 as well as to water molecules clustering in the membrane pores. Then, the separation performance of the membrane module remained relatively constant with a CO_2/CH_4 selectivity of more than 200 at 28 days of continuous testing. Overall, the membranes underwent more than 183 days of continuous testing, over an elapsed time of 364 days, also in presence of contaminants and water vapor, without being damaged or losing their capability to separate CO_2/CH_4 stream, thus confirming to be attractive candidates for their application in biogas upgrading.

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Critical aspects of high-pressure CO_2-induced plasticization in polyimide membranes

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CO_2-induced plasticization is a major challenge for polymeric membranes for gas separation. These membranes often lose much of their selectivity, especially at high CO_2 pressures, due to plasticization resulting in poor membrane performances. CO_2 is in its supercritical (sc) state above 74 bar and 31 °C, which has significantly different properties compared to gaseous CO_2. Sc-CO_2 is often utilized as an extraction agent in the food industry or in enhanced methane recovery processes. The effect of the CO_2 fluid properties on the membrane performance and plasticization behavior is not much studied, while it has a large impact in high-pressure applications. In this presentation we explain the effect of high CO_2 pressures (up to 120 bar and 55 °C) on the plasticization of polyimide membranes. The transition from gaseous-like to liquid-like sc-CO_2 was found to have the most influence on the CO_2 density and thus also influences the CO_2 sorption and permeability. The CO_2 sorption is closely related to the CO_2 density and determined the extent of plasticization, but showed no time-dependent behavior or hysteresis in the liquid-like region. In contrast, the CO_2 permeability strongly increased with increasing feed pressure and showed significant time-dependent behavior even in the liquid-like region. However, the extent of plasticization was found to be independent of the applied feed pressure in the liquid-like region. Moreover, the exposure to liquid-like sc-CO_2 changed the permeation behavior at all subsequent pressures due to severe plasticization and low chain relaxation rates. These results proof that the CO_2 fluid properties, exposure time and permeation history are critical aspects for plasticization at high CO_2 pressures. Finally, suppression of plasticization was achieved using three conventional methods (blending, thermal and crosslinking treatments), which showed that a strong interchain rigidity is required to suppress plasticization at liquid-like conditions.

Use of green(er) solvents for preparation and upscaling of polyimide gas separation membranes

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Biogas draws growing attention as a crucial renewable energy source, complementing EU objectives in achieving up to 45% renewable energy shares by 2030. Presently, biogas is principally converted as-is into electricity and heat, using CHP units, leaving immense prospect in valorization of biogas to biomethane, through membrane-based purification of its multi-component stream consisting of CH_4, CO_2, and other trace impurities. Industrial membrane units classically comprise of flat-sheet or hollow-fiber polymer membranes, synthesized using non-solvent induced phase separation (NIPS), with polymer processing typically performed using solvents such as dimethylformamide (DMF), N-methylpyrrolidone (NMP), dimethylacetamide (DMAc), chloroform, etc. However, use and subsequent waste handling of conventional solvents present significant hazards due to their high toxicological nature, calling for a shift to employ safer materials to facilitate sustainable scale-up production of these membranes. A novel methodology for the synthesis of flat-sheet CO_2/CH_4 gas separation integrally skinned asymmetric membranes was developed with commercially available Matrimid® 5218 polyimide, using Tamisolve NxG, dimethylsulfoxide (DMSO) and 2-methyltetrahydrofuran (MeTHF) as green(er) solvent alternatives. Systematic studies on effect of polymer concentration and solvent mixture ratio in the casting solution, membrane drying, and cross-linking on gas separation performance were conducted on throughput gas permeation devices, along with thermal stability and plasticization resistance measurements. In tandem, a fundamental analysis of phase inversion and structure-property relations involving the new polyimide/solvent-mixture system was assessed through ternary phase diagrams, viscosity profiles, and other characterization techniques. The developed methodology provides a sustainable framework for synthesizing polyimide-based membranes with tunable gas separation performances, where a mixed gas CO_2/CH_4 selectivity of 40 was achieved, facilitating implementation of this protocol to scale-up membrane synthesis on the pilot-scale roll-to-roll membrane manufacturing unit of MTG at KU Leuven.

Postcombustion carbon capture via nanoporous graphene membranes: a techno-economic analysis

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Membrane processes are emerging as an alternative to absorption for postcombustion CO_2 capture. Membranes based on nanoporous graphene atom-thick selective layer have shown high promise in this regard with extremely large CO_2 permeance (10000 GPU) and attractive CO_2/N_2 selectivity (30) [1,2]. We report a techno-economic model and assess the most performing process configuration for CO_2 capture from wet flue gas (CO_2 concentration of 13.5%). We analyze the role of feed and permeate pressure on energy consumption and capture penalty, by comparing scenarios with compression in the feed channel, vacuum in the permeate channel or a combination of these. When the permeate is under vacuum, the energy consumption corresponding to a certain pressure ratio is lower than when feed is compressed, but the membrane area increases. However, the high permeance of graphene membranes uniquely makes this scenario more competitive than feed compression in most cases (large range of pressure ratio and membrane cost) [3]. Optimizing the double-stage process configuration, we find a minimum capture penalty of 34.3 \$/ton with permeate pressures of 0.05 and 0.1 bar in the first and in the second stage, with membrane module cost of 500 $\$/m^2$. This already promising value of capture penalty can be further reduced by taking into account improved membrane performances, already achieved in our lab. When the selectivity increases to 85, the energy consumption decreases by 25% and the capture penalty goes down to 28.9 \$/ton. Instead, with increased permeance of 15000 GPU, the membrane area decreases by 33% and the capture penalty results 30.6 \$/ton. These results show that graphene membrane-based CO_2 capture is competitive with state-of-art amine absorption and the optimised polymeric membrane-based systems.

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A unified view on transport electrolytes and non-electrolytes in NF and RO membranes

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The use of NF and RO membrane has been steadily growing over the last decades. While the RO market is still dominated by polyamide composites [1], NF membrane show more diversity, with new types of commercial NF membranes emerging recently as new commercial products, such as polyelectrolyte complex (PEC) memrbanes. Compared with polyamides, the latter show similar ion-rejecting properties, but much higher rejection of organic micropollutants [2]. This distinct behavior adds to the puzzle of ion transport in RO and NF memrbanes, which is still poorly understood in general [3]. In this talk, we outline a unifying view on selectivity of NF and RO memrbanes, in which we will will demonstrate how rejection of electrolyte (e.g., salts) and non-electrolytes (e.g., micropollutant rejection in PEC membranes) may be explained within the same thermodynamic picture. At the heart of this picture are the dielectric charactersitics, responsible for rejection of salts and ion selectivity in memrbanes as well as various types of nanochannels, that explain many puzzling results that commonly used models fail to rationalize. We show and elaborate how organic rejection of PEC membranes in NF and other processes (e.g., pervaporation) fits smoothly to this emerging physical picture.

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GO- and grafting-GO embedded PVAm-based nanocomposite membranes for CO_2/H_2 separation

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Global warming and energy shortage have been the major concerns of the 21st century, most of which are due to the increasing demand of burning fossil fuel for energy which causes the emission of CO_2, the major green-house gas, into the atmosphere. To relieve the concerns, one approach is to shift to green and clean energy such as using non-carbon-based fuel (e.g., H_2). Currently, steam reforming of hydrocarbons followed by the water-gas shift reaction is the main process to produce H_2. Pressure swing adsorption (PSA) is the most conventional method for the H2 purifiaction in today's industry and membrane separation is a promising alternative owning to its lower energy consumption, smaller footprint, lower capital and operating cost, higher energy efficiency, operation simplicity, and high modularity. In this work, a CO_2-selective membrane based on facilitated transport mechanism is developed. GO and grafting-GO-embedded polyvinylamine (PVAm)-based nanocomposite membranes are prepared and optimized for CO_2/H_2 separation. PVAm has a great positive effect in the CO_2/H_2 separation due to reversible reaction between CO_2 and fixed site carriers (amino groups). Incorporating GO or polyvinyl alcohol-grafted-GO (PVA-GO) nanosheets in a polymeric membrane has the potential to improve the selectivity of CO_2 over H_2 because GO nanosheets are impermeable of small molecules such as H_2 and provides additional CO_2 adsorption sites. The surface modified GO with higher CO_2 affinity and better compatibility with polymer matrix also benefits the competition of CO_2 sorption over H_2. GO- and PVA-GO-based PVAm membranes have been prepared and characterized to evaluate their physical, chemical and separation properties using SEM, FTIR, TGA, and gas permeation tests. The effect of the thickness of membranes were also tested. Compared with a neat PVAm membrane, the CO_2/H_2 selectivity of the two above-mentioned membranes increased.

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Influence of the forces on the adsorption of steroid hormone micropollutants in the nanoconfinement of vertically aligned carbon nanotube membranes

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In nanofiltration (NF) and reverse osmosis (RO), micropollutants (e.g. steroid hormones) can be adsorbed to the membrane material and gradually permeate through the membrane, and as a result the experimental removal is lower than expected from pore models [1]. The complexity of the polymeric NF/RO active layer presents a challenge to understand adsorption. Vertically aligned carbon nanotube (VaCNT) membranes offer an ideal platform to examine interactions inside the nanopores because of the well-defined pore geometry. Water conducts rapidly through their hydrophobic, smooth, and low-tortuosity pores [2], but this may limit surface interactions with contaminants. In this study, hormone adsorption in nanopores was explained by the interplay of forces acting on the hormone molecule: i) hydrodynamic drag force along the flow direction, ii) opposing hormone−wall friction force resisting the molecule movement, iii) adhesive force directing the hormone toward the wall, and iv) repulsive force between the electrons of the wall and the hormone [3]. With a fast flow velocity inside the VaCNT pores ($4.7 \cdot 10^{-4}$ m/s, in the NF range), 17β-estradiol (E2) hormone adsorption was insignificant because the strong drag force ($3.3 \cdot 10^{-3}$ pN) pushed E2 to move and exit the pores. When the drag force decreased from $2.2 \cdot 10^{-3}$ to to $4.3 \cdot 10^{-4}$ pN, the friction force was significant as E2 adsorbed mass increased from 0 to 0.4 ng/cm^2. At a constant low drag force ($1.6 \cdot 10^{-3}$ pN), the adsorbed mass of four hormones was correlated to the adhesive force. This adhesive force drives the hormone toward the wall until it balances the repulsive force; and influences the friction force resisting the hormone movement with the flow. The proposed framework to explain micropollutant adsorption in nanopores (e.g. VaCNT and NF) by the interplay of these four forces can help design membranes with improved selectivity.

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Green supported liquid membranes: A graphical activity-based process design method

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Supported Liquid Membranes (SLMs), composed of a thin porous support containing an extraction media, can be used for the selective separation of diluted compounds in liquid mixtures. The extraction and back-extraction stages are combined into a single process, with significant solvent requirement reduction as compared to conventional liquid-liquid extractions [1]. Our group has demonstrated that coupling an SLM with green solvents, such as ionic liquids (IL), eutectic solvents (ES), or biobased solvents (BS), a so-called green-SLM, leads to good separation performance of bio-organic acids from synthetic fermentation broths [2]. However, implementation of SLM at industrial scale is being held back due to the poor maturity and lack of technological sophistication. Among the key tasks to overcome such hurdles, enhanced predictive models and design algorithms are needed. Therefore, we propose a green-SLM process design using the permeability activity-based linear operation (PABLO) method [3]. The model considers the solute–phase affinities, quantified by the activity coefficient, to describe the mass transfer driving force. This approach improves the extraction prediction over the current concentration-based models. Recovery of succinic acid from model fermentation broths has been accomplished using four different green-SLM based on the ESs: DL-menthol:OctA{1:1} and N4444Cl:OctA{1:2}, the BS: eucalyptol, and the IL:[C4pyrr][Tf2N], with a stripping phase-facilitated transport mechanism. Activity coefficients were estimated using the quantum chemical COSMO-RS method and corrected with the experimental data. Derived from the proposed permeability model, a graphical methodology is provided to determine the theoretical number of stages required to reach a target solute extraction as well as the mass transfer area in a multistage countercurrent cascade process. This work displays an innovative tool for the green-SLM process design that will substantially contribute to its technological maturity in the biorefinery industry.

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Submerged osmotic processes: Impact of aeration on fouling and external concentration polarization mitigation

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Submerged forward osmosis (FO) is of high interest to treat and concentrate complex streams where cross flow module suffers from limitations such as breakage of fragile compounds, viscosity issue or clogging limitations1-2. In this study, 0.1 m^2 submerged plate and frame FO modules were designed using commercial FO membranes. Tests were conducted in a submerged reactor with FO modules dept in 4L feed solution and 2 L of 35g L^{-1} NaCl draw circulated through the FO module. Impact of aeration (with/without), aeration intensity (from 10 to up to 200 L h^{-1}) and on/off cycles of aeration (i.e., 10min/10min, 2min/10min, 1/5min respectively) were assessed. Deionised water, 5 g L^{-1} NaCl and 1 g L^{-1} Bentonite were used as feed solutions to evaluate the impact of aeration on external concentration polarization (ECP) and fouling propensity. Operation of aeration in on/off cycles confirmed that once aeration is stopped, very rapid ECP layer is formed at the membrane surface due to both reverse salt diffusion and the initial salinity in the feed solution; flux decreased from 12 down to a plateau of 9 L m^{-2} h^{-1} within 5 min. When switching on the aeration, ECP layer is removed immediately, and flux recovered up to its initial value. Similar tests performed with bentonite as feed solution, confirmed also that such low rate of continuous aeration is sufficient to avoid particulate fouling. On/off aeration cycles demonstrated that, when aeration is stopped, flux decrease is higher when bentonite is present and thus that both ECP and fouling phenomenon are simultaneously occurring. Switching on the aeration allowed for incomplete recovery of the initial flux, attesting for irreversible fouling along on the on/off repeated cycles. Thus, this study provides novel insight of the impact of aeration in ECP and fouling mitigation and will propose recommendations for sustainable operation of submerged FO processes.

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Artificial Water Channels-toward Biomimetic Membranes for Desalination

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This lecture discusses recent development of the first artificial water channels systems. We include only systems that integrate synthetic elements in their water selective translocation unit. Therefore, we exclude peptide channels because their sequences derive from the proteins in natural channels. We review many of the natural systems involved in water and related proton transport processes. We describe how these systems can fit within our primary goal of maintaining natural function within bio-assisted artificial systems. In the last part, we present several inspiring breakthroughs from the last decade in the field of biomimetic artificial water channels. All these examples demonstrate how the novel interactive water-channels can parallel biomolecular systems. At the same time these simpler artificial water channels offer a means of understanding water structures useful to understanding many biological scenarios. Moreover, they can be used for the preparation of highly selective membranes for desalination.

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Nature-inspired engineered hydrodynamics for highly efficient membrane separations

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Membrane-based filtration processes are increasingly used in industrial environments, for being energy-saving and cost-effective. However, these processes also have some drawbacks, membrane fouling being one of the most difficult to deal with. Natural filtration processes such as the kidney's blood filtration, cellular protein transport, and the filter-feeding system of certain animals are examples of highly effective membrane filtration techniques. Their efficiency is likely a result of multiple factors, such as the physicochemical and geometrical characteristics of the membranes. When seeking to develop a nature-inspired membrane, one should understand the underlying mechanisms behind such natural systems, from the nanoscale to the macroscale, in addition to possible dynamic factors.1 Previous research drawing inspiration from the kidney has shown that tuning charge and hydrophilicity of the membrane surface can affect their antifouling properties.2 Also, a cell membrane-inspired graphene nanomesh membrane exhibited superior antifouling properties, while maintaining a high water flux through the membrane.3 Since multiple scales are very important in natural systems, the hydrodynamics might also influence anti-fouling in membrane filtration. The glomerulus curvature, the pulsation of blood in the glomerular blood vessels and the oral cavities of filter feeding species might generate fluid instabilities that decrease fouling. Inspired by these natural phenomena, CFD simulations were performed to investigate how the hydrodynamics influence membrane filtration. Hydrodynamics can be influenced by multiple subfactors. Preliminary results showed that, for Re = 250 and a membrane represented by a square wire screen with 2% porosity, having a spiraling tube instead of a straight tube, increased the number of particles contained in the retentate by more than 3%. This indicates higher efficiency of the membrane filtration and a decrease in fouling. The effects of the filtration operation, the membrane module's geometry, and the flow type (continuous or pulsating flow) efficiency on preventing fouling will be discussed.

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Artificial compartmentalization of enzymes in different places of the same membrane by using switchable pH-responsive membranes

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In living cells, the multhienzymatic biosynthesis is possible through the biomolecules compartmentalization in a process called "methabolic channeling"; in which microcompartments, delimited by biological membranes, promote high specific enzymatic processes, interconnected with other enzymes in a very efficient methabolic pathway. The development of biofunctionalized artificial membrane could simulate the natural compartmentalization of one enzyme, but the one of several enzymes is difficult, since it needs to spatially localize them in the different membrane compartments (e.g.: membrane surface, within the pores). In this work two different enzymes were immobilized, one on the membrane surface and the other within the membrane pores, though the use of switchable pH-responsive membranes. In particular the compartmentalization was obtained by using polistirene-b-poli-4-vinil pyridine membrane, which is able to close (pH: 2- 3.5) and open the pores (pH: 4-8) and two enzymes, important in biorefinery (pectinase and beta-glucosidase), with highest activity at the pH values compatible with pore opening and closing. Pectinase was covalently (demonstrated by XPS) immobilized on the surface of the membrane to control membrane fouling due to pectin accumulation (pH optimum around 3), while β-glucosidase (pH optimum 6.5) was immobilized within the membrane pores to hydrolyze oleuropein into the phytotherapic oleuropein aglycone [1,2]. Both enzymes are active at pH 6.5 with a conversion of oleuropein and pectin of 93 and 95 %, respectively, but the reaction can be shifted to the prevalent pectin hydrolysis moving the pH to 3. Characterization of the biocatalityc membrane by AFM and confocal microscopy demonstrated that pectinase is prevalently present on the surface, since no changes in rugosity was observed after the second immobilization and that it is very well distributed without aggregates formation. Besides, SEM analysis after immunolocalization demonstrated that pectinase is present just on the membrane surface, while beta-glucosidase is mainly present within the membrane thickness.

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Revisiting the fabrication of hemoglobin-based membranes for oxygen facilitated transport

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Biocompatible porous membranes have been largely reported as beneficial scaffolds for Tissue Engineering (TE) [1-3] in bioreactors as they can provide a 3D porous platform for cell culture and to facilitate the nutrients supply and metabolites removal. Nevertheles, sufficient oxygen supply to 3D cell cultures maintained in vitro is a challenging issue that remains unsolved. In this work, the old concept of incorporating hemoglobin as oxygen carrier in polymeric membranes for oxygen facilitating transport has been revisited. Commercially available oxygen-binding hemoglobin (Sigma Aldrich) has been first encapsulated in the biocompatible polymer PEBAX®2533 nanoparticles (NP) using coaxial electrospraying. Electrospraying processing variables were optimized achiving Hb/PEBAX NPs with average size ranging between 250-750 nm. Hemoglobine successful encapsulation was confirmed by TEM-EDX and Raman spectroscopy. Further, the capacity for oxygen adsorption of Hb/PEBAX NPs was characterized. Experimental oxygen adsorption data of the Hb/PEBAX NPs were in agreement with estimated theoretical data according to Hb concentration in the NPs. As a proof of concept, 6 mg of Hb have been electrosprayed as Hb/PEBAX NPs on a 12.5 cm2 ultrafiltration PVDF membrane support prepared by NIPS at the laboratory and further cover by one layer of spray-coated PEBAX2533. The fluxes of O2 and N2 and selectivity ratios were characterized using a custommade gas permeation cell. At partial pressures of O2 in the atmospheric level or higher the PVDF/Hb/PEBAX membranes presented O2/N2 selectivities about 3.3, mostly characteristic of PEBAX2533 gas permeable properties. Further studies are currently in progress to evaluate the effect of O2 partial pressure and Hb concentration on the posible O2 facilitating transport through these new PVDF/Hb/PEBAX membranes for TE.

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Investigation of the effect of quorum quenching (QQ) enzyme on biofilm formation on the biocatalytic membranes

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Biocatalytic membranes, which are fabricated by taking advantage of the synergetic effect of membranes and enzymes, have been one of the attractive approaches for anti-biofilm systems as an environmentally friendly solution. In this study, biocatalytic membranes were developed for preventing biofilm formation by degrading AHLs. The crude quorum quenching (QQ) enzyme was covalently immobilized on the polyethersulfone (PES) ultrafiltration membrane, which is purified by ammonium sulfide precipitation from Bacillus T5. Enzyme loading efficiency and activity were observed to be more than 50%. The performance of the fabricated membranes was assessed by water flux, attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR), contact angle, pore size, and scanning electron microscopy (SEM). Moreover, Pseudomonas aeruginosa was used as a model bacteria to assess the antifouling performance of membranes under static incubation. Additionally, the crude QQ activity of the immobilized membrane was also confirmed by visualizing the distribution of cells on the surface of each membrane using confocal laser scanning microscopy (CLSM) image analysis. This research demonstrates the high potential of immobilized quorum-quenching enzyme membranes for preventing biofilm.

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Water transport in graphene oxide membranes

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Carbon-based materials such as graphene, carbon nanotube and amorphous carbon, have attracted great interests in developing high performance membranes for various applications. Among them, graphene oxide (GO) membranes have been reported to be a promising separation barrier that can retain small molecules and multi-valent salts because of the well-defined interlayer space between GO flakes. However, it is still puzzling that while many studies suggested fast liquid transport through the extremely tortuous transport path, contradictory observations have also been obtained. This presentation will discuss the water transport in GO membranes to understand the real water permeation flux of GO membranes. In our study, we reveal that all GO membranes prepared by varied methods and on different substrates present severe reduction in water permeability due to the compaction of their original loose microstructure. The water flux drop continuously from tens of LMH bar^{-1} down to the range < 0.1 LMH bar^{-1} after more than ten hours. This result demonstrates that the structure of GO membranes prepared by current approaches is far from the ideal laminar structure. Therefore, the high permeability of GO membranes observed in previous studies could be contributed from the disordered membrane microstructure.

High-Performance CO_2- and O_2-Sieving Nanoporous Single-Layer Graphene Membrane

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Nanoporous single-layer graphene (N-SLG) membrane is a highly promising porous interface to realize energy-efficient gas separation. The extremely low diffusion resistance at the nanopore makes it capable of yielding ultra-high gas permeance and an attractive molecular selectivity [1-3]. However, it remained challenging to achieve the predicted unprecedented separation performance of the N-SLG membrane experimentally. The major bottlenecks have been the incorporation of high-density nanopores in single-layer graphene with sub-angstrom precision in molecular differentiation. Herein, we developed a millisecond ozone-based carbon gasification chemistry to etch nanopores in graphene with narrow pore-size distribution (PSD), obtaining the needed sub-angstrom resolution for gas separation [4]. The rapid etching kinetics with millisecond pore expansion time led to uniform and high-density (10^12 cm^{-2}) nanopores in N-SLG. The obtained nanopores effectively sieve various similarly-sized gas pairs, including several industrially relevant mixtures such as CO_2/O_2, CO_2/N_2 and CO_2/CH_4. Furthermore, the molecular cutoff can be further tuned by 0.1 Å via ozone-based pore-edge functionalization and oxygen-based slow etching. The resulting N-SLG membrane reached O_2/N_2 selectivity of 3.48 with corresponding O_2 permeance of 1300 gas permeation units (GPU), and CO_2/N_2 selectivity of 21.7 with corresponding CO_2 permeance of 11850 GPU. The excellent gas separation performance makes these membranes highly promising for energy-efficient post-combustion carbon capture.

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Tailoring of a Piezo-Photo-Thermal Solar Evaporator for Simultaneous Steam and Power Generation

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Water and electricity shortages constitute a global energy crisis that cannot be ignored. The sun is an unlimited source of energy, and oceans provide abundant water and renewable energy resources. In this study, poly(vinylidene fluoride) (PVDF)/graphene solar evaporator membranes are fabricated for simultaneous freshwater production and power generation. Graphene addition transformed the PVDF crystal from the α-phase to the piezoelectric self-assembly β-phase. The resulting membrane is used to convert the mechanical energy of waves to electrical energy. The membrane has an output voltage of 2.6 V (±1.3 V) and an energy density of 2.11 Wm^{-2} for 1 Hz simulated waves, which are higher than values reported in the literature. The stacked graphene and polymer formed a wood-lumens-like mesoporous structure with a photothermal effect. Under one sun illumination, the water production rate is 1.2 $kgm^{-2}h^{-1}$, and the solar-thermal energy conversion efficiency is 84%. Finally, a prototype is built to prove a single evaporator's feasibility that can simultaneously obtain freshwater and generate electricity. Thus, this membrane serves as an ocean wave power generation device that can provide all-weather energy generation, convert stored electrical energy into thermal energy at night and on cloudy days, and continuously provide safe drinking water.

Mixed Polymeric Substrate RO Membranes with Nanographene-Surface Modification

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Nanographene oxide is considered one of the most promising key players in the industry of surface coating generally and specially in membrane surface modification. These modifications have been strongly emerging nowadays; to cope with the increase in demands for RO-membrane desalination units with polyamide severe deterioration and decline in performance due to higher chlorine remains to control biofouling. This problem reflects directly on increase in OPEX due to increase in RO cartridge cost and lifetime reduction. In this work mixed polymers, polysulfone and polyacrylonitrile were used as mixed polymeric substrate to enhance substrate properties and performance of polyamide layer. Modified RO/ or polyamide layer was reached using two routes: PVA layer deposition over blend membranes (P-layer) or thin film composite layer (T-layer). Over these layers, chemical grafted layer using methacrylic acid to form Poly-methacrylic acid (P-MAA) in presence of graphene oxide (G-layer). Characterization for surface modified membranes were fulfilled using BET area, SEM, contact angle, FTIR, and others for results discussion and interpretation. Successive layer by layer deposition were applied for surface modification using GO /or ZnO-glycerolate nanostructures together with tannic acid as hydrophilic additive. This shows a certain change in surface hydrophilicity which is highlighted in measurements for water contact angle together with the porosity denoted in BET area. Contact angles were found to give lowest angles for 3rd family of membranes nominated 3P to reach about 41.5°C which increases to reach 46°C for 1P to increase more when chemically grafted to reach almost the same value 49 °C and 51°C respectively. The highest salt rejection reached was about 88% for 3TG and decreases to 80% for 1TG with a corresponding permeate flux 25 and 30 LMH respectively. Optimized surface modification was reached through best permeate flux and salt rejection together with accepted chlorine resistance correlated to biofouling.

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Graphene Enhanced Membranes for Drinking Water Treatment

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We describe graphene enhanced hollow fiber membranes with synergic filtration (micro-ultrafiltration) and adsorption properties. Graphene embedding or coating of the polymeric membrane were exploited to realize core-shell polymeric hollow fibers or polymer-graphene composite membranes, respectively [1-2]. Graphene nanosheets were also chemically modified through covalent chemistry to tailor the adsorption properties toward the removal of selected contaminants in drinking waters [3-4]. As case study we will discuss on Polysulfone-graphene oxide hollow fiber membranes (PSU-GO HFs) showing simultaneous adsorption and ultrafiltration capabilities will be described and proposed for enhanced and simplified Point-of-Use (POU) drinking water purification. The PSU-GO HFs were prepared by phase inversion extrusion by a customized semi-industrial plant and their morphology, surface properties, and porosity were investigated in relation to different GO:PSU ratios (1-5% w/w GO vs PSU) and to the final adsorption-ultrafiltration properties. Filtration modules of PSU-GO HFs of filtering surface (FS) in the range 0.015-0.28 m^2 showed same ultrafiltration capability of PSU-HF standard filters. Synergic adsorption properties were demonstrated by studying the adsorption maximum capacity of ciprofloxacin antibiotic (CIPRO) vs GO ratio in dead end in-out configuration, the standard configuration used for PSU HFs commercial modules. Heavy metals (Pb, Cu and Cr(III)) and polyfluoroalkyl substances (PFAS) removal capabilities from tap water were competitive and in some cases outperformed Granular Activated Carbon (GAC), the standard industrial sorbent. Moreover, release of GO from working PSU-GO modules was excluded by Surface Enhanced Raman Spectroscopy (SERS) analysis of treated water having the state-of-the-art limit of quantification of 0.1 ug/L for GO nanosheets.

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Hydrophilization of PVDF Membrane Via Surface Modification

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Polyvinylidene fluoride (PVDF) is thermoplastic polymer characterized by a low reactivity. It is intrinsically hydrophobic and it is used for applications where high thermal stability and high chemical resistance are needed. Due to its outstanding properties is one of the most widely used membrane materials[1], but its strong water repellency make it unsuitable for handling aqueous solutions. The aim of this work is to develop an effective post-treatment method for hydrophobic PVDF membrane. In particular, the focus will be on development of a surface modification process for the hydrophilization of the pristine membrane [2]. Supported hydrophobic PVDF membranes are produced via NIPS and subsequently treated through a free radical polymerization process in order to graft a hydrogel layer on the membrane surface. The process consists of several dipping steps: the reactants and initiators are adsorbed by the membrane which is then placed in contact with the catalyzer to starts the polymerization. The modification steps are followed by different washing station before the drying unit. The surface modified PVDF membrane were succesfully produced and used for syringe filters and cartridges application. The novelty presented in this work is related to the versatility of the treatment proposed and to the simplicity of the steps involved. Tuning the crosslinker and the grafted monomer, it is possible to confer to the membrane the desired characteristics maintaining the base membrane production process unchanged.

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Hofmeister series of ions: effect on LbL membrane formation and membrane performances

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The Layer-by-Layer (LbL) technique is an efficient, cheap and easily tunable method for nanofiltration (NF) membrane preparation [1]. It consists of the deposition of several oppositely charged polyelectrolyte (PEs) layers on top of a support. The nanometer thickness of the LbL layer guarantees high water permeabilities while the presence of charges and ionic linkages between the PEs impart charge and size selectivity to the membranes. Often, the PE solutions used for LbL membrane preparation contain NaCl to control interactions. Opposite to intrinsic charge compensation at lower salt concentrations, higher salt concentrations result in extrinsic charge compensation between salt ions and the PE. This allows the formation of fewer ionic linkages between the oppositely charged PEs giving rise to thicker and more open membranes [2]. Although much effort was devoted to the investigation of different coating parameters, the effect of the type of salt used has not been extensively researched while this is a very easily tunable variable that can have large impact on performance. In this work we explore the use of different salts on the preparation of LbL membranes and their effect on the membrane performances. The salts selected are classified based on their interaction with water (water structure forming or kosmotropic vs. water structure breaking or chaotropic [3]) taking into account the Hofmeister series [4] as well. The performances of the membranes prepared with kosmotropic ions often exceed the one of the base membrane prepared with NaCl. Divalent salt retentions beyond 95% can be reached even after just 3.5 layer deposited with a MWCO below 400 g/mol. This without compromising the water permeability which remains often higher or comparable to the base membrane. In contrast, when using NaCl the retention of divalent ions is heavily subject to the odd-even effect and does not go above 86% retention even after 7 bilayers.

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Surface modification of polymeric hollow fiber membranes via atomic layer deposition of titanium dioxide (TiO_2) for water purification application

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As surface and underground water has been more and more contaminated, availability and safety of drinking water became an on-going global challenge. Thanks to their high separation efficiency and easy operation, membranes are among the most advanced water purification processes. Polysulfone (PSU) is one of the leading choice for hollow fiber (HF) membranes fabrication because of its excellent chemical and thermal resistance during membrane washing, and its good mechanical properties suitable for long-term use. However, the main drawback of this polymer is its intrinsic hydrophobicity that affects especially antifouling performances [1]. One solution to counteract this problematic would be to add an oxide coating on the membrane surface using atomic layer deposition (ALD). ALD is a unique technology developed over the last four decades [2] which allows the deposition of inorganic coating layers such as oxides with high uniformity and precise subnanometer thickness control. Recent studies have shown that ALD is applicable to flexible and temperature-sensitive polymeric membranes and suggested that it would increase their hydrophilicity, affect their pore size and allow to create low-fouling membrane surface [3]. However, there are still few studies combining ALD and polymeric membranes compared to ceramic membranes because of their lower mechanical property and thermal resistance to undergo the process. This study focuses on a titanium dioxide (TiO_2) ALD coating developed for PSU HF membranes and its influence on the fibers properties. The effect of ALD treatment on the membrane morphology is investigated and the performances of the HF membranes for water purification application are characterized (water permeability, antifouling using Bovine Serum Albumin protein and mechanical properties). Furthermore, the comparison between raw and ALD modified PSU HF membranes behavior is performed. The obtained results are a first step for a more in-depth study on TiO_2 coating by ALD on HF membranes.

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Asymmetric polyelectrolyte multilayer membranes: recent insights

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Polyelectrolyte multilayer (PEM) membranes hold great potential for the optimization of nanofiltration membranes because their properties can be controlled to a large extent. The layer-by-layer assembly of a separation layer on top of a porous support, by alternatively exposing it to polycations and polyanions, provides control over layer thickness. Density, hydrophilicity and charge density can be fine-tuned by the type of polyelectrolytes and coating conditions such as ionic strength and pH. A few years ago, our group demonstrated the potential of PEM membranes by developing asymmetric polyelectrolyte multilayer membranes that are highly suitable for the removal of organic micropollutants from water [1]. The key property of these membranes is that the PEM consists of a low-density, high-permeability layer that closes the pores of the support, with on top of it a thin, high-density layer to define the actual separation properties. The low-density bottom layer ensures a high (up to 12 L/(m^2 h bar)) overall permeability, while the top layer facilitated 98% micropollutant retention in our experiments. However, not much was yet understood about the interactions between the bottom and top layers of these asymmetric PEM membranes. Therefore, recently we explored the contributions of intermixing, swelling and odd-even effects on the total performance of asymmetric membranes. As expected, intermixing is an important factor in asymmetric layer performance. Interestingly, the odd-even effect plays a determining role in our system as well. We also looked into alternative layer types and process conditions. This presentation will give an overview of our most important recent insights in asymmetric PEM buildup and tunability, and how different parameters allow us to control material and membrane properties.

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Anti-wetting modification of commercial polymeric distillation membranes by a simple Plasma Oxygen-FAS grafting method

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Membrane distillation and related technologies have attracted much attention in the last decade due to their ability to operate with low quality energy and their resistance to highly concentrated feeds such as brines. However, in order to reach technological maturity, key problems limiting their industrialization and large-scale development, such as fouling, scaling and pore wetting, need to be solved and consolidated. Modification of membrane materials is one of the most relevant strategies in this case. In this work we explore a simple method of modification of commercially available membranes via plasma-chemical treatments (Plasma O2 - FAS agent grafting) to improve the surface hydrophobic capacity. Commercial polypropylene (PP), polyvinylidene fluoride (PVDF) and polytetrafluoroethylene (PTFE) membranes were subjected to oxygen plasma etching treatments for short activation times (1-10 s), followed by controlled fluorination by grafting with 1H,1H,2H,2H,2H,2H-perfluorooctyltrichlorosilane (PFOCTS) in anhydrous medium. The hydrophobic properties of the modified membranes were determined by techniques such as WCA and tested in direct contact membrane distillation experiments (DCMD) against 70,000 ppm SWRO-type brines. Precise control on plasma-modification times and FAS-agent dosages achieved to significantly increase the water contact angle of all polymeric matrices (102±1° to 126±8° for PP; 124±3° to 147±2° for PVDF and 130±4° to 153±2° for PTFE), reaching the range of superhydrophobicity for the modified fluoropolymers and improving the hydrophobic properties of the modified polyolefin membrane. In terms of operational performance in SWRO brine dehydration, the modified membranes show moderately higher permeate flux yields compared to their commercial counterparts (PP=12%, PVDF=12% and PTFE=15% improvement approx.) maintaining the original membrane rejection levels (>99.95%). These results show potential for application in direct modification of existing membrane materials for water recovery distillation processes and high fouling processes such as membrane crystallization for valuable recovery.

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Mastering membranes

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With the transition to a sustainable, circular economy and a strong emphasis on the recovery and reuse of materials, minerals and nutrients, membrane technology will become increasingly important. Associated to this, the complexity of separations will increase requiring membranes with molecular selectivity suitable for the specific recovery of target valuable components. This comes with demanding membrane characteristics in terms of properties and separation performance. Obviously with this in mind, dedicated membrane design and tunable membrane development and production methods are essential.

Polymer membranes are currently often prepared using methods like phase separation and interfacial polymerization. This allows to a certain extent control over pore size and membrane polymer-component interactions, but real molecular separations are out of reach still as phase separation results in broad pore size distributions and interactions are limited due to the use of only few membrane polymer chemistries. This presentation will give an overview of several, recently developed approaches to tailor membrane selectivity and performance for several sustainable process applications related to water and energy. It connects materials chemistry and interactions with membrane development with characterization and application studies.

The role of surfactants in membrane fouling

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Oil/water separation is a major concern in a wide range of industries. While highly successful at performing the required separation, membranes suffer from fouling by the accumulation of emulsified oil droplets on the surface, resulting in performance loss and increased operating cost. In the present study, we examine the role of surfactants in membrane fouling by means of direct observation via confocal microscopy. Specifically, we assess the correlation between hydrodynamics, surface charge and surfactant size with their effect on interfacial properties and surface interaction. Furthermore, we link the microscopic events observable through the microscope, with macroscopic system pressure-flux behavior. Results correlate membrane performance, i.e. flux losses, with real time 3D images of droplets at the surface of a UF membrane. When the membrane is exposed to a surfactant (TX-100), the system loses 70% of its performance, while the surface is covered with only a few small (<20 µm) droplets. In contrast, separating oil alone results in only 20% flux decline, even after longer times and with far greater fouled surface coverage. From spectrophotometer results of TX-100 absorption in the permeate, it seems that TX-100 is not only absorbing on the surface, but penetrating the membrane. These results motivates re-consideration of fundamental questions regarding the fouling mechanisms of emulsified oil, and their implications for fouling mitigation.

Evaluation of cleaning techniques for Blue Energy fed with natural feed waters using profiled membranes

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Reverse electrodialysis (RED) is a promising technology to harvest the Salinity Gradient Power (SGP) that is available where fresh and seawater mix. Salinity gradients can be used as renewable energy source by applying alternating anion and cation exchange membranes in a RED stack. Fouling of ion exchange membranes is one of the main challenges for RED, since it leads to a reduction in exploitable net power density output [1, 2]. Thus, prevention and management of fouling is essential and can be achieved with pre-treatment and cleaning strategies [2-4]. In this study, we combined the use of profiled ion-exchange membranes that created compartments of 200 µm thickness with an effective pre-treatment strategy using a dual media filter for both water streams. We compared the electrical and hydrodynamic stack performance using new and more established cleaning procedures (i.e., reversed and increased flow, reversed flow and feed switch, increased flow, and air sparging). The goal was to investigate how these cleaning techniques led to a different fouling build up and performance loss during long-term operation (67 days) under real operational conditions (Lake IJssel and Wadden Sea). Cleaning with air sparging achieved better results than other techniques, limiting the pumping losses to minimal values. The cleaning technique with reversed flow and feed switch also kept the pressure drop across the stack lower than 100 mbar for both water streams as long as this technique was applied on a daily basis with complementary air sparging when needed. Post experiment membrane autopsy showed that CEMs were more prone to suffer from particulate fouling than AEMs and that a lower incidence of fouling by particulate matter resulted in a higher concentration of humic acids and biofouling on the membrane surface. This showed that a fouling layer of particulate matter was beneficial for limiting other types of fouling.

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Microfluidic Analysis of Soft Colloid Filtration

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During soft matter filtration, colloids accumulate in a compressible porous cake layer on top of the membrane surface. The void size between the colloids predominantly defines the cake-specific permeation resistance and the corresponding filtration efficiency. While higher fluxes are beneficial for the process efficiency, they propel filter cake compression and increase permeation resistance. Despite the significant impact on colloid filtration, it is not fully understood how soft particles behave during cake formation and how their compression influences the overall cake properties. The shape of the colloids and the morphology of the cake strongly influence permeability and pore size in the cake. We report on spatially resolving the compression of a colloidal filter cake by rejecting soft microgels in a microfluidic channel in front of a pre-clogged model membrane. During cake formation, we analyze particle-particle interaction and single-particle movements inside the filter cake voids and demonstrate the interaction with the whole filter cake morphology. This analysis reveals avalanche-like v-shaped displacement of a particle network inside the cake strongly affecting the cake morphology. To study the single colloid deformation amorphous and crystalline domains were built up and visualized on-line using confocal fluorescence microscopy while adjusting the transmembrane flux. Results show locally pronounced asymmetric deformation in amorphous domains, while the microgels in colloidal crystals approached regular polyeder shape. Finally, we demonstrate fabrication of microscopic 3D particles with complex shape. From these particles assemblies with various particle shapes are built and analyzed regarding their permeability and morphology, representing convective accessibility of the assembly's porosity. This work links single colloid movements and filter cake compression to the overall cake behavior and narrows the gap between single colloid events and the filtration process. And we present a precise hydrodynamic analysis method, that enables research on adaptive, responsive, and permeable 3D micro-particle assemblies.

Oil-infused feed spacers for biofouling control in spiral-wound membrane modules

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Biofouling of spiral-wound modules used in membrane-based desalination and water purification can dramatically impact the process efficiency and cost-effectiveness. Studies have reported that initial biofilm formation occurs along the feed spacer and, with time, encroached upon the remaining membrane area. Current strategies to prevent and control biofouling include modifications of the feed spacers. However, in many cases, these strategies rely on coatings that produce a short-term effect. In the present study, we propose a different approach for modifying feed spacers, utilizing oil-infused slippery substrates based on polydimethylsiloxane (PDMS). PDMS feed spacers can be infused with silicone oil to create a stable, extremely slippery interface that exhibits remarkably low bacterial adhesion, particularly under flow conditions. The effect of the silicone oil viscosity on biofilm formation was examined, as well as the PDMS cross-linking, based on inhibition of bio-growth on the feed spacers. As viscosity decreases, oil absorption into the substrate increases, and the minimal oil effusion allows optimal wetting of the surface over a long period, inhibiting bacterial adhesion. Moreover, adhered bacteria can be readily rinsed off the lower viscosity-infused PDMS surface, while attachment to non-infused surfaces is irreversible. The infused PDMS feed spacer demonstrated extraordinary biofilm inhibition: the 20 cSt oil infused PDMS feed spacer accumulated approximately two orders of magnitude less biovolume than the control feed spacer. On the modified spacers, biofilm is observable in very few areas, primarily as a thin layer, distinctively different from the morphology seen on the control feed spacer. This biofouling-resistant feed spacer can be used to prolong membrane life and reduce membrane maintenance in desalination processes.

Measuring the local compaction of organic foulants due to pressure-driven permeate flux

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Membrane fouling, namely, the deposition of particles, colloids, and macromolecules on the membrane surface, represents the main operational drawback of membrane-based filtration. In a pressure-driven process, operating conditions can affect the properties of foulant layers composed of soft organic material. Particularly, the deposited layer can deform and compact under increased transmembrane pressure (TMP) and permeate flux. This compaction has been reported in the literature for organic- and bio- fouling and analyzed using noninvasive imaging technologies to measure the foulant's overall thickness. However, the compaction behaviour within the foulant is unknown, leaving unresolved questions: 1) Is compaction locally homogeneous or depth-dependent? 2) How does the compaction, and its profile, depend on the TMP/permeation? 3) How do these influence the foulant's hydrodynamic resistance? Here, we present a new technique to analyze, in-situ, the local compaction and stratification of organic fouling layers due to the combined effects of the TMP and permeate flux. The method can detect, with high precision, variations in the internal deformation of the examined foulant, while varying the operational conditions and simultaneously measuring the foulant's hydraulic resistance. The experimental setup is based on a custom-made flow-cell mounted on a confocal laser scanning microscope. 3D tracking routines are employed to measure the displacements of fluorescent micro-tracers trapped within the soft material, locally mapping the compaction. Results obtained for calcium-alginate layers deposited on nanofiltration membranes indicated a transition from depth-dependent to homogeneous compaction under TMP stepping experiments. Moreover, the profile of depth-dependent compaction indicated that foulant layers near the membrane surface are stiffer, pointing to a foulant stratification. Finally, this technique was employed to examine the effects of hydrostatic pressure and TMP on the foulant compaction, revealing the coupling between the TMP and the generated permeate-flow and their interplay in dictating the compaction and resultant hydraulic resistance.

The future of ultrafiltration: Paradigm change and economic prospectives

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Human beings are rarely prepared for changes before they occur, even if they forsee them and try to develop strategies to overcome them. Why? Because they shape their objectives with the mind set of the context they live in. This philosophical question applies to ultrafiltration (UF) process; consider fouling as a problem is not adapted anymore to the coming challenges, i.e., energy shortage and lack of raw materials. With our research consortium, we propose to the membrane community to change the paradigm of UF fouling; we should favor the development of a biofilm at the surface of the membrane, not fight it! This can reduce energy consumption of UF and improve the permeate quality for higher post-treatment efficiency. However, several challenges should be tackled for the implementation of such approach at centralized scale: (1) ensure stable flux with compact membrane geometries such as inside-out hollow fibers, i.e., low space available for biofilm growth, (2) maintain constant high biopolymer removal and (3) reduce the membrane capital expenditures, given that low fluxes lead to higher membrane surface area requirements. In this presentation, we will reveal our latest results demonstrating that these three challenges can be successfully tackled and that tolerate a biofilm at the surface of ultrafiltration membranes is an economically viable alternative to conventional UF.

Key differences in the rejection of solutes in large- and bench-scale membrane distillation

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Membrane distillation (MD) is a potentially suitable process for treating reverse osmosis concentrate to produce distillate and reduce volume of concentrate to be disposed. Theoretically, MD is also not limited by the concentration of water to be treated and can achieve 100% rejection of non-volatile components because only the vapor phase permeates through the membrane. However, as MD is scaled-up, heat losses increase resulting in a lower driving force and water flux [1]. Additionally, a larger membrane area increases probability of transport of contaminants through pore defects. Therefore, with scale-up, higher concentrations of feed lowers distillate production rate and quality, a phenomenon not prominently observed at bench scale. In this work, membrane rejection of non-volatile components in a pilot-scale air-gap MD element is measured at varying operating conditions. Rejection of organics and salts has been reported in several studies but its resolution is limited by the solubility of the contaminant in the feed and the detection limit of contaminants in the distillate. Bacteriophages MS2 and PhiX174 were spiked (4x105 PFU/mL concentration) to overcome the solubility limit of salts in the feed and achieve high feed concentration. Membrane rejection of bacteriophages, cations and organics was quantified at a constant flowrate and temperature difference between the feed and distillate (40/20 °C) and low and high vacuum in the air gap. Membrane rejection of bacteriophages was observed to be 0.5-log10 higher than cation rejection but 4.5-log10 lower than rejection observed at bench scale [2]. Rejection of organics, inorganics and bacteriophages reduced with increase in air gap vacuum, which suggests presence of pore flow through pore defects. Interestingly, exposure to high feed temperature (70 °C) did not lead to an instantaneous decrease in feed virus concentration which suggests that longer operating times are required to decrease virus concentration in the feed.

Acknowledgements

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Enhancement of Air Gap Membrane Distillation Performance by Incorporation Clay Nanoparticles in a PVDF Matrix Membranes for Water Desalination

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Membrane distillation (MD) is an emerging technology for treating seawater, brackish water, and industrial wastewater. MD uses the vapor pressure difference between both sides of a porous hydrophobic membrane as the driving force, allowing only the transport of water vapor molecules through the membrane's pores. However, MD research still faces some important challenges with regards to engineering efficient hydrophobic membranes with antifouling and antiwetting properties. The introduction of nanoparticles can impart the desired antifouling and antiwetting characteristics, while improving in some cases other properties such as porosity, mechanical strength, or hydraulic performance. Clays have attracted considerable attention because they are highly compatible with polymers and are relatively abundant In this work, phase inversion polyvinylidene fluoride (PVDF)-clay mixed matrix membranes were prepared, characterized and evaluated. Montmorillonite, muscovite, and silica-nanosheets were used as fillers, and the prepared membranes were applied to treat synthetic saline solutions containing NaCl and DI water via an air gap membrane distillation (AGMD) configuration. Different concentrations of clay nanoparticles, ranging from 0.5 to 10 wt.% (final concentration in the prepared membranes after solvent removal), and non-toxic solvent dimethyl sulfoxide (DMSO) in the dope solutions, were used. The membranes were prepared using the immersion precipitation technique in a water coagulation bath at a temperature of 25 °C. The presence of clay particles was confirmed by scanning electron microscopy with an energy-dispersive X-ray spectroscopy (SEM-EDX) analysis. Other characterization techniques such as water contact angle, liquid entry pressure, porosity, and pore size analysis were carried out to link MD membrane performance enhancement to their morphology and other structural properties.

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NiSe and CoSe-based thermoplasmonic platform for a sustainable desalination at the water-energy nexus

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The concern about the necessity to produce freshwater from seawater without stressing energy supplies (water-energy nexus) has led Reverse Osmosis (RO) as the prominent technology in desalination. In the recent year, Membrane Distillation (MD) has emerged as complementary hybrid thermal/membrane technology able to intensify the desalination process by post-treating the brine rejected from RO plants resulting in an improvement of the water recovery factor from ca. 50% to above 90%. However, the practical implementation of MD has been hindered by its poor thermal efficiency due to temperature polarization (TP)- mostly related to the removal of latent heat associated to water evaporation - raising the specific energy consumption to 200-300 $kWh \cdot m^{-3}$- roughly 2 orders of magnitude higher than RO [1]. The advent of photothermal nanomaterials- able to efficiently absorb the radiative energy subsequently dissipated by locally releasing heat (light-to-heat conversion)- opened unprecedented horizons for solar-driven MD revolutionizing the water-energy nexus. Our recent works demonstrated that photothermal nanoparticles (i.e. noble metals [2], graphene[3]) act as nano-heaters upon artificial or natural light locally heat harvesting at the membrane surface. Herein, NiSe and CoSe nanoparticles-characterized by localized surface plasmons matching with sunlight radiation- were immobilized into a coating of Polydimethylsiloxane (PDMS) on the surface of Polyvinylidene fluoride (PVDF) via spray coating. Noteworthy, the photothermal response of both NiSe and CoSe under the solar radiation provided to the membranes self-heating properties exploitable to boost the water evaporation rate. For Vacuum Membrane Distillation (VMD) operating under the solar radiation with artificial seawater, bare PDMS/PVDF membrane showed a transmembrane flux of ca. 2.6 $kg \cdot m^{-2} \cdot h^{-1}$ increased by 330% and 690% when NiSe and CoSe NPs were embedded into the silicone coating, respectively. Definitively, the cutting-edge concept of the solar-driven MD opens the door to a sustainable solar-driven desalination at the water-energy nexus.

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Ammonia recovery from liquid digestate - practical experience and cleaning requirements

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Introduction:

Ammonium removal from liquid digestate is a promising mean to deploy circular economy options for biogas plants and municipal waste water treatment plants. While our past work showed implementation of membrane distillation (MD) for ammonium removal and recovery as ammonium sulfate from centrate water in continuous 24h operation, this work evaluates MD for ammonium removal from digestate and goes one step further to include results on cleaning means and their effects on membrane characteristics as well as performance for centrate water and digestate foulants.

Methodology:

Ammonium recovery from liquid digestate and synthetic ammonium water was tested in a flate plate membrane distillation module equipped with 2 m² PTFE membranes (pore size 0.45 µm). The diffused ammonia was collected as ammonium sulfate on the permeate side. Temperature conditions ranged from 25 to 38°C and pH varied from 8.7 to 9.7. Data on temperature, pH and conductivity was constantly logged throughout the experiments and ammonia removal was additionally analysed by photometric NH4-N detection.

For cleaning tests, membranes after usage of ammonium removal tests from centrate water and digestate were cleaned and re-applied for ammonium removal tests. Different cleaning agents are evaluated and membranes are characterized by their performance after cleaning and via FTIR and SEM-EDX analysis.

Results:

This paper shows how ideal operating conditions evaluated for centrate water ammonium removal were further applied and extended for digestate treatment. Limits on digestate viscosity were faced in the lab plant, requiring dilutions for certain digestate. Ammonium removal was ranging between 65-99% after 3.5 hours. Finally the choice of feed temperature was less important, leaning towards a tendency to work at lower temperatures in order to minimize process energy input. The cleaning results suggest the necessity for mild acid cleaning for both applications (centrate water and digestate) to ensure continuous processing at high performance.

Membrane microgravity humidity separator

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The water harvesting from aeriform streams by membranes is a recently developed operation, which is attracting attention in these last years for recovering water by the dehydration of, e.g., cooling tower and flue gas streams. A device was designed and built for operating in steady state considering its next utilization in microgravity. In a typical application on earth, the membrane unit is vertical oriented, and the condensed water is usually collected on the bottom of the condenser since it falls. The appropriate design of the membrane module together with the suitable choice of membrane type and related configuration (i.e., capillaries) are indeed crucial aspects that have been considered to make this technology successful also in microgravity. Thus, specific solutions have been investigated to promote water harvesting for different orientation of the membrane humidity separator. Hydrophobic porous membranes (pore size of 0.1-0.2 microns) were utilized for harvesting (on feed membrane side) the liquid fraction of water - even the small size particles - contained in aeriform streams, allowing the permeation of water vapour and permanent gas through the membrane. The lecture will also discuss obtained results with the designed separation unit as a function of the main variables affecting the separation process such as, e.g., humidity, orientation, and feed location. The overall efficiency in separating the liquid water exceeds 99%. All variables and parameters play an important role in the specific field of interest in microgravity; therefore, specific attention has been paid to pressure drop through the membrane thickness. In microgravity, one of the most significant challenge to be faced is the removal of separated liquid water from the membrane module since the absence of gravity does not allow the water dragging. This requires specific measures in designing of the membrane unit, which also includes geometrical and fluid-dynamics considerations.

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Improvements in membrane distillation towards brine concentration

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The main limitation for implementing membrane distillation (MD) is its high thermal energy consumption [1]. It has been demonstrated that using multi-envelope spiral-wound modules in air-gap configuration with long channels maximizes the internal latent heat recovery and minimizes the specific thermal energy consumption (STEC) [2]. However, there is a trade-off between permeate flux and thermal efficiency and the minimum STEC corresponds to the lowest fluxes. The use of light vacuum to extract air from the gap enhances the permeate flux reducing the STEC to values as low as 40 kWhth/m^3 for seawater, with corresponding flux about 1.0 L/(h·m^2) [3]. As feed salinity increases, vapour pressure decreases, and in these long modules the transmembrane gradient of vapour pressure, which is the driving force, can be too low, resulting in no permeate flux. Moreover, scaling and membrane wetting increases. This study reviews how different large-scale MD modules perform with high salinity feeds, and quantifies the positive influence of using vacuum to improve the performance towards brine concentration. Vacuum-assisted air-gap configuration achieves the best energy performance but with low recovery ratios, which can be increased by recirculation in batch operation. The use of vacuum multi-effect configuration benefits from recovering the latent heat of condensation in successive evaporation processes (effects), allowing to increase permeate flux and heat efficiency at the same time. However, the maximum number of effects is limited by the boiling point elevation for high salinity feeds. Flux, total energy consumption and membrane wetting are assessed in all cases. The results are further particularized for the treatment of high salinity brines from a mine effluent, in the context of a combined treatment with reverse electrodialysis and membrane distillation proposed in the EU project Intelwatt, using solar energy as heat source. Expected performance figures are given and discussed.

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The Road from Novel Membranes to Pilot Demonstration: Recovery of Value-added Difluoromethane from Mixtures of High-GWP Fluorinated Hydrocarbons

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To date, most working fluids used in vapor-compression cycles consist of azeotropic or close-boiling mixtures of high-GWP fluorinated hydrocarbons (HFCs) that balance the environmental, energy performance and safety trade-offs. With a growing demand for refrigeration and air-conditioning and the tight restrictions being imposed on the production of HFCs, great interest has arisen for recovering exhausted refrigerants from end-of-life equipment and recycling the most valuable compounds [1]. Particularly, the recovery of difluoromethane (R32) is sought as it is being widely employed in the production of novel refrigerant mixtures due to its excellent performance and mid-GWP. In this context, we have explored the feasibility of separating R32 from some common refrigerant mixtures using membrane technology. Our initial works focused on the use of poly(ether-b-amide) copolymer (PEBA) membranes [2] and the engineering of composite ionic liquid polymer membranes (CILPMs), which proved to be a powerful approach to further increase gas permeability and selectivity towards R32 [3]. However, most polymer/IL membranes are tested as self-supported thick dense films that are not attractive for an industrial application, whereas the production of free-IL containing thin film composites (TFCs) remains quite an unexplored field of research. In this sense, we have taken a step forward by fabricating polymer/IL TFCs with simple coating techniques, in flat and hollow fiber configuration, that have demonstrated to retain the separation ability of the flat thick membranes while providing much higher permeate fluxes. The flat PEBA/IL-TFCs were scale-up to test the recovery-purity trade-off as a function of applied feed pressure. Eventually, the results were used to model the membrane separation in Aspen Custom Modeler and transferred to Aspen Plus to design a multistage separation process and optimize the operation conditions to reach the target purity of 99.5 wt.% R32 in the permeate stream.

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Matrimid dual-layer hollow fiber membranes for oxygen-nitrogen separation by gas permeation

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In recent years, the need to reduce energy consumption worldwide to move towards sustainable development has led many of the conventional technologies used at the industry to evolve or to be replaced by new alternatives. Obtaining oxygen from air is one of the most interesting separations due to the wide range of applications offered by this compound at industrial and medical level. So far, the most widely used techniques to obtain oxygen are cryogenic distillation and pressure swing adsorption, both of which require large amounts of energy [1]. In this sense, membrane separation technology is presented as a more efficient alternative, since it overcomes many of the barriers presented by traditional technologies. One way to make membrane technology competitive in oxygen separation is the implementation of hollow fiber membrane modules, instead of the current flat-sheet membrane configuration [2]. The aim of this work is to develop cost-effective advanced multilayer hollow fiber composite membranes made of Matrimid and polydimethylsiloxane (PDMS) for the separation of oxygen and nitrogen from air. PDMS is used as a cover layer, but can also offer performance enhancement of the membrane. In order to compare these two materials, three different configurations are studied. First of all, Matrimid hollow fiber membranes were produced using the spinning method. Secondly, by using dip-coating method, a PDMS dense selective layer was deposited on a self-made polyvinylidene fluoride (PVDF) hollow fiber support. Finally, the performance of a dual layer hollow fiber membrane of Matrimid and PDMS was studied. Membranes morphology was characterized by SEM and separation performance of the membranes was evaluated by gas permeation experiments. Membranes prepared in this work were compared to the current state of the art reporting an outstanding performance for the dual-layer membrane, reaching O_2 permeance of 30.8 GPU and O_2/N_2 selectivity of 4.7.

Acknowledgements

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Mixed matrix membranes based on MIL-101(Cr) and MIL-177(Ti) in PIM-EA (Me2)-TB and PIM-TMN-Trip tailored for efficient H_2 separation

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Polymers of intrinsic microporosity (PIMs) are attractive in gas separation for the excellent combination of their high permeability and good selectivity [1]. A further synergistic improvement can be reached by the addition of metal-organic frameworks (MOFs) to prepare the so-called mixed matrix membranes (MMMs). The aim of this study is to produce novel MMMs, based on the dispersion of two different MOFs, namely MIL-101(Cr) and MIL-177(Ti), in two PIMs: the PIM-EA(Me2)-TB [2] and the ultra-permeable PIM-TMN-Trip [3]. The influence of the MOFs on the gas permeability, diffusivity and solubility is investigated after different post-treatment protocols of the membranes. The pure gas transport is discussed on the basis of the Maxwell model. The permeability of the MMMs increases with increasing MOF loading, and the absolute values depend on the specific PIM/MOF combination. This increase is mainly due to a higher diffusion coefficient, which indicates that cavities in the MOF provide a preferential pathway for the gases. This effect is more evident for smaller gas molecules, and therefore the selectivity increases especially for those gas pairs with a large difference in kinetic diameter, such as H_2/CH_4 and H_2/N_2. The PIM-EA(Me2)-TB/MIL-101 membrane with 20 wt% MOF showed the best results for the H_2/N_2 separation, with a strong increase in H_2 permeability from ~6000 to 13000 Barrer and H_2/N_2 ideal selectivity from 14 to 21. The MIL-177(Ti) based MMMs, instead, showed an increase of the H_2/N_2 selectivity up to 19, without a significant change in H_2 permeability. A complete overview of the gas transport properties will be given, and the effect of long-term aging of the membranes will be discussed.

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Ammonia Separation via Immobilized Molten Salt Membrane

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Ammonia has been called one of the greatest inventions of the 20th Century. Since 1905, ammonia has been the basic feedstock for inorganic fertilizers supporting food production for approximately half of the world's population [1]. Today it is garnering further attention as a flexible long-term energy carrier and zero-carbon fuel. Despite its critical role to food production and great promise in alternative fuels, ammonia production through the Haber-Bosch process has seen little refinement. Currently, ammonia for fertilizer is produced at 400-500 °C and at pressures up to 30 MPa [2]. The high temperature, necessary for rapid kinetics, restricts conversion to ~20 percent [3]. As a result, the recovery of ammonia from the reactants mixtures of nitrogen and hydrogen is a critical unit operation in the production plants. A series of heat exchangers and a final refrigeration stage are usually used to recover ammonia, while the remaining gases are reheated and recycled to a catalytic converter. Membrane technology provides an opportunity to intensify the Haber-Bosch process by the use of an ammonia–selective membrane. On this regard, it is important to identify a membrane stable at high temperature and highly selective towards ammonia permeation with respect hydrogen and nitrogen. This study aims to investigate the performance of zinc chloride immobilized molten salt membrane (IMS) due to its stability at high temperature and its unique characteristics of showing reversible ammonia absorption at high temperature. The IMS is synthesized in the lab and characterized via permeation testing at a temperature range of 300-400 °C. In particular, permeation characteristics of the IMS are evaluated by using pure gases, binary and ternary mixtures. The SEM and EDS analyses on the pristine and used IMS are performed to analyze the physical and chemical properties modification prior and after testing.

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Synthesis of SAPO-34 membranes using Microwave

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In the context of energy transition, biogas could be an essential part of it. Biogas contains mainly methane and carbon dioxide with some impurities like water, dihydrogensulphide and oxygen. For the transportation of methane in the natural gas grid a purification step is necessary to separate carbon dioxide from methane. For that purpose a membrane based approach was chosen using zeolite membranes. In particular the SAPO-34 membranes are very suited for the separation task. This work deals with the preparation of SAPO-34 membranes using a microwave assisted approach coupled with some advantages, namely an easy handing, generation of many results per time and it's excellent for powder synthesis. Membranes with a length of 105 mm were synthesized and characterized by single and mixed gas measurements. Different parameters (temperature, time, synthesis cycles) during the synthesis were changed to optimize the results. CO_2-permeances of more than 20 $m^3/(m^2hbar)$ and CO_2/CH_4 permselectivities of more than 200 were reached. The powders were used as seeds for membrane preparation in conventional hydrothermal synthesis and scaling up of membrane length up to 250 mm in single channel tube geometry.

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Application of simultaneous monitoring of the upstream pressure decay and downstream pressure rise in the novel constant volume testing system to characterize glassy polymer films

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Glassy polymers are commonly used to prepare gas separation membranes. Gas transport in glassy polymers occurs by a solution-diffusion mechanism and dual-mode sorption. In the simplest case of dual-mode sorption polymer membranes, i.e. complete immobilization and instantaneous equilibrium between Henry's and Langmuir's sites, the complete membrane characterization requires the determination of the four parameters: solubility (S) and diffusivity (D) coefficients in the mobile phase (Henry's sites) and the saturation (CH') and the affinity (b) constants in the immobile phase (Langmuir sites). The determination of these four coefficients requires four independent dynamics (time-lag) gas permeation tests performed at four different feed pressures. We have provided the theoretical foundation for the characterization method that would allow the determination of S, D, CH' and b in two instead of four-time lag tests. However, the method requires simultaneous monitoring of the instantaneous downstream (TLD) and upstream (TLU) time lags and their ratio as a function of time [1]. Although the determination of TLD is well established, the determination of TLU requires accurate monitoring of pressure decay at the high-pressure side due to gas permeation into the membrane, which is very challenging. This study presents a novel constant volume (CV) gas testing system that allows accurate monitoring of pressure decay. The latter is accomplished by comparing the variable pressure in the working volume (due to gas permeation into the membrane) with the constant pressure in the reference volume after initiating a dynamic gas permeation test [2]. We compare the theoretically predicted ratio of the TLU and TLD as a function of time with experimentally observed values for the experiments at different feed pressures. The ratio of the TLU and TLD continues to change even after reaching a pseudo-steady state because the pressure changes upstream and downstream of the membrane.

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Evaluation of different Atomic Layer Deposition techniques for pore size tuning of ceramic membranes from ultra- to nanofiltration

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Many industrial processes often produce large amounts of aqueous wastes contaminated with solvents and other environmentally hazardous materials. Such effluents need to be treated before they can be discharged into the natural water bodies. The present treatment methods are based on energy-intensive technologies like distillation, evaporation, and drying. Implementation of nanofiltration (NF) membranes in treating such effluents can lead to significant energy savings – up to 90 % less energy than distillation [1]. NF membranes have been used in applications including food industry, pharmaceutical, wastewater treatment, and desalination industries before. However, the employment of commercially available NF polymeric membranes is hampered due to their severe fouling and instability of the membranes in the aggressive effluent media [2]. In the past, ceramic membranes have shown great potential for applications in aqueous solvent-contaminated waste streams. One of the main challenges in the production of porous ceramic membranes is obtaining a narrow pore size distribution in the nanofiltration range (pore diameter <2 nm) [3]. The potential of applying Atomic Layer Deposition (ALD), an established self-limiting gas-phase deposition technique for growing atomic-scale thin films, has been studied for narrowing ceramic membrane pores to the nanofiltration range (<2 nm). The study aims at gaining more knowledge how pores are modified during deposition of TiO_2 (from $TiCl_4$ and H_2O), with two different ALD techniques, temporal and spatial ALD (t-ALD, s-ALD). Experiments were performed to observe the effect of the most critical process parameters on the pore size reduction. Both techniques resulted in significant narrowing of the pores (MWCO from 10 kDa down to < 300 Da) and can be used for the production of ceramic nanofiltration membranes wherein t-ALD had the advantage that pores could be decreased with better control from 5 nm to below 1 nm while the spatial ALD process benefited from the high throughput.

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Ultrafiltration of nanoemulsions with state-of-art silicon carbide-alumina membranes

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In the past decades a large amount of oily wastewater has been produced from oil extraction, petrochemical, metallurgical, pharmaceutical, food, and beverage industries [1]. Most oil-in-water emulsions have oil droplet sizes ranging from a few nanometers to several hundred micrometers. For emulsions with micron-sized oil droplets, ceramic membranes show satisfactory rejection, typically in the range of 90-99 percent. Some industrial operations generate large quantities of oily wastewater with oil droplets from 2-200 nm [2]. These nano-sized oil droplets are far more difficult to separate because of their smaller size and higher colloidal stability. Silicon carbide (SiC) membranes are preferred for oil/water nanoemulsion separation. Nevertheless, SiC membranes are often fabricated using the sol-gel technique at a high temperature with excessive production time and expenses [3]. Therefore, we established a simple and cost-effective method for producing silicon carbide-alumina ultrafiltration membranes based on low pressure chemical vapour deposition (LPCVD). In this method, SiC was deposited on 100 nm alumina membranes using two precursors (SiH_2Cl_2 and C_2H_2). Different deposition times varying from 10 min to 30 min were used to tune membrane pore size and surface property. The pure water permeability of the membranes decreased from 360 L m^{-2} h^{-1} bar^{-1} to 120 L m^{-2} h^{-1} bar^{-1} when the deposition time was increased due to the reduction of membrane pore size and porosity. The nanoemulsion stabilized by Tween 80 and Span 80 was slightly negatively charged with zeta potential at -21.3 ± 0.73 mV and the average oil droplet particle size of is 80 nm. Nanoemulsion filtration experiments were carried out at a constant permeate flux (100 L m^{-2} h^{-1}) and results revealed that membrane fouling reduced significantly after modification due to improved hydrophilicity and charge interactions.

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Thin zeolite imidazolate framework films for membrane application

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The development of a rapid and reproducible crystallization route for the synthesis of polycrystalline metal–organic framework (MOF) films is attractive for the scalable production of nanoporous membranes on porous supports. Zeolitic imidazolate framework (ZIFs)1 owing to its rich structure diversity and various pore sizes, have been widely used for constructing molecular-sieving membranes. However, the three-dimensional crystalline and anisotropic nature of ZIFs has made challenging to synthesize sub-100-nm thick continuous ZIF films by simple and rapid synthesis protocols. Here, we report a macroscopically large ulttrathin ZIF membranes by simple route with synthesis time smaller than 10 min at the room temperature. We discuss two approaches for crystallization. In approach one, we report a crystallization using sustained precursors (CUSP) route that maintains a high precursor concentration in the growth step, hindering the undesired Ostwald ripening observed in the late stage of growth. In approach two, we hinder out-of-plane growth of the film forming ultrathin membranes. Attractive gas separation performance is obtained from ZIF-8 membranes grown in 8 min with H_2/C_3H_8 selectivity of 2433 and C_3H_6/C_3H_8 selectivity of 30. The versatility of this approach is demonstrated by synthesizing a ZIF-67 membrane, as well as for the first time, a sub-1 mm-thick continuously intergrown ZIF-90 membrane, also in a few minutes, yielding H_2/CH_4 and H_2/C_3H_6 selectivities of 19.2 and 107.1, respectively. Such advances are expected to bring the scalable production of the high- performance polycrystalline MOF membranes a step closer to reality.

Porous ZIF8-SiO2 Nano-Filtration Membranes for Water Purification

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With the rapid development of industry and intensive agriculture, increasing volumes of wastewaters are discharged into the environment, causing large-scale pollution of water resources with a potential negative impact for aquatic life and humans. Membrane nanoniltration (NF) exhibits high performance in water depollution1. Metallorganic frameworks (MOFs) have received substantial attention as nanomaterials for molecular separation systems, owing to their intrinsic porous characteristics, unique chemical versatility and functionalities.[2] Among MOFs, ZIF8 presents a theoretical accessible window aperture of 0.34 nm and a cavity of 1.11 nm in diameter, offering great potential in water filtration3. In order to get higher mechanical, structural and thermal stability, in this project, we focus on the development of NF membranes through a combination of crystaline ZIF8 and sol-gel derived amorphous silica. Specifically, ZIF8 nanocrystalls were embedded into a ZrO_2-doped SiO_2 matrix. An optimized ZIF8-SiO_2 thin layer (<1µm) was deposited on a porous TiO_2 substrate by using the dip-coating method. The silica was doped with ZrO_2 to increase the membrane performances and stability during filtration process. The combination of the SiO_2 with the ZIF8 crystals allowed to combine high porosity, mechanical strength in an easy fabrication procedure. This research provides a new way to make new NF membranes for water purification through a combination of amorphous and crystalline materials.

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Zeolite membranes and more - The importance of support analysis

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Zeolite membranes are highly attractive in energy efficient, selective separation technologies. Their high selectivity originates from selective adsorption, diffusion and even molecular sieving. The asymmetric high-flux zeolite membranes (> 1 mol s–1m–2) with a sub-micrometer thickness are mechanically stabilised by a porous, often multilayer, support. Transport mechanisms in zeolite layer and support, however, are counteracting regarding selectivity, and a support may also act as a flux resistance. In the development or improvement of selective materials the analysis of the permeation and separation performance of membranes measured on the labscale is a crucial exercise for quantifying quality and transport modelling. Several examples are analysed quantifying the impact of the support on the observed performance, showing the effect of layer thickness, orientation of the asymmetric membrane and operational conditions, resulting in recommendations for the configuration of gas separation membrane modules and specifically zeolite membranes.

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Could 2D graphene-based materials be potential candidates for membrane materials?

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Two-dimensional (2D) materials of atomic thickness have emerged as nano-building blocks to develop high-performance separation membranes that feature unique intrinsic nanopores and/or assembled nanochannels. These two-dimensional-material membranes exhibit extraordinary permeation properties, opening a new avenue to ultra-fast and highly selective membranes for water, ion and gas separation. The atom-thick 2D materials stimulates the continuous pursuit of ultrathin and selective membranes for separation, regardless of fundamental and engineering studies. Does recent research indicate that 2D-material membranes are a temporary curiosity or a new family of high-performance separation membranes? In this presentation, this question will be discussed by analysing our recent progress in the design and fabrication of either nanosheets or laminar 2D-graphene based membranes for high efficiency membrane separation. Fundamental and technical challenges for advancing 2D-material membranes for liquid and gas separations will be also prospected.

Biocatalytic regenerated-cellulose membrane hyperactivated by surfactants

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Synthetic membranes are promising supports for enzyme attachment, because they provide microenvironment that can mimic native conditions, they offer high specific surface for immobilization and they are available in different materials. Enzymes immobilization on/into membranes is an appealing strategy to develop efficient and sustainable biohybrid devices for inactivation/detection of xenobiotics. However, fundamental understanding of the factors affecting the performance of biohybrid membranes as well as find out methods to improve their activity/stability is crucial to promote this technology. Material physical-chemical properties, membrane structure and topography have a key role on enzyme activity/selectivity [1]. A method for tuning and improving biocatalytic membrane performance is the use of surfactants [2]. In this work the activity/stability of a biocatalytic membrane, prepared by covalently immobilizing a thermophilic phosphotriesterase on regenerated-cellulose, in the presence of different surfactants was studied. Cetyltrimethylammonium bromide (CTAB, positively-charged), sodium dodecylsulphate (SDS, negatively-charged) and Triton X100 (TX100, not-charged) as model surfactants were selected. The kinetic parameters were determined and compared in the absence and presence of the different surfactants. Results revealed that the activity of the membrane-loaded phosphotriesterase towards the substrate (paraoxon-ethyl) was hugely improved in the presence of SDS and CTAB (88% and 77%, respectively), whereas TX100 showed a less effective boosting ability (40%). A similar trend, obtaining an even higher increment of activity, was observed using the free phosphotriesterase. The kinetic studies pointed out an increase of the catalytic constant of the immobilized enzyme due to the surfactants action. Similar results were obtained for the free phosphotriesterase, furthermore, in this case a decreasing of the KM was revealed. To further investigate the underlying mechanism responsible of the enzyme hyperactivation, DLS, native electrophoresis, zeta potential and CD measurements were carried out. The present study offers deep understanding of surfactants boosting action on the catalytic behaviour of the biocatalytic membrane.

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Biocatalytic Ceramic Membranes for Emerging Pollutants Transformation

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Biocatalytic nanofibrous membranes, integrating the benefits of membranes and enzymes, have drawn attention in wastewater treatment due to their high specificity and their operational catalytic stability. Polymeric membranes, as opposed to brittle ceramic membranes, are more commonly used as scaffolds for enzyme immobilization. However, polymeric membranes are typically non-biodegradable, which raises environmental concerns. Here, we report the fabrication of electrospun highly flexible SiO_2 nanofiber membranes (NFMs) and the covalent immobilized of the green industrial biocatalyst, laccase (EC 1.10.3.2). Several strategies for surface modification and functionalization of the NFMs were evaluated to improve the catalytic properties of the NFMs, in terms of enzyme load and apparent activity per membrane weight. Enzyme immobilization with the co-deposition of polydopamine (PDA) and polyethyleneimine (PEI) resulted in the best immobilization yield of 57.9±0.5% and apparent activity of 6.4±1.1 U g^{-1} membrane. Compared to the free enzyme, the fabricated catalytic membranes possessed good reusability, with 70% of their activity retained after five cycles. In addition, the immobilized laccase transformed the emerging pollutants diclofenac, mefenamic acid, sertraline, bicalutamide, and clarithromycin at >95% efficiencies. This is the first study to employs biocatalytic flexible ceramic nanofibers in wastewater treatment, thereby bringing novel insight into a previously less explored realm of biocatalytic membrane applications. Our work offers a technological platform for improving the sustainability of biocatalytic membranes, as compared with the end-of-life polymeric membranes modules.

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Porous poly(ionic liquid)-PVDF composite membranes with immobilized carbonic anhydrase and their application in CO_2 separation by gas-liquid membrane contactors

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In our current climate crisis, an impressive global effort is being deployed for the development and implementation of technologies to mitigate CO_2 emissions. For example, CO_2 capture and storage/utilization, CCUS, will be fundamental during the energy transition [1]. In the context of CCUS, the enzyme carbonic anhydrase, CA, has gained growing attention because it acts as green catalyst in CO_2 capture by absorption [2]. A few works investigated the preparation of membranes with immobilized CA for CO_2 capture by absorption in gas-liquid membrane contactors [3]. Our work addresses the fabrication, characterization and testing of novel composite membranes with immobilized CA for CO_2 capture. The membranes were comprised of an hydrophilic poly(ionic liquid), PIL, top layer coated on a porous PVDF support. The layer was prepared by in-situ polymerization of an ionic liquid monomer solution casted on the PVDF. CA was easily immobilized on the PIL layer by physical adsorption. The catalytic activity of the immobilized enzyme was evaluated in order to identify the optimum formulation of the PIL layer (monomer and cross-linker concentrations) and immobilization protocol (enzyme concentration in the buffer solution and pH). Despite of the simplicity of the immobilization protocol, the enzyme was strongly bounded as the membranes could be reutilized 10 times while retaining above 85% of their original activity. Among the possible mechanisms of immobilization are ionic forces due to the opposite charge of the enzyme and the PIL coating according to Zeta potential measurements. These membranes were further characterized by SEM, EDX, ATR-IR and water contact angle. Moreover, to demonstrate their applicability, the biocatalytic composite membranes were tested in a gas absorption set-up showing an increase in the overall mass transfer coefficient with respect to the pristine support by a factor 2.5. The effect of solvent concentration and liquid flowrate in the absorption performance were also investigated.

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Liposome-integrated Seawater Reverse Osmosis Membrane: From Lab-scale Membrane Coupon to 2.5-inch Spiral Wound Module

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We demonstrated the effectiveness of using spray-assisted interfacial polymerization (IP) technique in fabricating liposome-integrated thin-film nanocomposite (TFN) membranes for seawater reverse osmosis (SWRO) [1]. First, the spray-assisted IP protocol was optimized by embedding liposomes into the PA layer. The vesicle loading within the selective polyamide (PA) layer of the TFN membrane can be precisely controlled by manipulating spraying conditions to eliminate the wastage of precious additives compared to the conventional blending method. Spraying the liposomes after applying the aqueous phase of M-phenylenediamine (MPD) could prevent the loss and disruption of liposome distribution over the substrate layer. Upon incorporating 4 mg/m2 of liposomes, the best-performing liposome-integrated membrane can achieve 27% higher water permeability than the liposome-free membrane, demonstrating a permeability of 3.24 L/(m^2·h·bar) along with 99.3% NaCl rejection for seawater desalination. Characterization showed that incorporated liposomes had affected the IP process by creating a larger miscible reaction zone for IP, which led to the formation of a rougher PA surface with more nanovoids in the ultra-thin selective layer, thus, contributing to a higher effective surface area and less hydraulic resistance for improving water permeability. A 1.2-m wide flat-sheet substrate was casted in-house, and spray-assisted IP was utilized to prepare the membrane. The TFN membrane was rolled into a 2.5-inch element module. A PVA coating protocol was specially designed to introduce a hydrophilic layer atop the membrane surface to effectively improve the membrane stability and fouling resistance, leading to a comparable level of salt rejection with the state-of-the-art commercial membranes in treating real seawater. The fabricated element module demonstrates a high flux of 42.6 L/(m^2·h) with 99% NaCl rejection. In conclusion, this work presents a readily scalable spray-assisted IP technique to fabricate liposome-integrated SWRO membrane module in a larger dimension for seawater desalination.

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Synergistic Water Transport Through Heterogeneous Artificial Water Channels for Desalination

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Artificial water channels have attracted much attention for environmental industrial application including water purification and desalination [1, 2]. Inspired by the natural fine craft Aquaporin, impressive progresses on amphiphilic synthetic channels have been made by researchers, providing the alternatives to achieve water flowing but reject ions for keeping permeability-selectivity balance [3]. But there is less research on the co-transport and synergy behavior among different channels with variable structure and functions. Thus, to deeply explore the inner interaction and potential transport mechanism is worthy. In this study, we are aiming to explore the co-transport behavior of structurally different artificial water channels by evaluating the corresponding water permeation and then disclose the key mechanism of water co-transport. Based previous reported strategies [4], we obtained a class of channel forming compounds grafted with imidazole, hydroxy, carboxylic acid, crown ether, guanidium and so on. The specific chain lengths endow channel with the optimal hydrophobicity, assisting it to intercalate into the lipid-soluble phospholipid bilayer and form channel-type structures. The involvement and cooperation of different head groups will enhance by an order of magnitude the water transport. It is believed that the transport capabilities mainly rely on intramolecular hydrogen bondings between the grafted groups and water molecules within the self-assembled pores. The narrow spatial pore could provide sufficient distance for water molecules to pass through with a rejection of ionic solutes. Based on this research, more functional alternatives are expected to be exploited for achieving selective and enhanced water permeation. Furthermore, by applying in real solid system, a promising generation of membrane can be envisioned for efficient desalination and water purification.

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Beyond aquaporins: Selectivity and permeability mechanisms for water, ions, and protons in new artificial structures

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Fast and selective water transport through cellular membranes has triggered the development of bioinspired membranes for possible separations applications. An emerging approach is to use artificial water channels (AWCs) as a key transport element of the membranes for aqueous separations. AWCs are synthetic mimics or even further engineered versions of naturally occurring aquaporin (AQP) water channels. In this talk, we provide a brief overview of very recent and exciting AWC developments, where AWC designs and performance has moved beyond AQP based designs and benchmark performance. These new paradigms include non-traditional single file transport [1], non-single file transport [2], proton exclusion by transient breaks in the proton wire [2], unique ion exclusion mechanism by preserving waters of hydration on ions, and intriguing use of fluorous surfaces [3,4]. We will also provide some perspectives on how these novel channel designs could be leveraged to create high performance membranes.

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PVDF Membranes Functionalized with 2D-Materials for Enhanced Production of Freshwater and Better-Quality Salt Recovery

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Membrane distillation (MD) and Membrane Crystallization (MCr) are two promising processes for desalting water and recover salts with high purity. New membranes also with different functionalization are developed and proposed in order to enhance the performance of these two membrane operations [1]. Herein, a study is proposed on PVDF-based membranes functionalized with highly-exfoliated graphene and/or other 2D materials for enhancing MD/MCr processes. High-quality exfoliated materials have been obtained with the innovative such wet-jet milling (MJW) technique. In particular, graphene and bismuth telluride have been proposed as materials for their fruitful effects on the host membranes' productivity and efficiency [2,3]. A more available interface between the 2D materials and water has indeed yielded higher fluxes and prevented the salts from passing through the membranes. When graphene or Bi_2Te_3 platelets are blended in PVDF matrix, the testing of these membranes in MD with 35 g/L NaCl solution as feed, show an enhancement trans membrane flux of 99.7% and 200% for graphene and Bi_2Te_3, respectively. High salt rejection has been achieved with all the membranes, especially with graphene where a total salt rejection has been detected. The testing of the aforementioned membranes in MCr proved that very fast nucleation and high growth rate of salt crystals have been also induced, resulting in better-quality minerals for size and shape. For example, in the case of hybrid membrane with Bi_2Te_3 allows to reduce the nucleation time from 285 minutes to 140 minutes compared to the PVDF-pristine membrane. This work provides new insightful indication about the great potential of exfoliated few-layers materials in membrane engineering dedicated to water desalination.

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Scalable and Highly Selective Graphene-Based Ion-Eexchange Membranes with Tunable Permselectivity

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Reverse electrodialysis (RED) arises as an alternative technology that converts the Nernst potential between two water streams with different salt concentrations into electricity. Nevertheless, the feasibility of this process relies on the performance of the ion-exchange membranes (IEM). 2D materials have been studied as an alternative to traditional polymeric IEM due to their enhanced transport properties, higher ionic conductivity, improved mechanical strength, and antifouling characteristics [1]. Graphene oxide (GO) membranes are proposed to be used as cation-exchange membranes as they are naturally negatively charged due to their oxidized functional groups, improved mechanical strength properties, low cost, and facile synthesis. In literature, small area GO membranes produced by non-scalable methods have been reported [2]. One of the novelties of this work is the use of doctor blade technique as a scalable method. Our membranes showed great stability in harsh conditions and also in organic solvents. In this work, GO membranes have been studied and optimized to increase permselectivity and reduce ionic membrane resistance. These membranes showed a higher monovalent cation selectivity than the state-of-the-art polymeric membranes. Electrical impedance spectroscopy technique was used to measure ionic membrane resistance. Results show a direct dependency on thickness with permselectivity and ionic resistance while the lateral size of GO flakes played the opposite role, showing a dominant effect of the solution inside the membrane pores. UV irradiation has been proposed as chemical-free reduction mechanism. Reduced GO membranes showed an increase of permselectivity by 10% due to a decrease of the nanochannels in the 2D structure and reduction of the membrane swelling degree, even though ionic membrane resistance increases. As well, binders were added to the membrane matrix to increase the mechanical stability. Polyvinylpyrrolidone has been found to be the one with better performance in terms of mechanical strength, permselectivity, and ionic resistance.

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Carbon Nanomembranes (CNMs): Molecular and Ionic Transport in Tortuous Sub-nm Channels

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Carbon nanomembranes (CNMs) are two-dimensional membranes (thickness ~1 nm) made by electron-induced cross-linking of aromatic self-assembled monolayers (SAMs). CNMs made from terphenylthiol (TPT) possess tortuous sub-nm pores of a density of ~1018 m^{-2}, which corresponds to one sub-nm channel per square nanometer [1]. TPT CNMs let water molecules rapidly pass through, while they efficiently hinder the translocation of ions. TPT CNMs have thus been utilized as semipermeable membranes in forward osmosis. Their membrane resistance reaches ~104 $\Omega \cdot cm^2$ in 1 M Cl^- solutions, comparable to lipid bilayers of a cell membrane [2]. To further investigate molecular transport through the tortuous sub-nm pores of CNMs, we studied the permeation of gases and vapors of different sizes (D_2O, He, N_2, O_2, CO_2, $CHCl_3$, C_7H_8 und C_6H_{14}), as well as of binary mixtures of water with the above molecules. In all mixtures the water permeation was much higher than the permeation of the other molecules, resulting in a high selectivity of the CNM. To explain this behavior, models of adsorption-controlled permeation (ACP), and water-assisted permeation are introduced. These consider the kinetics of the entire permeation process, starting from a molecule that is adsorbing on the membrane surface, diffusing over the surface until it encounters a pore and that is then translocating through the sub-nm pore [3]. We also found that in water-alcohol mixtures the water permeation rate depends on the alcohol concentration, which leads to effects like molecular jamming [4] and a concentration-driven disruption of single-file water [5].

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Preparation and Properties of Highly Permeable Two-Dimensional Layered Double Hydroxide Membrane

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In recent years, two-dimensional (2D) nanomaterials have promoted the development of ultrathin separation membranes with molecular sieve separation capability due to their atomic thickness [1]. The 2D layered membranes refers to the membranes constructed by stacking 2D nanosheets. The transverse or longitudinal channels of the lamellar will directly determine the path and mass transfer resistance of the diffusion channel [2]. Therefore, shortening the mass transfer path is expected to improve the permeability of 2D membranes. In this work, 2D layered MLDH membranes with high permeability were obtained by introducing nano-level holes on 2D LDH nanosheets. The membrane showed excellent permeability, which was nearly four times higher than that of LDH membrane with no holes in the lamellar. The water flux increased from 233.6 LMH bar to 842.7 LMH bar. At the same time, MLDH composite membrane also showed excellent screening performance in the interception of organic small molecules. MLDH composite membranes also shows excellent stability and pressure resistance in long-term operation.

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Pushing the Limit of Å-Scale Nanopore Engineering for Carbon Capture from Graphene Membrane

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Energy and cost-efficient carbon capture is a highly urgent requirement because of the increasing carbon emission and deteriorating global warming. The current commercial amine-based scrubbing is suffered from the high energy-penalty and the thermal degradation after the regeneration cycles. In this context, nanoporous single-layer graphene (N-SLG), hosting atomic-thickness and a short CO_2-sieving pathway, is an ideal candidate. Incorporating high-density CO_2-selective pores in the graphene lattice still remains a bottleneck. It is attributed to nucleation, and pore expansion happens simultaneously. By prolonging the etching time, nucleation events can be increased; nevertheless, it also leads to unwanted further pore expansion. Unlike this, O_3 gasification is quite effective in limiting pore expansion at a high pore density [1]. In this presentation, I will introduce several new concepts and well-controlled approaches for high-density CO_2-sieving pores in the graphene lattice. I will also discuss a novel multi-pulse millisecond etching route where pore expansion is limited by a high number of nucleation events. This approach yielded CO_2 permeance of 4400 ± 2070 GPU and a CO_2/N_2 selectivity of 33.4 ± 7.9, where the highest CO_2/N_2 separation factor was close to 40. Another route, the nucleation-decoupled nanopore expansion regime, can be achieved by the high number of existing pores, and the fresh nucleation is avoided. Further, I will discuss the polymer-functionalization on graphene nanopores to enhance carbon capture performance. Briefly, a ~10 nm thin polymeric layer was grafted on graphene lattice via the ring-opening chemistry between epoxy groups and amine groups[2]. CO_2 permeance of 8730 GPU and CO_2/N_2 separation factor of 33.4 could be obtained from this strategy. Finally, we demonstrate this approach to fabricate centimeter-scale membranes.

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Advanced graphene oxide-based membranes for gas separations

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We report on the fabrication, modification, and gas separation performance of high-flux graphene oxide (GO) membranes and composite membranes based on GO and single-walled carbon nanotube (SWCNT) supports. Thin selective GO layers[1] were tested as free-standing or deposited on the top of ultrapermeable SWCNT supports [2]. A combination of GO and SWCNT led to similar or higher H2/CO2 selectivity compared to self-standing GO membranes [1]. However, GO-SWCNT membranes exhibited substantially higher fluxes and dramatically better mechanical and aging resistance (time stability). Fabricated GO-SWCNT membranes exhibited remarkable gas separation performance towards H_2/CO_2, H_2/CH_4, H_2/N_2, and N_2/CH_4 gas pairs that far exceed the corresponding 2008 Robeson upper bound. Furthermore, a modification of GO by doping via selected metal ions improved gas permeability and selectivity substantially. Presented composite GO-SWCNT membranes demonstrate a promising route towards large-scale fabrication of high flux hydrogen-selective gas membranes for H2/CO2, H2/CH4, or H2/higher alkanes separations.

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Selective coated membranes for Na/K separation with crown ethers in electrodialysis: Enabling resource recovery from greenhouse wastewater

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Water and nutrient reuse from greenhouse horticulture wastewater in the Netherlands can help recover valuable resources and minimize high nutrient discharge. However, sodium (Na) concentrations prove to be the main bottleneck as it harmful for plant growth. Electrodialysis (ED) is a promising technology to selectively separate ions from greenhouse wastewater. Moreover, ED with monovalent selective membranes (MSED) can aid the recovery of divalent ions over monovalent ions [1]. However, for the horticulture sector, the challenging separation of Na over other monovalent ions such as potassium (K) with high separation potential can further boost the recovery potential from these streams. Recently, various materials and modification methods to achieve Na/K separation have been reported in literature. Specifically, modification with synthetic ionophores such as crown ethers have been gaining interest [2]. Modifications by co-deposition with dopamine and 4′-aminobenzo-15-crown-5 (K- selective) have shown promising results [3]. For simplicity and ease of up scalability, in this study, we modify commercial ion-exchange membranes by co-deposition of dopamine with both 4′-aminobenzo-15-crown-5 (K-selective) and 4′-aminobenzo-18-crown-6 (Na-selective) crown ethers respectively. Following which the members are characterized for Ion exchange capacity, water uptake, FTIR, SEM and resistance. Finally, there Na/K separation performance with greenhouse wastewater as feed is observed with (ED stack) and without electricity (diffusion cell) as a driving force, and the transport mechanism investigated. It is found that the electro-chemical gradient had a significant effect on the transport of properties of Na and K. The Na/K selectivity with an electro-chemical gradient is expected to be higher with the modified membranes.

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Porous PVDF Membrane with Controllable Wetting Property in Oil Water Emulsion Separation

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Abstract: There is an urgent problem [1] in oil water emulsion pollution due to the massive oil leakage and oily wastewater, and membrane technology could play a significant role in the separation of oil water emulsion. The porous polyvinylidene difluoride (PVDF) membrane fabricated via vapor induced phase separation method was coated with dopamine, and it was grafted with poly(N-isopropylacrylamide) (PNIPAm) [2] on the polydopamine layer. Due to the function of surface chemistry and microstructure, [3] the PVDF membrane showed thermal responsive property in contact angle (31 to 37 °), in the meantime the water permeance increased from 1 100 to 2 000 L m^{-2} h^{-1} bar^{-1} as the temperature raised from 25 to 45 °C. The PVDF membrane was applied to separate n-hexane-in-water emulsion with good permeance (600 L m^{-2} h^{-1} bar^{-1}) and high oil rejection (99.8%), and it showed an excellent antifouling property attributed to the same permeance and oil rejection even in three subsequent oil water emulsion separation cycles. Therefore, the PVDF membrane with controllable wetting property could show good potential in the oil water emulsion separation.

Key words: oil water emulsion separation, controllable wetting property, antifouling property

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Tailoring Poly(vinylidene fluoride-trifluoroethylene-chlorofluoroethylene) Membrane Microstructure for Advanced Applications

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Membrane technology is the base of several technologies including filtration, sensing, biomedical and energy applications [1]. The processing technique and the corresponding processing parameters allow to properly tune membrane porosity and pore size, key factors to tune the membrane characteristics for specific applications [1,2]. There are different membrane preparation techniques, such as template-leading, electrospinning, or dip-coating, one of the most used ones being phase inversion [1]. The dry-cast process, non-solvent (NIPS), thermal (TIPS) and vapor (VIPS) induced phase separation and solvent evaporation promote phase inversion [1]. Further, the addition of other polymer in the polymer-solvent system allows to create a spongy structure by enhancing pore formation, increasing pore interconnectivity and hydrophilicity [2].

Poly(vinylidene fluoride), PVDF, and its co-polymers are characterized by high polarity and by a suitable control of the degree of porosity, allowing to tune membranes for application in areas such as biomedicine, environmental remediation and energy storage applications.

In the present work, Poly(vinylidene fluoride-trifluoroethylene-chlorofluoroethylene), P(VDF-TrFE-CFE) membranes were prepared by different techniques such as, TIPS, NIPS, electrospinning, and salt leaching. The microstructural, thermal, and mechanical properties are presented and discussed and well as the application of the membranes in the area of environmental remediation and energy storage.

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A Facile Tuning of Polyelectrolyte Cross-linking Chemistry for Improved Permselectivity and Stability of a Cation Exchange Membrane

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Monovalent selective cation exchange membranes are gaining attention in the desalination of brackish groundwater for irrigation, treatment of seawater and enhanced mineral recovery from seawater RO brine. These selective membranes effectively retain the divalent ions in the diluate where they improve water quality instead of passing to the concentrate that could cause scaling/fouling. This work highlights the preparation of a monoselective membrane with high selectivity, stability, and low resistance compared to state of art membranes. The key challenge of modified membrane surface stability with low resistance was successfully improved by structural tuning of cross-linking chemistry between the selected polyelectrolyte, polyethyleneimine (PEI), and cation exchange membrane (CEM) in a controlled manner. A facile three-step single side modification approach was implemented, where the hydrophilic, bio-inspired polymer PDA layer strongly binds the PEI to the CEM, followed by GA cross-linking via Schiff base reaction. The change in surface morphology, physico-chemical and electrochemical properties of the modified membranes were investigated. Selectivity of the prepared membranes was tested at different current densities (i.e.,30% limiting current density (LCD);50% LCD and 70% LCD) with two different synthetic solutions mimicking the cation composition of brackish groundwater and Reverse Osmosis concentrate. The observed practical permselectivities at different current densities and compositions ranged between P_(〖Ca〗 ^(2+))^(〖Na〗 ^+)=3-4 and P_(〖Mg〗 ^(2+))^(〖Na〗 ^+)=10-16. Moreover, the membrane became less monovalent permselective with increasing current density. Modified membranes are stable after 8 cycles of desalination with almost constant selectivity. The synthesized membrane is promising in terms of high stability and permselectivities (two and four folds P_(〖Ca〗 ^(2+))^(〖Na〗 ^+) and P_(〖Mg〗 ^(2+))^(〖Na〗 ^+)respectively compared to a standard commercial membrane modified with electroadsorption of PEI [1]),whereas CEM modified with co-deposition of PDA/PEI with Mil(53)-Al followed by trimesoyl chloride crosslinking resulted P_(〖Mg〗 ^(2+))^(〖Na〗 ^+)=3.34 [2]. Notably, the resistance of our modified membrane was 40% lower than the leading commercial monovalent selective membrane.

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Surface Modification Strategies for Polyethersulfone Affinity Chromatography Membranes for Selective Adsorption of Monoclonal Antibodies

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IgG, a monoclonal antibody that has gained significant attention, especially in disease treatment and diagnostics [1], has been predominantly purified by affinity chromatography in industrial applications. However, the ligands used (e.g., Protein A) present serious drawbacks, including cost inefficiency, leaching, and activity loss during elution, which decrease the overall performance and efficiency [2]. Membrane affinity chromatography has been proposed as a promising alternative, offering high throughput and faster mass transfer kinetics. However, challenges, including low specific surface area and low binding capacity, limit the progress of these membranes. Therefore, affinity membranes with alternative synthetic ligands that are stable, cost-effective, and with a high specific surface area should be introduced. The current study aims to produce polymeric affinity membranes equipped with synthetic affinity ligands for highly-selective adsorption of IgG. PES/PVP blend membranes were fabricated via phase separation. The fabrication conditions, including dosage and additive types, were investigated to produce substrates with desirable pore and morphological characteristics. Preliminary results from characterizations have shown that highly porous substrates with an MWCO of ~150 kDa and minimal non-specific BSA adsorption were produced. Successful immobilization of a synthetic IgG-specific ligand peptide "HWRGWV" [3] was also achieved on membrane surfaces via carbodiimide chemistry. The concept was successfully proven at NHS/EDC concentrations of 0.1 M with a ligand density of 0.07 mg/mL, and a binding capacity of 50 mgIgG/mLadsorbent was reached for the static binding mode. Subsequently, a detailed parametric study on the surface modification conditions in terms of ligand type, density, and spacers will be conducted to reveal the relationship between IgG adsorption and membrane surface chemistry and the impacts of binding conditions on the membrane performance, including pH and ionic strength. The incorporation of new ligands into the membrane functionality would significantly broaden the pathways for developing next-generation membrane chromatography for highly selective and fast capture of IgG.

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Study on Performance of Enhanced UF PES Membranes with Novel Low-Fouling Coating in Lab-Scale Side-Stream Membrane Bioreactor

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Membrane bioreactors (MBRs) widely employ ultrafiltration polyethersulfone (UF PES) membranes for municipal and industrial wastewater treatment to reject suspended particles and micropollutants. Submicron particles depositing on the membrane surface during MBR operation cause significant membrane fouling as time progresses. So, the active membrane surface of commercial grade Microdyn UP150P polyethersulfone (PES) membrane was enhanced by spray-coating anti-fouling layer of polymerisable bicontinuous microemulsion (PBM) materials, followed by 1 minute of ultraviolet (UV) radiation exposure to complete the polymerisation process. Preliminary tests for fouling propensity of membranes with 0.1 g/l humic acid solution as model foulant exhibited 25% lesser decline of permeability for PBM spray-coated membrane than commercial PES membrane during critical flux determination. The performance of PBM-coated membrane was studied in comparison to the commercially available Microdyn UP150P membranes in Sartorius AG's Biostat® lab-scale side-stream MBR to treat low-strength model domestic wastewater. Based on three key parameters – permeate flux, transmembrane pressure (TMP) and permeability, the fouling behavior of the membranes was investigated by flux-step tests with activated sludge. The PBM-coated membranes enabled better flux control accompanied with higher TMP and better rejection rate than commercial PES membrane. Assessment of the PBM-coated membranes will also be carried out by long term fouling tests in the MBR followed by tests with high strength model wastewater like olive mill wastewater. The PBM-coated membranes are being further enhanced by addition of copper nanoparticles which will be studied for anti-bacterial and anti-viral properties.

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Ultrasound-assisted crossflow ultrafiltration of cellulose nanocrystal suspensions: multiscale approach by SAXS and MicroPIV

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In the field of biorefinery, ultra and microfiltration techniques may be relevant for the valorization of high value-added wood-based products. Regarding cellulose nanocrystals (CNC), the different production methods lead to heterogeneous mixtures in size, containing both CNC and micrometric particles. In this context, membrane separation would solve this problem by isolating the CNC from the larger particles. However, this process is limited by the accumulation of particles on the membrane surface (fouling and concentration polarization phenomena). The strategy chosen in this study is to control clogging and improve mass transfer, by coupling ultrasonic fields with shear and pressure flow fields acting during crossflow ultrafiltration [1]. The objectives of this work are to provide an understanding of the mechanisms involved in the formation of concentration polarization layers with/without ultrasound using a multiscale approach combining, i) small angle X-ray scattering (SAXS) and ii) particle image velocimetry (micro-PIV) measurements during tangential ultrafiltration. SAXS measurements allow the determination of concentration profiles at the nanoscale [2] while micro-PIV gives information on the velocity fields in the vicinity of the membrane surface and during filtration on scales of a few hundred micrometers [3]. A crossflow filtration cell coupled to ultrasound has been developed to apply the ultrasonic waves close to the plane membrane and to obtain an in-situ characterization of the colloidal organization, concentration profiles and velocity fields in the polarization layer. Ultrasound induces a decrease in the thickness of the concentration polarization layer, associated with its reduction of concentration, as well as a modification of the anisotropic organization in the vicinity of the membrane, enhancing the filtration performances. These results reveal an equilibrium between the rheological behavior of the suspension at the membrane surface and various external forces due to pressure, shear flow and local shear stress induced by ultrasonic waves.

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Linking adhesion of EPS with the temporal gene expression of microbial biofilms developed in wastewater on RO membranes: Meta-transcriptomic analysis combined with QCM-D

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Biofouling of reverse osmosis (RO) membranes is a major concern for desalination, leading to deteriorating membrane performance and lifetime. Advanced physical and mechanical characterization of extracellular polymeric substances (EPS) combined with advanced meta-transcriptome methodology, providing a huge data set of gene expression in RO biofilm consortia, have led us to link between (i) the evolution in EPS adhesion and mechanical properties; (ii) the involved biofouling microbial communities; and (iii) the function of the consortia developed on the membrane. Biofouled RO membranes coupons were sampled after 48 and 96 hours of biofilm growth with synthetic tertiary wastewater in the RO flow cell. Extracted RNA was sequenced using NGS techniques and was analyzed using bioinformatic tools. In addition, extracted EPS was studied for its adhesion and viscoelastic properties using quartz crystal microbalance with dissipation monitoring (QCM-D) and biofilm imaging was carried out by scanning electron and confocal laser scanning microscopes (SEM and CLSM, respectively). Taxonomic analysis of the microbiota was based on assembled 16S rRNA transcripts, followed by meta-transcriptomic analysis: functional and taxonomic annotation of transcripts were derived from the mRNA fraction. For taxonomic analysis of the mapped reads (rRNA sequences), the single-end mapped reads were analyzed using the phyloFlash pipeline. For functional and taxonomic analysis of the unmapped reads (mRNA sequences), the reads were assembled into transcripts using Genome-Guided Trinity Transcriptome Assembly. A direct link is provided between (i) biofilm growth microscopic observations; (ii) evolution of EPS adherence; (iii) elevation of the EPS shear and viscosity moduli; and (iv) robust changes in the expression of large data set of polysaccharide synthesis relating genes rather than a robust microbial communities analysis. Hence, we highlight the importance of the microbes’ function rather than their taxonomy for biofilm structure and integrity on the RO membrane surface.

Predicting performance decline in a reverse osmosis plant with an on-line biofilm sensor

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Biofouling control represents a major challenge for the correct functioning of reverse osmosis applied to solutions with high content of dissolved organic carbon. Most available monitoring systems are difficult to use at full scale [1] (e.g., impedence spectroscopy, nuclear magnetic resonance and optical coherence tomography). An interesting alternative to these techniques are biofilm sensors, which are able to gauge the amount of biofilm accumulating on a surface [2]. In this work, a biofilm sensor was embedded in a flow cell and integrated in a lab scale reverse osmosis system. The flow cell was set parallel to two flat sheet membrane modules operated in cross flow. Biofilm growth was monitored on the sensor surface as well as on the membrane using optical coherence tomography. At first, the membrane fouled, although no biofilm accumulated on the sensor surface. The sensor surface was then sandblasted to promote attachment and the experiment was repeated. Thereby it was possible to observe biological growth both on the sensor and on the membrane. The sensor displayed an increasing signal for biofilm accumulation as the permeability decreased, showing the possibility to be used as an early warning tool. Additionally, the system was cleaned to restore membrane performance and the response of the sensor to cleaning was assessed. In summary, the sensor offers a new and reliable possibility to on-line monitoring of biofouling on reverse osmosis membranes.

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Impact of interaction of foulants and chemical cleaning agents on membrane ageing

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Ultrafiltration membranes are a popular technology for drinking water treatment. Membranes operate with repeating cycles of filtration and cleaning, for a number of years, typically ~7-10, until they must be replaced because of irreversible changes to physical, chemical and/or operational characteristics of the membranes. These long-term changes, are often referred to as membrane ageing. Previous accelerated ageing single soak studies have linked the extent of ageing to the cumulative exposure (i.e. dose) to chemical cleaning agents. Although easy to undertake, single soak accelearated ageing does not consider conditons typical of full-scal operation. The present study addresses this knowledge gap and enables potential interations between foulants, chemical cleaning agents and the membrane material to be comprehensively investigated. Two approaches were considered for accelerated ageing: single soak with hypochlorite and cycling (repeating filtration, hydraulic cleaning and chemical cleaning with hypochlorite; typical of full-scale operation). Changes to the membrane material during ageing was quantified using differential ATR-FTIR. Various scavengers and chemical probes were considered to investigate potential oxidation mechanisms responsible for the observed changes. The impacts of single soak and cyclic ageing on the membrane were observed to differ, but only in terms of rate. For single soak, the observed changes were relatively slow and could be attributed to the direct oxidation of the membrane material by hypochlorite. However, for cyclic ageing, changes to the membrane material were much more rapid. The result indicate that in the presence of retained foulants and hypochlorite, hydroxyl radicals are formed that accelerate changes to the membrane material. Ongoing research is investigating the impact of foulant components on the generation of radical species and changes to membrane material. This new knowledge could explain why membranes in some systems age more rapidly, and more importantly, be used to design protocols that slow down the ageing process.

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The curse of fouling in ion exchange membranes: Identification of real organic foulants and comparison between homogeneous vs heterogeneous membranes

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Fouling in electrodialysis (ED) requires frequent cleaning of ion exchange membranes (IEM) and their ultimate replacement toward their end of lifetime [1]. Anion exchange membranes (AEM) foul more due to adsorption of negatively charged organic particles on its surface [2]. So far, fewer studies have investigated the real organic fractions that foul the surface of IEMs [3]. This work investigates different organic fractions that foul AEM surfaces (homogeneous and heterogeneous type). In this study, 2 different types of AEMs (homogeneous and heterogeneous) were fouled with 3 different industrially relevant feed streams. Later, they were cleaned with 0.1M HCl and NaOH respectively to mimic industrial cleaning in place (CIP) operations, followed by their immersion in NaCl (35 g/L) and EtOH (12%) - H_2O solution to extract the organic foulant fractions. An LC-OCD analysis of the above revealed highest extraction of foulant fractions from corn steep liquor stream, used for fouling both AEM types. Building blocks and low molecular weight neutrals having concentration of 175 ppb-C and 100 ppb-C seemed to desorb the most from homogeneous type AEMs. In case of heterogeneous type, 700 ppb-C of building blocks followed by 500 ppb-C of humic substances desorbed the most/mg dry wt of IEM, indicating that a difference in structure of foulants and AEM itself led to its higher desorption. This was also proven by determining the single membrane area resistances of both membrane types, where heterogeneous AEMs showed a value of 159 ohm·cm^2 in comparison to homogeneous ones with a value of 115 ohm·cm^2. A difference in their thicknesses and grafting structure can be adhered to the higher increase in membrane resistance, as well as, to the higher amount of desorption due to exchanging of NaCl ions with foulant groups.

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The Temperature Effect on Fouling in Anaerobic Membrane Bioreactor: Roles of Extracellular Polymeric Substances and Soluble Microbial Products

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Biofouling is the main challenge of anerobic membrane bioreactors (AnMBR) operation, affected by various environmental conditions including temperature. Here, we explored the effect of two operational temperatures, 35 °C and 25 °C, on the AnMBR performance, delineating the interactions of soluble microbial products (SMP) and extracellular polymeric substances (EPS) with the membrane surface and their associated viscoelastic properties. We hypothesized that membrane performance at different temperatures (with similar biological performance) will be differentially affected due to different SMP and EPS properties, causing membrane biofouling. AnMBR operation showed a drastic membrane blockage after 11±2 and 18±2 days at 25 and 35 °C, respectively. Although the AnMBR biologically performed similarly at these temperatures (DOC removal, ORP biogas composition), by changing the hydraulic retention time and maintaining similar permeate flux (changing membrane surface area), more significant biofouling indicatives were detected at 25 °C versus 35 °C: (i) confocal laser scanning microscopy (CLSM) analysis showed significantly higher biofilm amount; (ii) quartz crystal microbalance with dissipation monitoring (QCM-D) showed more adhesive, viscous and elastic EPS layer, and (iii) Ex-Situ filtration experiments with SMP and EPS extracted from both the mixed liquor suspended solids (MLSS) and the membrane surface showed faster membrane fouling with EPS rather than with SMP. Our main conclusion, corroborate with our previous aerobic MBR studies: The cohesion of EPS originated from MLSS and its viscoelastic characteristics are critical for AnMBR fouling behavior, likely due to effects on flocs integrity at the AnMBR shear induced environment. As such, EPS layer produced at 25 °C, was more fluidic and accessible to the UF membrane pores in the AnMBR than EPS produced at 35 °C. Future analysis of the microbial communities structure composing the AnMBR flocs and the membrane biofilm will clarify the above differences of EPS properties and the consequent membrane fouling at different temperatures.

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Scaling in membrane distillation—effect of membrane properties and configurations

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Membrane distillation (MD) is a promising technology to produce freshwater from high-concentrated solutions by using waste-grade heat. Despite its huge potential, the process suffers from scaling that reduces flux, permeate quality and lifetime of the membrane. Scaling of CaSO4 has proven especially detrimental to flux and membrane integrity. The current study investigates the effect of membrane properties (pore size, overall porosity, and configuration i.e., flat sheet and hollow fiber) and operating temperature on $CaSO_4$ scaling in direct contact MD. The study was carried out by using two commercial PTFE flat sheet membranes (PTFE1 and PTFE2) and a hollow fiber PP membrane. The average pore size of PTFE1, PTFE2 and PP membrane was 0.45, 0.2 and 0.2 µm respectively whereas the porosity was 83, 58 and 73%, respectively. The membranes were enclosed in a transparent modules and scale formation at the surface was directly monitored in real-time by using a microscope mounted at the module and by following the flux and conductivity of the solution. Morphology of the scaling crystals was studied by performing scanning electron microscopy on the used membranes. The tests performed in controlled mode demonstrated that the membrane surface for each of the membranes acted as a site for heterogeneous nucleation and reduced the induction time. For all the membranes, the intensity of scale formation increased with feed temperature and the morphology of scale shifted from sheet to needle-like with increase in temperature. A comparison of PTFE1 and PTFE2 revealed that the large pore size caused the formation of isolated spherulite shaped crystals (scale) whereas small pore sizes produced compact rectangular crystals which were more detrimental to the flux. The scaling pattern in hollow fiber membrane was significantly different from the flat sheet counterparts where non-uniform scaling was observed around the fiber, suggesting low-scaling potential of this configuration.

Treating challenging industrial wastewater by membrane distillation: Understanding the impact of the fouling

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This study investigates the efficiency of membrane distillation (MD) for the treatment of challenging industrial wastewaters, by focusing on process performance and membrane fouling. The treatment of textile dye solution is considered one of the most challenging wastewater treatments, representing a major concern for the ecosystem. The effect of the feed temperature and feed flow rate on the MD fouling were investigated at different feed water temperatures and feed flow rates. Results show that (i) negligible fouling was observed at a feed temperature of 40 °C, (ii) the increase in temperature impacted the process performance, leading to severe flux decrease (~70%) and to the formation of a thick fouling layer (240 µm) at a feed temperature of 80 °C, (iii) fouling thickness was reduced with increasing feed flow velocity. In-situ non-invasive fouling monitoring by optical coherence tomography (OCT) allowed evaluating the fouling spatial distribution on the membrane and linking the MD process performance to the fouling accumulation, indicating a linear correlation between the flux decrease and the fouling deposition. In this study, we present also the data on the treatment of brine with MD by focusing on process performance, chemicals additives and membrane scaling. The scale formation was monitored with the OCT, and results were validated by scanning electron microscopy (SEM). Results show found that depending on its concentration, the antiscalant prolonged the induction time of salt crystallization whereas antifoaming showed the opposite effect. Scaling mostly occurred due to calcium sulfate crystals formation with the large size needle-shaped crystals favored at higher feed temperature. Results show that thermal desalination brine, which is already preheated and chemically pretreated, could be an appropriate feed source for MD to further increase the overall water recovery and reduce the marine environmental impact by reducing the brine discharge volume and its temperature.

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Freshwater and minerals recovery from acid mine drainage through membrane crystallization

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Rapid increase in industrialization is generating new streams of wastewater and is consuming the resources at unprecedented rate. Thus, implementation of efficient wastewater treatment and resource recycling technologies is highly desired. Unavailability of efficient technologies, capable to treat complex solutions at low energy requirements, has been the limitation to recover water and valuable minerals from different wastewater streams. Membrane crystallization (MCr) is a technique that has the potential to treat solutions to their saturation level and thus offers the opportunity to simultaneously recover fresh water and minerals [1]. In this study, MCr was tested on synthetic acid mine drainage from South Africa. The water fluxes were 0.80 L/m^{2}h, 1.84 L/m^{2}h, and 2.32 L/m^{2}h at feed inlet temperatures of 34.6 °C, 41.5 °C, and 52.4 °C respectively. The permeate was of high quality and its conductivity did not exceed 7 µS/cm despite the high-concentrated and complex feed solution. Moreover, MCr was able to turn more than 90% of the wastewater into freshwater in this study. In parallel to the freshwater production, crystals of various salts were also recovered. X-ray diffraction analysis revealed that the majority of the recovered crystals were $CaSO_4 \cdot 2H_2O$, but also other salts like $KFeO_2$ and NiO(OH) were observed, which highlights the potential of valuable salts recovery from acid mine drainage. The crystal size was relatively small corresponding to average diameter of ~14 µm.

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Potential and Limitations of Ceramic Membranes in MD Processes

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Conventional fresh water sources (groundwater, lakes and rivers) are no longer sufficient to cover the global water demand. This has led to increased efforts to produce fresh water by desalination processes. Despite the technical maturity of conventional desalination technologies technical limitations remain. For instance, reverse osmosis (RO) processes are limited by the osmotic pressure and require sufficient feed pre-treatment due to scaling and fouling. These limitations and stricter environmental guidelines drive optimization and commercialization of emerging desalination technologies such as Membrane Distillation (MD). MD processes operate with temperatures below the boiling point which enables the use of low-grade energy sources such as solar heat, waste heat and geothermal heat. MD processes are particularly suited for the energy-attractive (off-grid) treatment of highly concentrated solutions, potentially enabling Zero-Liquid-Discharge applications. Polymeric membranes (PTFE, PP, PVDF) are the most commonly used membranes in MD processes. This is due to their intrinsic hydrophobic surfaces, low costs and good mass transfer characteristics. Unfortunately, they are not robust enough when applied to aggressive feed solutions (e.g. highly concentrated abrasive brines, extreme pH, solvent laden solutions) which can lead to their permanent degeneration [1,2]. Modified ceramic membranes could enable the treatment of highly saline solutions and facilitate minimal (MLD) or zero liquid discharge (ZLD) application. In order to understand the suitability of ceramic membranes for membrane distillation processes, modified ceramic membranes were characterized in respect to their hydrophobic characteristics and subsequently tested in vacuum membrane distillation. For instance, it was found that the thin final layer of asymmetrically structured ceramic membranes is robust enough for abrasive suspensions and that TiO_2 membranes outperform Al2O3 membranes in respect to the mass transport. A maximum permeate flux of 35 kg/(m² h) at excellent rejections was determined in vacuum membrane distillation treating a highly saline solution.

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On-field testing of a photothermal membrane distillation plant for seawater desalination

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Membrane distillation (MD) has a great potential to become one of the best technologies for the development of off-grid desalination/wastewater treatment plant thanks to its modular configuration, low power consumption and ability to exploit low grade energy resources [1]. To this end, many attempts to design solar MD plants have been carried out. A Particular configuration developed to exploit solar energy is the so-called photoactive membrane distillation (PMD) that employs a membrane that is capable of converting the light into sensible heat directly on its surface. The feed temperature is raised in the boundary layer in contact with the membrane surface reducing – or even reversing – the temperature polarization effect [2]. In this work, a hydrophobic photoactive membrane has been prepared including carbon black (CB) inside the starting polymeric dope solution followed by a simple non-solvent induced phase separation technique. A small sweeping gas membrane distillation pilot setup was developed in order to evaluate the long-term stability of the membrane operating under real conditions. To this end, the membrane was mounted in the cell and the plant was placed under direct sunlight at the Department of Chemistry and Industrial Chemistry of the University of Genoa, Italy. The plant feed was seawater and the main process parameters were continuously monitored as well as the actual incident solar irradiance. Exposing the membrane to solar light increased the feed temperature up to 45 °C and induced a distillate flux enhancement of around 100% compared to the tests performed in similar conditions, but covering the membrane from solar light.

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Investigation of membrane surface charge effects on fouling phenomenon on anion dyes wastewater treatment using Air Gap Membrane Distillation

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Dyes in the environment in large quantities can be very dangerous because some dyes are carcinogenic, mutagenic, and toxic. To prevent environmental damage, dyes discharged into the environment must be treated first. One technology that successfully removes the dyes from wastewater is Membrane Distillation (MD). The notable advantage of MD is that it applies low-grade heat and low operating pressure. However, one of the main problems for MD is membrane fouling. Therefore, this study aims to investigate the influence of membrane surface charge on the fouling phenomenon on anion dyes wastewater treatment using Air Gap Membrane Distillation (AGMD). This research was carried out by adding the negative charge onto the Polyvinylidene fluoride (PVDF) membrane surface. The membrane surface was prepared through plasma treatment using O_2 and functionalized using acrylic acid (AA) and Sodium hydroxide (NaOH). The different values of negatively charged membrane surfaces were used in the AGMD process to treat anionic dyes, CI reactive red 241, and CI Acid Yellow 79. The performance evaluation was confirmed with permeate flux, color rejection factor, and contact angle analysis. The result showed that AGMD's color rejection efficiency is more than 99%. Furthermore, the experiment demonstrated how membrane surface-charged interaction with anionic dyes could influence the fouling phenomenon during the MD process. The charge repulsion effect allows the negatively charged membrane surface with good anti-fouling performance.

Electrochemically Grown ZIF-8 Membranes on Ag-sputtered Nylon for Propane/Propylene Separation

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The separation of olefins and paraffins, produced by processes such as steam cracking, is highly important to the petrochemical industry [1]. In particular, light olefins such as ethylene and propylene are important raw materials for the production of polymers. Membrane technology is an energy-efficient way of achieving such separations and is gaining increased attention as alternative method to the traditional cryogenic-based process. A number of novel membrane materials have been studied for their olefin/paraffin separation performance, among which, zeolitic imidazolate frameworks (ZIFs), a sub-class of metal organic frameworks, show notable potential [2]. These materials exhibit tunable porosity and physicochemical characteristics as well as superior chemical and thermal stability. To achieve competitive separation performance, the materials are required to have high permeance and selectivity, yet, there is typically a trade-off between the above two key performance indicators [3]. To this end, novel ultra-thin membranes and fabrication procedures such as vapor phase synthesis and electrochemical growth are being explored. In this work, a novel ultrathin ZIF-8 membrane was fabricated on an Ag-sputtered Nylon substrate via electrochemical growth. The morphology and the surface chemical characteristics of the obtained membranes were examined by SEM and XRD confirming the presence of ultrathin layer of ZIF-8 and Ag on the membrane. Olefin/paraffin single gas permeation tests, along with sorption experiments were carried out at 25, 50 and 75 °C and it was found that the new membranes exhibit propane/propylene ideal selectivity of ~50 at 25 °C with a propane permeance of ~3 x 10^{-7}mol/m^2/s/Pa.

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Continuous online mass-spectrometric analysis of mixed gas permeation: a tool to study transient phenomena and real-time changes in polymeric and mixed matrix membranes

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The successful introduction of novel membrane materials requires the accurate knowledge of their transport properties. The most common tool for the analysis of gas transport parameters in polymeric membranes is the pressure increase instrument with fixed permeate volume, with application of the so-called time-lag method. This allows the determination of the gas permeability, diffusion coefficient and –indirectly– the solubility, but a major limitation is that it works for pure gases only. The present paper describes a new method which overcomes this limit and can measure transient phenomena of the individual gases during mixed gas permeation, using a traditional cross-flow cell and a variable volume instrument. The unique feature is that the traditionally used gas chromatograph is replaced by a quadrupole residual gas analyser for the continuous mass-spectrometric analysis of the permeate composition [1,2]. Its rapid response allows the implementation of different measurement approaches, including the exposure of the membrane to a step or pulse in the feed pressure or feed composition [3]. We will present the broad spectrum of membrane properties that can be studied with this instrument. Most importantly, online analysis of the permeate composition enables the determination of the diffusion coefficients of the individual components in the gas mixture. Such data are extremely rare in the literature and provide valuable information on the membrane performance under real operation conditions. In addition, the method allows the analysis of time-dependent phenomena, such as physical aging and sorption-induced dilation of the sample, or coupling phenomena due to competitive sorption of gases, humidity or trace impurities, as well as apparent anomalies like ‘immobilizing’ sorption in mixed matrix membranes, or non-Fickian transport. The advantages, disadvantages and some specific difficulties and their possible solutions of the mass-spectrometric analysis of the gas mixtures will be discussed to show the full potential of this method.

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Coupling FTIR spectroscopy and Barometry to investigate sorption equilibrium and kinetics of pure and mixed gases in rubbery and glassy polymers

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The volumetric method based on Gas Chromatography and Barometry is the standard technique to investigate sorption of multicomponent gas mixtures in polymers. A mole balance over the gas phase is made for each absorbed species to retrieve indirectly its concentration in the polymer phase [1]. Starting from the work of Hong et al. who used FTIR Spectroscopy in the ATR mode to investigate the diffusion of Methyl Ethyl Ketone and Toluene in Polyisobutylene, we implemented a new method coupling FTIR Spectroscopy in the Transmission mode and Barometry to investigate the diffusion of pure and mixed gases in rubbery and glassy polymers [2]. The IR absorbance signals of the species being absorbed by the polymeric membrane are calibrated both in the gas and the polymer phase with Barometry. Then, two approaches may be followed: either the classic volumetric method is applied to the gas phase, or the polymer phase is directly investigated to retrieve the concentration of each absorbed species [3]. We first validated each approach by studying sorption of carbon dioxide and methane in Polydimethylsiloxane (PDMS) at room temperature up to 9 bar. In this case, preliminary results related to the sorption of CO_2 in PDMS from binary gas mixtures with ~0.5 and ~0.25 mol mol^{-1} CO_2 mole fraction were obtained and compared with the literature [1]. Then, the case of CO_2/CH_4, CO_2/C_2H_6 and CO_2/C_3H_8 in Poly(2,6-dimethyl-1,4-phenylene) oxide films was investigated at ambient temperature and 35 °C to extend the method to the case of glassy and semicrystalline polymers and to other gases. Sorption of CO_2/CH_4 was tested up to 1.3 bar whereas CO_2/C_2H_6 and CO_2/C_3H_8 up to 0.27 bar. The technique allows to quantify sorption induced swelling and specific interactions with the absorbate when the polymer spectrum is measured. The partial pressure of each component is measured in situ.

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H_2 selective membranes based on self-assembled polymer-graphene oxide with controlled morphology

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2D materials, and graphene oxide in particular, offer unique opportunities for the fabrication of gas separation membranes due to their peculiar gas transport properties, combined to extremely high aspect ratios [1]. This work is focused on the use of layer-by-layer (LbL) self-assembly to prepare thin coatings on polymeric substrates, depositing alternated graphene oxide (GO) and polyelectrolyte nanosized layers (10 bilayers), leading to well-ordered membrane architectures, thanks to the electrostatic interactions that drive the coating growth [2]. Such structure is exploited for size-selective gas separation membranes, e.g. for hydrogen purification, in which the transport of larger molecules such as CO_2 is significantly hindered by the large diffusive paths around the piled 2D materials. The obtained membranes are characterized by SEM and XRD, revealing an effective and homogeneous deposition of the coating in the alternated fashion, also determining the average thickness of each layer. The gas separation performances are evaluated by pure gas permeation measurements, and the results show the clear sieving ability of the coating, as the gas permeability decreases dramatically as the penetrant size increases, with very pronounced H_2/CO_2 selectivities. The gas permselectivity performances of the thin membranes may then be conveniently modified by thermal partial reduction of the GO sheets (T up to 200°C). Such treatment induces the removal of some oxidized species from GO nanosheets and it alters the layers arrangement, ultimately leading to different gas transport behaviors. Interestingly, the permeability of small molecules such as H_2 shows a 30-fold increase, accompanied by one order of magnitude increase in the H_2/CO_2 selectivity, which reaches values as high as 200, overcoming the Robeson upper bound [3]. That represents a simple and scalable method for the fabrication of H_2 selective nanosized membranes with outstanding sieving performances, using a 2D material and a self-assembly technique.

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ON THE OPERATION STABILITY AND INCREASE OF ACID ACETIC CONVERSION IN PACKED-BED STEAM REFORMERS BY ELECTROLESS PORE-PLATING MEMBRANES CONTAINING TiO_2 INTERMEDIATE LAYERS

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Hydrogen coming from renewable energies or waste valorization is proposed as ideal solution to reach an effective decarbonization of the current energy system. Among multiple alternatives, residual liquid streams coming from microalgae liquefaction could be used as interesting feedstock to transform their hydrocarbons content, mainly acetic acid, into green hydrogen. In this context, the use of membrane reactors, in which high-purity hydrogen is simultaneously separated while progressing the chemical reactions, provides important advantages such as higher conversion, lower required reactor volume, lower energy consumption, and, in general, promotion of process intensification. Typically, Pd-based membranes provide an excellent performance in terms of H_2-purity and permeate fluxes, especially those prepared as composite structure in which a thin Pd-film is incorporated onto a porous support to guarantee an adequate mechanical resistance. Particularly, the incorporation of Pd-films with around 10 µm thickness by electroless pore-plating (ELP-PP) on porous metal supports modified with diverse ceramic particles to adequate their original roughness and porosity has demonstrated an excellent performance in terms of permeation and mechanical resistance for multiple configurations. In this work, ELP-PP membranes containing a TiO_2 intermediate layer between both metal support and Pd-film have been used in a tubular membrane reactor to transform acetic acid into hydrogen with cylindrical pellets of SBA-15 doped with nickel as catalyst active phase. All experiments were carried out at a constant temperature (400ºC), analyzing the influence of pressure (2-3 bar), steam/acetic acid ratio (6-8), GHSV (2500-4500) and system regeneration conditions. The quality of the membrane allows to reach enough permeation driving forces even while operating with relatively low pressures and, at the most favorable conditions, a rise of around 25% on the reference acid acetic conversion obtained in a conventional packed-bed reactor. Regeneration in air also maintains a good performance of both catalyst and membrane for several cycles.

Proton ceramic electrochemical reactors for sustainable hydrogen production

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Electrochemical gas separation emerges as an attractive technology as the cost of electricity declines. Proton ceramic electrochemical reactors (PCERs) extract hydrogen from reaction mixtures and can offer a compact and efficient approach to hydrogen production [1]. Because of the solid-state and gas-impermeable nature of the membrane, the hydrogen produced can in principle be entirely free of impurities, as unreacted molecules cannot get to the other side of the membrane. Through a one-step process, balancing the energy from the endothermic reactions with the separation and compression of hydrogen allows for much higher efficiencies compared with other hydrogen production technologies. In the case of CH_4 reforming, the possibility of extracting H_2 at conditions suitable for the reforming reaction enables the overcome of the thermodynamic limitations in terms of CH_4 conversion. Furthermore, the electrochemical H_2 compression allows to go beyond the driving force limitation of traditional membrane reactors (based for example on Pd membranes). PCERs offer attractive performance including mechanical stability and chemical robustness over a wide range of temperatures (300–800 °C) and pressure conditions (> 50 bar). In our recent work [2], we demonstrate the upscaling of an efficient 36 cell x 15 cm^2 reactor with coupled heat, reaction and current distribution. Hydrogen extraction > 99 % shows complete conversion of CH_4 for conditions relevant both for natural gas and biogas, and a hydrogen purity up to 99.995 %. The by-product leaving the reactor is a concentrated CO_2 stream, which can facilitate the implementation of efficient carbon capture. The results suggest that proton ceramic electrochemical reactors can offer the lowest CO_2 intensity for on-site hydrogen production. Furthermore, to expand on the wide applicability and flexibility of the proposed technology, cell performance for ammonia decomposition will be presented, showing similar conversion with both anhydrous and aqueous NH_3.

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L-cysteine Modified Cellulose Nanocrystals Improve the Heavy Metal Removal/Desalination of Nanofiltration Membranes for Water Treatment

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The discharge of industrial waste effluents into the environment without adequate pre-treatment is a significant cause of water pollution. Heavy metal ions are one of the principal contaminants in industrial wastewater; they are highly toxic and difficult to metabolize in biological systems. Hence, it is essential to remove heavy metals from wastewater to the safe concentration levels published by the World Health Organization (WHO) [1]. We synthesized L-cysteine functionalized cellulose nanocrystals (CysCNCs) and incorporated them into the polyamide layer of thin-film nanocomposite (TFN) membranes for a nanofiltration process. Because L-cysteine contains strong chelating groups, CysCNCs are a good candidate to improve the heavy metal removal efficiency of CNC-TFN membranes [2]. The CysCNCs were synthesized via a straightforward, iodine-catalyzed method for the first time. The successful modification of CNCs with L-cysteine was confirmed using ATR-FTIR spectroscopy, 13C-NMR spectroscopy and EDS-SEM elemental analysis. The salt rejections of the TFC control and the TFN membranes followed the order of Na_2SO_4 > $MgCl_2$ > NaCl. In terms of water permeability, TFN membranes with a 0.1 wt% loading of CysCNCs resulted in a 60% increase in water permeability (16 L/m^2 h bar) compared to the reference TFC (6 L/m^2 h bar) and CNC-TFN (11.5 L/m^2 h bar) membranes. At the same loading, the CysCNC-TFN membranes provided an excellent removal rate for copper and lead ions, 98.1 ±0.1 and 95.2±0.2%, respectively, confirmed with ICP-MS. The improved metal removal rate of CysCNC-TFNs is attributed to the adsorption of ions on the membrane surface due to the thiol chelating groups available on the CysCNCs surfaces.

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Advanced Polyester Loose Nanofiltration Membranes for Resources Recovery

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With the continuous pressure of water contamination caused by the textile industry, loose nanofiltration (LNF) membranes prepared with green materials with an extraordinary water permeability are highly desirable for the recovery and purification of dyes and salts. To obtain LNF membranes with excellent filtration performance, we illustrated the mechanism of large monomers with low cross-linking reactivity in the IP process and the effects of resultant loose structure on the selectivity for dye and salts. In addition, low-pressure LNF membranes with ultrahigh permeability were fabricated via one-step interfacial polymerization (IP), in which inexpensive natural carbohydrate-derived sugars with large size and low reactivity were utilized as aqueous monomers to design a selective layer. A systematic characterization by chemical analysis and optical microscopy demonstrated that the formed polyester film features not only loosen the structure, but also results in a hydrophilic and negatively charged surface. The optimized sucrose-based membranes with an excellent water permeability of higher than 50 LMH bar^{-1} were found to have a high rejection of dyes and a high transmission of salts. In addition, the sugar-based membrane manifested an excellent anti-fouling performance and long-term stability. Furthermore, the non-optimized polyester LNF membranes also showed a high water permeability, while maintaining a competitive dye/salt separation performance, which confirmed the universal applicability of the membrane design principle. Therefore, the proposed new strategy for preparing next-generation LNF membranes can contribute to textile wastewater treatment.

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Novel ceramic filtration disks, first characterization and application in the field of nanofiltration

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Inorganic membranes based on oxide and non-oxide materials have undergone constant development in recent years. Especially in the field of oxide materials, the availability, also with regard to the selection of different tubular geometries, and the pore size spectrum have expanded enormously. Even in the field of nanofiltration membranes (TiO_2/ZrO_2), ceramic-based tubular elements have been successfully developed with and for industrial partners. These ceramic based membranes, which are in general stable in the pH range from 0-14 and show a high temperature stability, have proven their performance in many industrial (water treatment) applications. Typical applications focus on the separation of organic molecules, the retention of multivalent ions, the decolorization of waters, the retention of micropollutants or a combination thereof. Whereas the development started on small single channel tubes the ceramic nanofiltration membranes are now available on an industrial scale with specific membrane areas of up to 1.2 m^2 per membrane as 151 channel tubes from project partner Rauschert. Newest developments focus on rotating disc filters. This new type of rotating disk brings enormous mechanical stability. In current projects, attempts are being made to synthesize them in different diameters but also with different pore sizes (MF, UF, NF). Nanofiltration membrane layers on rotating disc filters are a worldwide novelty. Membrane systems with rotating disc filters certainly form an application niche. However, they have some advantages. They are considered to be operated more efficiently in terms of energy, since the energy required to rotate the disc filters is lower than the pump energy required to operate the crossflow filtration systems. In some applications, they also allow the use of membrane processes for the first time. The presentation will give an introduction into relevant membrane properties and first realized applications using ceramic NF membranes (tubular and disk).

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Steroid hormone micropollutants transport and energy barrier through nanofiltration membranes

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Steroid hormone micropollutants are ubiquitously present at trace level in surface water and human exposure to contaminated water may affect both male and female reproductive systems, fetal development and cancer [1, 2]. The European Directive limit for estradiol concentration of 1 ng/L in drinking water and 0.4 ng/L in surface water (EU, 2020/2184) poses a challenge for nanofiltration (NF) as these contaminants are not fully removed. Indeed, adsorption and subsequent permeation occurs resulting in a breakthrough curve and decrease of removal over time [3]. In order to understand how the transport through the NF membranes can be limited, the energy barriers of four steroid hormones for adsorption (Ea), permeation (Ep) and surface diffusion (EDS) are determined. Solute flux, adsorption and surface diffusion of steroid hormones in tight and loose NF membranes are investigated and the Arrhenius concept is used to calculate the activation energy [4]. Results show that the energy barrier for permeation for the tight NF membrane is underestimated compared to lose NF as the steric effect at the pore entrance is dominant. For the loose NF membrane, partitioning at the pore entrance occurs and the energy barrier for surface diffusion of the adsorbed hormone molecules along the porous structure surface is higher compared to the adsorption energy barrier, indicating that surface diffusion is the limiting step to the overall permeation barrier [5] These findings represent a step forward in the understanding of breakthrough curve as they indicate that the low adsorption energy promotes the breakthrough and by increasing the hormone binding to the membrane may contribute to hinder more the permeation through it. This requires the design of NF membranes with stronger binding properties for steroid hormone micropollutants.

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Comparative Study of Selectivity of Ions in Polyamide Membranes

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Polyamide membranes dominate today's desalination technology, unique properties of semi-permeable polyamide (PA) layer being the key enabling factor. However, there is still a knowledge gap in understanding the ion rejection mechanisms in PA membranes [1]. Analysis of activation energies of ion permeation has been shown recently to shed much on its mechanism. Towards obtaining a more complete picture, here we report new results on temperature dependence and activation energies of ion conduction in polyamide layer measured using electrochemical impedance spectroscopy (EIS). The results are obtained for a wide selection of salts of mono- and divalent in 288-318 K temperature range. In addition, a concentration dependence of activation energy was examined for KCl and HCl. Unlike the activation energies of permeation controlled by the slowest ion, conduction activation energies are controlled by the fastest permeating ions and therefore provide a complementary insight into ion permeation and partitioning. Results reveal a more complex picture of ion uptake and membrane charging than is commonly presumed, which is nevertheless aligned with some other recent findings [2]. In particular, it points to (i) close values of activation energies for alkaline metals ions; (ii) preferential uptake of proton or hydroxide ions as an important charging mechanism, especially, at lower pH and salt concentrations, as well as for salts of divalent ions.

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Removal of selenium and arsenic by nanofiltration with the emphasis on the role of organic matter and retention mechanisms

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Selenium (Se) and arsenic (As) are toxic inorganic contaminants with serious health effects. Their contamination in groundwater can be found at different salinities and with the presence of organic matter (OM) that enhances their immobilisation [1,2]. Such changeable environments require a roust process. This implies that nanofiltration (NF) may be well suited technology for the treatment of different water types contaminated with Se and As. Experiments with NF270 and NF90 membranes were carried with a cross-flow filtration set-up at constant flow rates mode. The retention mechanisms at different species (SeIV, SeVI, AsIII, AsV) were investigated with varied pH, with the effect of membrane charge screening and in the presence of organic matter (OM), such as humic acid (HA). Donnan steric pore partitioning model with dielectric exclusion (DSPM-DE; [3]) was applied to evaluate the retention mechanisms [4]. Asymmetric flow field-flow fractionation coupled on-line with inductively coupled plasma – mass spectrometry (FFF-ICP-MS) was used to quantify OM complexation [5]. Selenium and arsenic retention with NF showed a strong pH dependence, which was due to speciation. The membrane charge screening, at higher salinity (20 g/L NaCl), had no influence on Se and As retention with NF. The presence of OM enhanced the retention of Se and AsV by 10-20% at 10 mgC/L, while 40% of increase was observed for AsIII when increasing HA concentration to 100 mgC/L. The presence of HA enhanced the membrane negative charge and hence the Donnan exclusion mechanism with the negative Se and AsV species. For the neutral AsIII species, FFF-ICP-MS measurements showed that AsIII-HA complexation was the responsible mechanism [4]. While NF can only treat As at oxic conditions, this technology was effective in removing Se from real water (Gahard groundwater, Ille et Vilaine, France) achieving both EU (20 µg/L) and WHO (30 µg/L) drinking water guidelines.

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Recovery of phenolic compounds from by-products of the winemaking chain by ultra- and nano-filtration membranes

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The winemaking process generates different types of waste and by-products such as grape pomace, grape stems, wine lees and wastewater. Although these residues are mainly produced in the harvest period, they are distributed throughout the year causing environmental problems if they are not properly disposed of. Among wastes, wine lees and grape pomaces constitute a valuable source of bioactive compounds, especially phenolic compounds, with potential uses as food additives, nutraceuticals, and/or cosmeceuticals [1]. For this reason, the winemaking industry is geared towards innovative and sustainable waste management systems, with an increasing demand for recycling or valorization strategies which deal with the principles of the Circular Economy [2]. The coupling of membranes and other physical-chemical treatment appears to be a promising research domain in this field [3]. In this work ultra- and nano-filtration laboratory-made flat-sheet membranes were investigated for the recovery of phenolic compounds from wine lees and red grape pomace extracts. The membrane performance was measured in terms of productivity, fouling index, cleaning efficiency and rejection coefficients towards total phenolics content, antioxidant activity, proanthocyanidins, glucose and fructose. UF and NF membranes were effective in separating target compounds from wine lees rejecting more than 92% of polysaccharides, with polyphenols preferentially permeating through the membrane. Concentrated fractions with high antioxidant activity can be produced through a combination of UF and NF processes in a sequential design. Among the prepared membranes, a specific NF membrane provided the best results in terms of separation between sugars and phenolic compounds of wine lees, with low rejections for glucose and fructose (19% and 12%, respectively) and high rejections for total phenolics content and proanthocyanidins (73% and 92%, respectively). This membrane exhibited also high productivity, with permeate flux of about 50.58 L/m^{2}h (at 20 bar and 25 °C) and low fouling index.

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Tuning ultrafiltration/nanofiltration cascade process to recover enriched fractions of bioactive compounds from beetroot juice

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Membrane technology has proven its efficiency in processing of juices for biomolecules recovery. Beetroot juice (BJ) is attracting researchers attention, due to its human health benefits attributed to its high polyphenols content (50–60 μmol/g dry weight) [1] and red pigments, betalains. Betalains are used as natural colorants and are composed of betacyanins (red to purple color) and yellow betaxanthins. Betalains separation can be achieved by chromatography and/or other techniques, such as aqueous two-phase systems [2], but these are time and reagents consuming. The use of membranes to fractionate BJ has been rarely reported. Ultrafiltration (UF) and reverse osmosis have been used for the concentration of betalains, but their fractionation evidence were absent [3]. The present work aims at filling this gap. A systematic study was performed using cross-flow UF/NF cascade on a prototype scale under constant temperature and pressure. UF membrane (300 kDa) used for removing the suspended solids and NF membrane (400 Da) were selected for separating betaxanthins from betacyanins. Adsorption experiments carried out before rejection measurements showed that UF lost ~53% of pure water flux permeance (PWFP). There was an additional (~39%) PWFP loss after BJ processing up to a volume reduction factor (VRF) of 7.5. However, fouling resulted largely reversible and after cleaning with sodium hydroxide, 95% of the initial PWFP was recovered. As expected, most pigments and polyphenols (98%) passed in UF permeate, whereas turbidity was ~ 90 NTU (~ 70% reduction). NF step proceed up to VRF of 3.5 with rejection of 89% and 35/9% for betacyanins and betaxanthins/polyphenols, respectively. PWFP loss for NF after BJ processing was only ~18%, whereas after cleaning NF has recovered 83% of initial PWFP. Enriched fractions of betacyanins and betaxanthins/polyphenols in NF retentate and permeate, respectively were obtained. Process stability, and recovery factor will be discussed.

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Preparation and characterization of biodegradable films from purified arabinoxylan extract derived from corn fiber

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The corn starch industry produces various by-products in vast amounts, including corn fiber, which is mainly used in animal feed applications. This raw material is very rich in arabinoxylan, a hydrophilic polysaccharide with film-forming properties. In this work, arabinoxylan was extracted from corn fiber with a mild alkaline solution [1] and further purified with membranes processes, more precisely, a pre-concentration step by ultrafiltration [2] followed by purification with ultrafiltration in diafiltration mode [3]. The purified arabinoxylan extract was used as the basis for the production of films for packaging applications [4]. The crude and purified extracts were characterized in terms of bioactivity (cytotoxicity and antiproliferative activity), showing no toxicity to Caco-2 cells (IC50>10mg/mL), which potentiates their application in the production of edible films, and some antiproliferative activity to HT29 cells (the lowest being EC50=0.12 ± 0.02mg/mL), related to a potential anticancer effect [5]. The resulting purified aqueous extract presented an intense brown color. Therefore, decolorization was attempted, for a more appealing appearance, employing activated charcoal or hydrogen peroxide. Decolorization with activated charcoal was not successful, however, partial decolorization was achieved with hydrogen peroxide, resulting in a light-yellow solution [4]. Films were formulated with 30% (w/wdry basis) glycerol as plasticizer. Though presenting a high solubility in water, they showed promising properties to be used as wrapping materials. Decolorized films still presented significant antioxidant activity ($(3.21\pm 0.40)\cdot 10^{-5}$mmol Trolox/mg film), and water vapor permeability values ($(2.94 \pm 0.49)\cdot 10^{-11}$mol·m/m^2·s·Pa) similar to that of non-decolorized films and other polysaccharides. In addition, they showed good mechanical properties under perforation (Tension of Perforation = (1.22 ± 0.41) MPa and Deformation = (53.0±1.7)%, meaning that the decolorization process did not alter significantly the properties of the films. These results show that these films have promising properties for food packaging applications, especially for lower moisture content food products [4].

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PEG-aided isolation of Extracellular Vesicles from citrus limon fruit through cross-flow Ultrafiltration.

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Extracellular vesicles (EVs) are lipidic nanovesicles secreted by cells. They display a prominent role in cell physiology, being involved in several intercellular communication processes. EVs contain a rich bio-cargo of proteins, lipids and nucleic acids that can be exploited in therapies, diagnostics and in drug delivery applications from nanomedicine to cosmetics and nutraceutics. The major challenges in the field are the translation from lab-scale to industrial production and the strict clinical demands, such as the requirement for reproducible and GMP-adherent EVs processing techniques. EVs production is based on the use of mammalian culture-derived EVs, but researchers are currently exploring alternative sources such as edible plants. Many purification techniques are rising as alternatives to the most used lab-scale ultracentrifugation protocols. In the context of process scale-up, membrane-based processes for EVs isolation are the most promising, as stand-alone or used in combination with chromatographic techniques, being flexible, adaptable to continuous operations and time-effective. The aim of this work is to present an integrated membrane process for the purification of EVs from Citrus Limon, that was chosen as model source to develop the process. Citrus Limon represents an economic source of EVs with a promising therapeutic use, as EVs from lemon extracts are proved to inhibit cancer cell proliferation on different cell lines [1]. In the proposed protocol lemon juice extracts are clarified through centrifugation and microfiltration steps and further purified with a diafiltration process carried out in an ultrafiltration cross-flow cell. To increase product yield the use of PEG prior to diafiltration was tested, allowing EVs to precipitate in a polymeric mesh, subsequently resuspended and diafiltered. EVs recovery, PEG and co-isolates removal were mainly assessed through SEC chromatography and their integrity characterized with SEM microscopy. Overall, the preliminary results are promising, displaying the beneficial effect of PEG on the high-purity EVs preparations.

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Novel electrospun nanofibrous anion exchange membranes for high throughput downstream purification of biomolecules

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The development of efficient and sustainable novel technologies for downstream biomolecule purification, such as, proteins and viruses, based on highly porous adsorptive membranes is expected to generate a significant impact on biopharmaceutical applications [1,2]. Nevertheless, the progress is significantly limited by the low specific surface area (SSA) (1-2 m^2/g) of the state-of-the-art membranes prepared by phase inversion that renders limited binding capacity, limited choice of membrane materials and less versatile surface functionalization approaches [2]. Attempts including surface functionalization and fabrication of novel membrane morphology with a significantly high SSA have been performed to enhance the protein binding capacity of membranes [3], however, the responsive intensity of the membranes has never been studied. This work aims to fabricate a novel 3-dimensional poly (ethylene-co-vinyl alcohol) (EVOH) nanofiber membrane matrix by combining electrospinning technique and pre-functionalization mechanism for protein purification. Anion exchange composite membranes were prepared by blending EVOH with ion-exchange copolymer, synthesized via RAFT polymerization. The membranes were characterized by SEM, XPS, FT-IR, TG, NMR, zeta potential, N_2 adsorption, and water contact angle, and tested for static protein adsorption capacity. Surface morphology results confirm the success of the proposed method to fabricate nanofiber membranes (200 nm average fiber diameter) with higher SSA than those reported in the literature, large and highly interconnected pores coupled with the desired anion exchange surface chemistry. Moreover, pre-functionalization mechanism ensures uniform and high ligand distribution and avoids unnecessary mass steric hindrance challenges while allowing broader choices of substrate materials. On the other hand, static adsorption results reveal that pre-functionalization is a promising approach to enhance binding capacity and throughput simultaneously. Moreover, the membrane demonstrated outstanding biocompatibility. This study provides new insight into designing cheap, efficient, and remarkably high throughput membrane chromatography for streaming downstream biomolecule purification in addition to increasing the versatility in terms of material choice.

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Virus adhesion to archetypal membrane surfaces: Implications for virus removal in water treatment systems

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The presentation will describe recent studies aimed at elucidating physicochemical bases of virus adhesion to common membrane surfaces. Adhesion of human viruses (enveloped and non-enveloped) and bacteriophages onto several "archetypal" surfaces is predicted using the extended Derjaguin-Landau-Verwey-Overbeek theory and experimentally assessed in tests with quartz crystal microbalance with dissipation. The results are interpreted based on physicochemical properties of viruses and membranes. The surfaces are typified on the basis of their surface energy components including the dispersive (Lifshitz-van der Waals) component and two polar (electron donor and electron acceptor) components. Surfaces include common polymers, polyelectrolyte multilayers, ceramics, and passivated metals. We show that hydrophobic and electrostatic interactions govern virus attachment while van der Waals interactions play a relatively minor role. In higher ionic strength solutions, the extent of virus attachment correlates with the free energy of virus-surface interfacial interaction. The proposed methodology can guide screening and selection of membrane materials with controlled virus adhesion. The information on the efficiency of virus attachment to membranes as a function of their surface energy components can help design membrane materials, develop membrane cleaning solutions and protocols, and inform transport and fate models for viruses in water treatment systems.

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Liquid Phase Exfoliation Free Energy of Graphenes from Graphite using Umbrella Sampling Simulations

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We employ umbrella sampling molecular dynamics simulations to evaluate the exfoliation free energy of graphene from graphite in different solvents such as dimethyl sulfoxide (DMSO), N-methylpyrrolidone (NMP), N, N-dimethylacetamide (DMA), cyclohexane (CHX) and water. Graphite is modeled as a columnar structure with four parallel graphene nanosheets, the three bottom layers of which define the substrate and the top layer is the one which exfoliates. To visualize exfoliation, we steer the top layer away from the substrate at a fixed pull rate on the shear direction. We divide the path of the exfoliated graphene into a series of intermediate states, where we perform a separate umbrella sampling simulation on each state and compute the free energy differences between the intervals. We show that the overall exfoliation free energy increases as the solvents become less polar. The high free energy implies that the dispersed graphene has small probability to re-aggregate with the graphitic substrate, meaning that is more stable when solubilized. A key parameter for the efficient exfoliation of graphene is that the solvent-graphene interactions must be at least comparable to those existing between the stacked nanosheets in graphite. As an example, cyclohexane yields the highest free energy among the specified solvents because it resembles graphene in its lack of polarity and hydrophobicity. In this regard, cyclohexane molecules have greater affinity with the carbon surface than to themselves providing high repulsive energies when the nanosheets come in contact to each other. We believe that a systematic molecular dynamics study of the liquid-phase exfoliation of nanosheets facilitates the design of better solvents to more effectively induce exfoliation and dispersions, realize the potential applications of graphene layers and 2D materials in general and design molecular models of graphene-based membranes and composites.

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Porous Graphene Oxide (PGO)-ZIF-L Composite Hollow Fiber Membranes with the Tailored Porous Structure for Water Separation

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Though graphene oxide (GO) membranes have drawn considerable interest as a new nanoporous platform for water purification, they suffer from low water permeance due to the tortuosity of permeation pathways and poor structural integrity induced by solvation in water [1]. Here, we proposed a new class of porous GO (PGO)-ZIF-L composite hollow fiber (HF) membranes with nanoscale pore size demonstrating not only ultrahigh water permeance but also good dye rejection performance compared to pristine GO membranes. HR-TEM images displayed the nanoscale pores generated on PGO nanosheets. SEM images showed the uniform coating of PGO nanosheets on alumina UF supports. XRD, Raman spectroscopy, FTIR and XPS analyses were used to gain more insight into the structural and chemical properties of membranes. PGO HF membranes exhibited substantial improvement in water permeance due to shortening water diffusion pathways given by pores created on nanosheets. For instance, PGO membranes made of chemically etched GO nanosheets for 5 hours (named PGO-5h) exhibited the water permeability of 2.98 LMHbar^{-1}, 23 times higher than that of GO membrane The water permeance enhancement in PGO HF membranes is accompanied by the rejection drop (MWCO=430-495 g mol^{-1}) [2]. We developed a new in-situ method to grow two-dimensional (2D) ZIF-L nanosheets on the defects and edges of PGO membranes, leading to PGO-ZIF-L composite HF membranes. Taking the advantage of small pore size of 2D ZIF-L nanosheets (3.4 Å), PGO-ZIF-L composite membranes showed promising size-sieving performance for dye nanofiltration (MWCO$\leq$ 269.3 g.mol^{-1}) without sacrificing water permeance. ZIF-L nanosheets grown between PGO nanosheets led to composite membranes with much better structural stability in the water as confirmed by interlayer d-spacing measurement under dry and wet conditions. To the best of our knowledge, this is the first report on the fabrication of PGO-ZIF-L composite membranes with such high water permeance and great dye rejection.

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Improvement of Water Filtration Performance of Graphene Oxide Membranes by UV-Assisted Reduction Treatment: Control of Molecular Weight Cut-Off

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Reduction of graphene oxide (GO) membranes leads to a removal of surface oxygen groups, which enhances salts and organic compounds rejection, but also affects to hydrophilicity and modifies water transport. The reduction can be achieved by chemical, hydrothermal or radiation [1-3]. In this work, the performance of graphene oxide (GO) membranes in the filtration of polluted water was improved and molecular weight cut-off (MWCO) was controlled by UV-assisted reduction. Pyridine, phenol, 2-naphthol and disperse blue 3 were used as probe pollutants to determinate the MWCO of the membranes prepared. Non-irradiated membranes were prepared with different GO loads, showing MWCO ca. 300 Da, according to rejection values above 90 % for disperse blue 3, while relevant rejections for 2-naphtol (64 %) and phenol (19 %) were obtained. The UV-reduction of formed membranes resulted in the removal of oxygen surface groups, staking of GO sheets, lower interlayer space and narrower pores, enabling 83 % rejection for 2-naphtol, i.e. MWCO close to 150 Da. Rejection increased while permeance decreased using membranes prepared with a higher GO loading, due to higher thickness and higher staking in the uppermost layers due to higher settling during preparation. UV-reduction shows a high potential for tuning the properties of GO membranes and achieving high rejection of small molecules.

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MXene-based Membrane with Facilitated Water Transport

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The discovery of two-dimensional (2D) transition metal carbides as well as nitrides (MXenes) in 2011 has expanded the structure pool and property window of 2D materials available for a wider range of applications. Particularly, MXene-based lamellar membranes bearing interlayers of tunable hydrophilicity are promising for high-performance water purification. The current challenge lies in how to engineer the pore wall's surface properties in the subnano-confinement environment while ensuring its high selectivity. In this contribution, poly(ionic liquid)s, equipped with readily exchangeable counter anions, succeeded as a hydrophilicity modifier in addressing this issue. Lamellar membranes bearing nanochannels of tailorable hydrophilicity are constructed via assembly of poly(ionic liquid)-armored MXene nanosheets. By shifting the interlayer galleries from being hydrophilic to more hydrophobic via simple anion exchange, the MXene membrane performs drastically better for both the permeance (by two-fold improvement) and rejection (~99%). This facile method opens a new avenue for building 2D material-based membranes of enhancing molecular transport and sieving effect.

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Ultratough and Ultrastrong Graphene Oxide Membrane Engineered by Polycationitrile Chemistry

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Graphene oxide (GO) is a benchmark two dimensional (2D) building block to develop high-performance materials for various applications in particular in the energy and environment fields. Currently, the precise assembly of GO nanosheets into macroscopic nanocomposites with both superior strength and toughness faces challenges and trade-offs. Here we exploited “polycationitrile chemistry” as an innovative polymer crosslinking strategy to engineer an ultratough and ultrastrong GO/polymer composite film, in which a covalent triazine-based network was constructed in a mild condition to reinforce the interfacial interactions between GO nanosheets and polymer chains. The tensile strength and toughness reached 585±25 MPa and 14.93±1.09 MJ m^{-3}, respectively, which are to the best of our knowledge the new world-record in all GO-based GO/polymer composite films. As an added merit of the unique polymer crosslinker, the high mechanical performance can be maintained in large part at an extremely high relative humidity of 98%. This emerging interface-engineering chemistry paves a new avenue to produce integrated strong-and-tough 2D nanocomposites that are potentially useful in aerospace, artificial muscle, energy harvesting and tissue engineering.

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Scale-Up of CO_2-Selective Nanoporous Graphene Membranes

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Atom-thick gas-sieving nanoporous single-layer graphene (NSLG) membranes have shown great potential in post-combustion carbon capture. CO_2 permeance of above 10000 GPU and CO_2/N_2 separation factor near 30 from NSLG membranes was recently achieved by our group [1,2], and a competitive capture penalty (30 to 40 \$/tonCO_2) for the case of post-combustion capture was assessed by a technoeconomic analysis [3]. However, the current high-performance graphene membrane is still miniscule and scalable fabrication routes are needed. The most predominant factor that restricts the up scaling of the graphene membrane is the crack formation during the transfer of graphene film from metal foil to a porous support. In this presentation, we will discuss several interventions that we carried out to realize large-area graphene membranes. We adapted the crystallization protocol of graphene to a low-cost Cu foil. We used acid pre-treatment on Cu foil to remove contamination particles that are detrimental to the graphene membrane. The acid treatment in acidic solution was found to be effective even for low-cost Cu foils. High-quality 0.3 m x 0.1 m graphene coupons could be synthesized with a low density of intrinsic defects. The graphene film was mechanically reinforced with a nanoporous layer and was transferred on a low-cost stainless-steel mesh while avoiding cracks and tears. The membrane was assembled inside a customized module and could be sealed without any leaks. The graphene films without any intentional pore generation step showed a low gas permeance confirming a low intrinsic defect density from graphene and crack-free membrane preparation. Incorporating graphene with CO_2-selective pores resulted in attractive CO_2-sieving performance. Our work paves the way to up-scaling graphene membrane and achieving a competitive carbon capture cost.

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Nitrate removal from contaminated groundwater of the Israel Coastal Aquifer by micellar-enhanced ultrafiltration using hydrogel-stabilized ZIF-L layer on polyacrylonitrile membrane

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Contamination of groundwater with nitrate due to intensive agricultural activities is a serious problem globally. In Israel, excessive fertilization led to nitrate contamination in the Coastal Aquifer. Here we show that efficient removal of nitrate from contaminated groundwater is obtained by a specially tailored antifouling membrane using micellar-enhanced ultrafiltration (MEUF). Graft polymerization with hydrophilic poly(methacrylate) [1] and incorporating the porous frameworks ZIF-L nanoparticles [2] were performed as antifouling strategies on poly(acrylonitrile) (PAN) membrane. The membrane showed a high water permeance (82.2 ± 1.7 $L \cdot m^{-2} \cdot h^{-1}$ bar^{-1}). Nitrate removal efficiency by MEUF was tested with cetylpyridinium chloride as surfactant using a solution simulating the groundwater composition in the Coastal Aquifer of Israel and also the nitrate-contaminated groundwater samples collected from the Coastal Aquifer of Israel. The membranes reduced the nitrate levels, depending on the groundwater composition, from 40–70 $mg \cdot L^{-1}$ to 6.8–29.5 $mg \cdot L^{-1}$, which was further reduced to 6.1–24.1 $mg \cdot L^{-1}$ with complete surfactant rejection using 2-stage membrane filtration, in high permeate flux (between 32.1 ± 0.9 $L \cdot m^{-2} \cdot h^{-1}$ bar^{-1} and 45.9 ± 0.6 $L \cdot m^{-2} \cdot h^{-1}$ bar^{-1}). Notably, the modified membrane maintained stable separation performance for multiple cycles, and the flux recovery ratio was >93%. Nitrate removal achieved concentrations that were well below the drinking water standards and therefore enable unlimited use of the treated water. Therefore, the poly(methacrylate) hydrogel/ZIF-L modified PAN membrane has a potential to efficiently remove nitrate in remediation of contaminated groundwater via MEUF.

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Novel Surface Modification Strategies for Blood-Contacting Membrane Applications

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Extracorporeal membrane oxygenation (ECMO) is a heart-lung support device for open-heart surgery and lung failure disease. Currently, the emergence of many serious lung diseases (MERS and COVID-19) has highlighted the importance of blood oxygenation membranes but also revealed that the ECMO membrane has limitations when used for an extended period of time. Nowadays, commercial ECMO membranes are PP and PMP hydrophobic membranes with low biocompatibility due to the blood coagulation over a long operating time. To prevent blood coagulation, the goal of reducing the amount of protein absorbed to the surface is a prerequisite because this is the first step to activating the clotting process. There are three novel approaches to the blood oxygenation membrane, which are superhydrophobic, superamphiphobic, and tethered liquid perfluorocarbon (TLP) coating. With the first approach, the superhydrophobic membrane has showed good hemocompatibility due to the high aqueous repellent. In our recent work, we demonstrated that a superamphiphobic surface for blood oxygenator applications with perfluorocarbon-silane coating on a nanostructure surface displays an excellent property for blood-repelling surface with high repellent properties for both water and oil. The perfluorochemical coating is effective in achieving low surface energy, but it can cause bioaccumulation and biomagnification for the perfluorochemicals that have a carbon backbone chain length from C8 to higher. For the last approach, TLP is a promising hemocompatibility and anti-protein adsorption material that consists of two parts: a tethered perfluorocarbon layer and a mobile perfluoro-liquid. The TLP has the tethered perfluorocarbon layer, so it also faces the same bioaccumulation as the superamphiphobic approach. Furthermore, the mobile perfluoro-liquid layer, which is the core component of preventing protein adsorption, is easily washed away over time. In this study, we compared all three promising approaches with the non-bioaccumulation chemicals to identify the optimal development path for the long-term blood oxygenation membrane.

Selective separation of nitrate and chloride by tuning the hydrophobicity of Anion-Exchange Membranes (AEMs)

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The selective removal or recovery of elements from a multicomponent aqueous stream is relevant for many industrial and agricultural applications. For instance, in closed horticulture irrigation systems the aim is to retain the nutrient nitrate in the recirculating water but to selectively remove the chloride. The technological challenge of this separation is related to the slight difference between these two equally charged ions (NO3 and Cl) in terms of their size and hydration energy. Epsztein et al. [1] explored ion permeation in ion-exchange membranes. Ions were shown to have an activation behaviour of transport – attributed to ion dehydration – according to an Arrhenius-type equation and smaller and more strongly hydrated ions showed a higher energy barrier for transport. Sata et al. [2] showed how the presence of a longer alkyl chain bonded to the quaternary ammonium functional group in the polymeric structure of the membrane increases the nitrate's permeation. Building further on these previous contributions, we studied the selective separation of nitrate and chloride by increasing the hydrophobicity of manufactured anion-exchange membranes (AEMs). Polyvinylidene fluoride (PVDF) was used as a polymer to enhance the hydrophobicity, while a polyaromatic anion-exchange polymer solution (Fumion FAS solution) was used as binder. Different membrane compositions have been manufactured via a facile casting process followed by further characterization (permselectivity, bi-ionic potential, selectivity under dynamic conditions). The membrane selectivity under dynamic conditions has been determined in a six-compartment electrodialysis cell, while varying the experimental conditions (ionic strength, current density). The experimental results show that increasing the PVDF concentration in the membranes increases the nitrate transport by a factor of 1.7. Moreover, experimental data indicated that ionic strength and current density also play a role in membrane selectivity.

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The pH as a tool to tailor the performance of symmetric and asymmetric layer-by-layer nanofiltration membranes

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Layer-by-layer (LbL) assembly of alternating adsorption of polycation and polyanion layers is a versatile method to produce nanofiltration membranes. The pH is an easy parameter to directly tailor the charge density of polyelectrolytes and thus induces major variation in the membrane formation, the morphology of the layers and the performance properties. Here, symmetric and asymmetric membranes are fabricated at different pH (4.8, and 9) with branched polyethyleneimine (PEI)/poly(sodium-4-styrenesulfonate) (PSS) layers (symmetric) or a combination of a polydiallyldimethylammonium chloride (PDADMAC)/PSS base membrane terminated with PEI/PSS layers (asymmetric). For all membranes, a higher pH results in more PEI adsorption due to the lower PEI charge density, as measured by optical reflectometry and a highly positive zeta potential under filtration conditions. Surprisingly though, the membrane performance differs significantly between symmetric and asymmetric membranes coated at high pH. The low charge density of PEI at pH=9 results in limited intrinsic linkages between the polyelectrolytes, giving low salt retentions for the open symmetric membranes. Asymmetric membranes, however, show retentions over 90% for $MgSO_4$ and Na_2SO_4. The base membrane is less positively charged compared to the symmetric membrane, causing the PEI and PSS to interdiffuse into the base structure, resulting in a denser membrane. Additionally, the benefit of asymmetric membrane formation is proven by comparing the best membrane performances. Asymmetric membranes prepared at pH=4 result in much higher permeabilities of ~12 $L/(m^2$ h bar) compared to only ~9 $L/(m^2$ h bar) for symmetric membranes, and a significant improvement of the $MgSO_4$ and Na_2SO_4 retentions (>95% compared to >90%). This is a result of the open base layer and the defect-free thin dense selective layer. By combining two well-known polycations and tailoring the pH, new and versatile nanofiltration membranes are produced, without the need for new synthesis or modification steps while obtaining high and improved water fluxes and salt retentions.

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Functionalized microfiltration membranes for the removal of micropollutants from water

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The use of polymer membranes is of increasing importance for application in modern, efficient filtration technology. These materials with tailored pore sizes are especially interesting for water treatment, sterile filtration, hemodialysis, or beverage production. Recently, studies in Europe demonstrated the presence of more than 150 pharmaceuticals in waste water, surface water, and even ground water. Although the concentration of pharmaceuticals in water is still very low (ng/L range) the concentration of the drug diclofenac reaches already the concentration of chronic fish toxicity. Furthermore, a chronic impact on humans and the environment by single pharmaceuticals as well as their mixture can't be excluded so far. Since common water treatment facilities can't retain or even degrade the compounds from water new methods are currently investigated for removing pharmaceuticals and their metabolites from waste water. The development of functional membrane surfaces allows simultaneous use as traditional filters and for degradation or adsorption of pharmaceuticals or hormones in the water to be filtered. For this purpose, polymeric microfiltration membranes were functionalized with photocatalysts such as TiO2, but also with photosensitizers, enzymes or adsorptive coatings and tested in various applications. The functional membrane surfaces were found to be highly active in eliminating or adsorbing micropollutants such as diclofenac, propranolol and estradiol from water [1,2]. Different reactor types were investigated and evaluated regarding their efficiency to remove the target molecule [3].

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Plasma induced superhydrophobicity for superior direct contact membrane distillation and anti-fouling performance

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Membrane distillation (MD), as a technique for high salinity feedwater treatment, receives increasing interest since many regions of the world are facing water scarcity and the demands for clean water are rising. To be capable of desalination a membrane needs to be porous, in order to allow water vapour to pass through and also hydrophobic, in order to prevent mass transfer of liquids and resist capillary condensation within the pores. While there are several MD methods, in this work we focus on Direct Contact Membrane Distillation (DCMD). In this method the membrane is in contact with liquids on both sides and due to temperature difference between the two streams, water vapour from the hot feed solution passes through the pores and condenses on the cold solution on the other side [1]. Recently superhydrophobicity became a requirement in membrane fabrication and modification since it has been proven to improve performance in terms of distilled water flux, stability, scaling and fouling resistance in long-term MD operations [2]–[5]. In this work we introduce a novel and environmentally friendly method by means of plasma micro-nanotexturing followed by plasma deposition for transforming commercial membranes, both hydrophobic and hydrophilic, to superhydrophobic [6][7]. To this direction, we render superhydrophobic initially hydrophobic Polytetrafluoroethylene (PTFE) as well as initially hydrophilic Cellulose acetate (CA) membranes. All plasma treated membranes give enhanced distilled water flux in MD with very high salt rejection due to superhydrophobicity, increased pore size and porosity. Additionally the bio-fouling resistance of plasma treated membranes was investigated by adding bovine serum albumin (BSA) to the saline solution; plasma treated membranes were able to resist wetting and pore clogging by preventing the adsorption of proteins and give increased permeate flux up to 75 % compared to pristine membrane samples.

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The emergence of functionalized feed spacers in membrane processes

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Feed spacers are indispensable components of spiral wound modules (SWMs), and perform the role of mechanical support for the membrane as well as the development of turbulence in the feed channel. In spite of the growing interest in spacer modification, particularly with the increasing adoption of 3D printing technologies in module development, the spacer has remained a passive component in the feed channel. However, in recent years there has been growing interest in evolving the feed spacer to play a greater, active role in membrane processes. As a result, spacers have been increasingly modified over the past decade, with these modifications broadly classified as geometrical modifications to improve the fluid hydrodynamics for increased flux and reduced fouling, or surface modifications to prevent biofouling and scaling through functionalization of the spacer surface chemistry. Some recent examples of functionalized spacers include the development of stainless steel spacers that can be heated by induction to reduce temperature polarization in membrane distillation (MD) [1], 3D printed feed spacers coated with photocatalytic nanomaterials to target the removal of soluble and insoluble organic foulants in wastewater [2], and 3D printed spacers that are platinum-coated and electrically conductive with the aim of detecting wetting in MD [3]. The success of these studies, among others, points to the potential for spacers to rival membranes as targets for functionalization in membrane processes. The functionalization of spacers offers excellent freedom and versatility for design and optimization, and can overcome many of the limitations facing membrane functionalization, such as pore blockage by the doped material, decrease in permeability and selectivity, and membrane damage during the preparation stage. Spacer functionalization will become increasingly prominent in the coming years as researchers look to harness this potential.

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Understand and characterize the role of organic matter to mitigate fouling in Granular Anaerobic Membrane Bioreactors

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Granular Anaerobic Membrane Bioreactor (G-AnMBR) is a hybrid anaerobic biotechnology combining anaerobic digestion by granular sludge and membrane ultrafiltration. G-AnMBR have a great potential to compete conventional aerobic treatment for domestic wastewater treatment by providing a high-quality effluent with energy recovery through biogas production. However, membrane fouling remains a major limiting element since it induces a reduction in fluxes and/or energy yield. The use of granular biomass has been identified as promoting biological activity, intensifying the process and reducing membrane fouling [1]. Nevertheless, the understanding of fouling phenomena and the identification of foulants in G-AnMBRs are essential to allow the economic validation of the process [2]. A long-term study was therefore conducted for almost 400 days on a G-AnMBR pilot for 4 different filtration flow rates. The G-AnMBR was operated at 25°C with low-strength synthetic wastewater (HRT = 13 h, OLR = 0.8 kgCOD.m-3.d-1) [3]. When the filtration flow was increased by +30%, the fouling rate increased from 0.05 kPa/day to more than 0.5 kPa/day which implies close maintenance frequencies. However, the fouling that has taken place in the G-AnMBR has been shown to be easily reversible in nature. 99% of the resistance caused by membrane fouling is eliminated by hydraulic cleaning. On the other hand, dead-end filtrations were carried out on different fractions of organic matter present in the supernatant of G-AnMBR to identify the major fouling agents and to quantify the synergistic and antagonistic effects of the organic compounds. The results obtained made it possible to carry out a technical and economic analysis to demonstrate that there is a trade-off between processing capacity, the resulting energy and operational expenses, and the footprint and its investment costs.

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On the fouling behavior for the separation of unilammelar liposomes by membranes

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Unilamellar liposomes (ULs) are vesicle like structures composed of a two-lipid concentric layered shell with a size ranging from some nanometres up to hundreds of micrometres. There is an increasing interest in the production of ULs due to their property to host different substances within their core and their capability to act as packaging medium and carrier. In particular ULs have found application in many industrial sectors, such as food, cosmetic, agricultural and pharmaceutical products. The substances that can be added to the UL core are wide ranged and includes drugs, nutraceuticals, pesticides, and genetic materials [1]. In this work, combined production process for concentrated ULs suspensions is presented. Briefly, the formation of ULs by adopting a spinning disk reactor will be described; focus of this work is the post treatment process by means of membrane technologies to separate the formed ULs in a more concentrated suspension. In detail, results from experiment determining productivity, selectivity, longevity will be provided. The latter aspect is evaluated by means of boundary flux measurements, adopting already validated methodologies [2]. Ultrafiltration appears to qualify as a valid approach for post processing to ULs production.

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Parametric Evaluation of Historical Data of Ultrafiltration Systems for Drinking Water Treatment

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Long-term historical data of full-scale ultrafiltration (UF) membrane facilities provide valuable information about the effectiveness of various operating protocols and bridge the knowledge gap between bench scale studies and full-scale plants. Despite being extensively used, there is limited published information on long-term timeseries data of membrane performance, especially as it relates to hydraulic and chemical cleaning. A comprehensive study was undertaken by considering long-term operational data of 6 full-scale UF membrane systems since start-up. The 6 partner UF systems have a total treatment capacity of approximately 700 MLD, treat different raw waters, employ different pre-treatment approaches, have been in operation for 4 to 14 years, and utilize the same type of membrane (i.e., ZW1000). Modeling was used to extract relevant summative parameters of timeseries resistance values as an indicator to assess the performance of frequent hydraulic cleanings (i.e., air scoured Backwash (BW)), periodic mild chemical cleanings (i.e., Maintenance Cleaning (MC)), and infrequent extensive chemical cleanings (i.e., Recovery Cleaning (RC)) over extended periods of time. Linear and non-linear relationships effectively model timeseries resistance values between hydraulic cleanings and chemical cleanings respectively. The study suggests that as membranes age, they tend to foul faster. Recovery of resistance from MC could not be consistently observed, suggesting that MC might not be an effective cleaning approach. The extent of recovery from RC and the rate of change of resistance post RC were significantly impacted by the type of cleaning agent used as well as the age of the membrane. As a result, site-relevant chemical cleaning approaches must be considered to maximize the overall performance of UF membranes. The presentation will expand on the above observations, contrasting long-term performance at the 6 partner facilities.

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Air scour flowrate optimization in a full scale MBR
(Title of your abstract written in times new roman 12 centered)

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MBR high quality effluent production has two main drawbacks: membrane fouling and high energy consumption (Dalmau et al., 2014). Most of the energy is required for membrane air-scouring to prevent membrane fouling. There are several strategies to balance the membrane fouling prevention and energy consumption, ranging from manual adjustment to automatic control systems. Monclús et al. (2015) developed an advanced air-scour flow control system for MBR energy savings (SmartAirMBR®). This work presents a full-scale validation of an improved SmartAirMBR® system for air-scour optimisation. The improvement involves real-time monitoring of TMP and permeability (K) trend as input parameters for a fuzzy logic control reasoning. This study has been developed in one of the biggest flat-sheet MBRs in Europe (Sabadell Riu Sec WWTP), located in Catalonia (NE of Spain). The plant configuration is based on 2 MBR reactors, with 4 filtration lines and 2 levels (upper and lower) each. The plant capacity is up to 36000 m3·day-1 and total membrane surface of 61440 m2 operating at an average of 23 LMH. Each filtration line has a unique aeration flowrate for both levels. Previous simulations results estimated a potential 29% of energy savings per line in one year. However, additional supervisory rules and restrictions were necessary to guarantee a safer implementation (i.e. minimum and maximum air-flow rate and TMP alerts). The validation period will take for 6 months in one line operating in a closed-loop mode. During this period, the TMP, K and fouling rates will be monitored. Validation results will be compared with the evolution of the other 7 lines operating at constant air scouring flowrate. At this moment, after 2 weeks of closed-loop operation, around 10% of energy savings have been achieved. Besides, the TMP and K trends did not show any evidence of extra membrane fouling compared to the other lines.

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Investigating Fouling and Cleaning of Membranes for Cell Recycle in the Continuous Production of Lactic Acid

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Introduction: As we move to reduce our dependance on fossil-based resources, we need to produce valuable products that do not accumulate in and damage our environment. This is the basis of bioeconomy research. Interest in the bioeconomy is increasing as the major challenges facing our planet get worse. Waste that once accumulated in our environment can now be converted into valuable products that do not harm the planet. In this project, whey permeate, a typical waste from the dairy industry, is converted to lactic acid. Lactic acid can then be converted into polylactic acid, a biodegradable plastic. This project investigates the integration of membranes for cell recycle in the continuous production of lactic acid with a particular focus on membrane fouling and cleaning. Materials and methods: Cell recycle via membranes in fermentation processes can increase cell density and lactic acid yield, (Xu et al., 2006). The fermentation of Lactobacillus lactis to produce lactic acid is examined in this study. Batch, continuous and continuous fermentation with cell recycle are compared. Membrane fouling and fouling mitigation methods are studied with the aim to optimize the process. Results: Preliminary results show that continuous fermentation performs better than batch with productivities of 0.91 and 0.45 g/L.h respectively and yields of 0.99 and 0.54 g/g respectively. Increasing cell recycle flowrate delays fouling yet fouling still persists over time. This is an opportunity for research into fouling mitigation and membrane cleaning methods to improve continuous fermentation with cell recycle. Future work: Future work will involve examining fouling mitigation via regular cleaning cycles and the development of an effective fouling control strategy. Physical cleaning strategies including surface flushing will be examined.

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Advances in Membrane-assisted crystallization for biomolecules purification

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Biological drugs are the greatest therapeutic/diagnostic modalities in modern medicine that have made it possible to fight cancer, cardiovascular and infectious diseases, inflammatory and autoimmune disorders, with fewer side effects. Downstream processing (DSP) of biologicals, like monoclonal antibodies (mAbs), are primarily based on labor-intensive, discontinuous, and expensive chromatographic methods, which cause a bottleneck in the production and impact goods and facility costs. Therefore, with the growing global demand for biological medicines to address new therapeutic areas, more economic, scalable and efficient alternatives are needed for incorporation in future biomolecular purification processes [1].

Within this context, membrane-assisted crystallization has been recently proven as radically new and disruptive technology for the extraction and purification of mAbs-products in the solid-state directly from cell-free culture fluids. Functionalized membranes, manufactured by a sustainable method [2,3], together with suitable feed formulations and major process design variables, allowed the recovery of Anti-CD20 mAb from clarified fermentation broth at high level of purity and yield in a continuous and easily scalable membrane-assisted crystallization system, that has been validated at TRL4 [4]. Furthermore, based on capital and costs of goods analysis, this technology appears as a cheaper and viable technological alternative to protein A chromatography for mAb purification and, in perspective, for other biological drugs or other mAb-derived medicines on a case-by-case basis.

On the bases of our current research, it is expected that the adoption of membrane-assisted crystallization for biopharmaceuticals purification will provide a breakthrough advancement in terms of productivity efficiency via continuous manufacturing, cost reduction by process intensification, and enhanced environmental sustainability avoiding extensive use of chemicals, compared to the standard chromatography-based platforms.

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A new design of Solar Air Gap Membrane Distillation for Water Desalination

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Remote arid and coastal areas have dry climates, limited freshwater supplies, and intense solar radiation. Solar still (SS) and photovoltaic (PV) technologies are preferred for small-scale water and power demands. However, these techniques have low thermal efficiency and thus limited water productivity. A compact hybrid system with a novel design combines a solar still air gap membrane distillation process with a photovoltaic panel. Outdoor experiments revealed daily average yields of produced water and power of 5.9 kg/m^2 and 0.68 kWh/m^2, respectively, when solar radiation intensity was 6.4 kWh/m^2. Throughout the day, the specific productivity (permeate flow) of potable water, electric power, and thermal efficiency coefficient is evaluated. The results indicate that as solar radiation increases, productivity increases. The daily yield of the hybrid system increased 1.6-fold when compared to a single-stage AGMD system and cascade solar still arrangement system, and 72% of solar energy was efficiently utilized.

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Tailoring polyvinylidene fluoride membrane hydrophobicity, porosity and thickness to control α-Glycine antisolvent crystallization

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Amino acids are small molecules that form the elementary basis of peptides and proteins crucial for life. They are synthetically produced using antisolvent crystallization resulting in random crystal size distribution (CSD). The control of kinetic and thermodynamic aspects such as rate of nucleation, growth and supersaturation is key to obtain a narrow CSD, saving further downstream processing [1]. The importance of tracking the evolution of crystallizing solution and antisolvent composition along the operation time was established in [2] to understand the transmembrane mass transport behavior responsible for the resulting crystallization dynamic. This study takes a step further by addressing the link between membrane characteristics of the hydrophobic polymeric membranes, polyvinylidene fluoride in this case, and the resulting crystal properties. Membranes were developed using non-solvent induced phase separation, with porosities of 0.83, 0.86 and 0.89, water contact angles of 99, 108 and 119° and thicknesses of 70, 100 and 140 µm, and evaluated according to their performance in membrane-assisted antisolvent crystallization (MAAC). Decreasing membrane thickness, increasing hydrophobicity and increasing porosity resulted in a higher transmembrane flux of antisolvent, a shorter induction time and smaller crystals. Most importantly, for all the studied conditions, the CSD was much narrower than that of commercially available glycine, without any compromise with the crystal purity. This tendency was less apparent for crystals formed at the membrane surface, and they were slightly larger than those formed at the bulk solution. That is attributed to the difference in turbulence and antisolvent concentration between the bulk and solution at the membrane vicinity [3]. MAAC is indeed capable of intensifying crystallization processes by providing a one-step narrow crystal size distribution.

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Nutrient recovery from blackwater digested with an air gap membrane distillation

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Production of commercial fertilizers relies on nonrenewable sources, such as natural gas and mining. On the other hand, most of the nutrients in our food are excreted through our feces and urine. A source-separating system for wastewater streams where greywater and blackwater are transported in separate pipes can be water and resource efficient, and also has a great potential for nutrient recovery. In this study, we focused on digested blackwater for nutrient recovery. The aim was to find suitable conditions to concentrate the nutrients in an Air Gap Membrane Distillation (AGMD) with recirculation of the retentate. In a previous study concentrated digested food waste was concentrated with an AGMD with a pH adjustment to 2.6, to keep the ammonium in the liquid phase [1]. The possibility to couple AGMD with anaerobic digestion or other waste heat source was also studied [2]. The objective of this study was to investigate at which temperature and pH the AGMD could be operated to maintain the largest share of ammonium in the retentate. Digested blackwater was taken from the liquid phase of an Upflow Anaerobic Sludge Blanket reactor and pretreated by a 5µm filtration. An 2^2 factorial experiment with three center points was carried out in May-June 2022. The ammonium was stabilized at pH 2.5, 2.8, and 6 with phosphoric acid. The AGMD was operated at 40 °C, 55 °C, and 70 °C (hot side). The cold side of the AGMD was kept constant, at about 8 °C. The permeate flux was continuously measured every minute. The concentrations of total suspended solids, volatile suspended solids, ammonium, total nitrogen, phosphate, and total phosphorus were analyzed in the feed, permeate, and retentate. The results of the analyses will be presented at the conference.

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Inorganic compounds recovery via membrane assisted crystallization: the membrane role

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Industrial minerals and Critical Raw Materials (CRMs) are of strategic importance to world's economy. CRMs are crucial for high tech products used in everyday life and modern technologies. One of the most important components is lithium. Membrane assisted crystallization (MCr) has been already proved to be a valid technology to crystallize inorganic salts as well as organic molecules. Among the inorganic compounds, it has been utilized for the crystallization of sodium chloride, epsomite, thenardite, lithium chloride, etc. The successful LiCl crystallization was achieved with either polypropylene membranes [1] or alumina membrane hydrophobized via coating with polymethylsilsesquioxane aerogels or fluoroalkylsilanes [2]. The present work demonstrates, both experimentally and theoretically, the inability of various PVDF based-membranes to obtain LiCl crystals. The EDX and Raman analyses, and the calculation of the variation of surface energy 〖Δσ〗 _S between the original membrane and the one used, showed that the limitation of PVDF-based membranes is due to the chemical interaction between lithium and PVDF [3] since, in MCr, the crystallizing solution is in direct contact with the membrane surface. In particular, the application of Fowkes model for the evaluation of 〖Δσ〗 _S allowed to prove the increase of surface energy of the membrane after the MCr process; EDX analysis showed the absence of lithium in the used membrane; Raman spectra revealed the reduction of various CF2 and CH2 vibration modes, and the formation of C═C double bonds. Therefore, the carried out investigations showed PVDF should not be treated as a completely inert material and proved the hypothesis of variation of the used PVDF membranes when in contact with high concentrated LiCl solutions. The results of this work are important for the correct choice of membrane materials to be used in MCr processes intended for the crystallization of highly reactive components (such as those containing lithium).

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Water and salt recovery from mining brines by membrane-assisted crystallization

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Brine discharge from process industries have a critical ecological impact endangering both soil and marine ecosystems. The good news is that brine can be considered not just a waste with potential negative effects on the environment, but also a resource to be exploited [1,2]. In fact, mining brines contain valuable resources, such as Ca, Mg, K and ammonium salts and their recovery is essential to decrease the environmental impact of the mining process while reducing production costs of these products at a commercial level. The aim of this work was the simultaneously recovery of clean water and valuable salts contained in brine from mining water using a membrane-assisted crystallization (MCr) approach [3]. The first part of the work included a theoretical study using the PHREEQC software to estimate which salts can crystallize from the brine. In the MCr process, the driving force is the vapor pressure gradient that forces only vapor molecules to pass through a porous hydrophobic membrane to concentrate the brine above its saturation limit creating a supersaturated environment where crystals can nucleate and grow. On the permeate side high-quality water was produced. This study provides a comparative analysis of two membrane crystallization configurations to treat the brine. In the first configuration the feed solution was kept at a constant high temperature, while in the second one the feed stream passed through a cold crystallizer to produce a thermal shock able to affects the nucleation kinetics and the final morphology of salt crystals. Results have shown that MCr process provide, besides freshwater recovery, the possibility to separate various and different mineral salts from brine playing on the operating conditions of the process. In fact, it was demonstrated that the crystal morphology and nucleation rate depend mainly on temperature and MCr plant configuration operated.

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Advanced membranes for carbon capture applications

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Membrane separation processes are among the most promising technologies for carbon capture applications. The competitiveness of polymer membranes with the currently state-o-the-art solvent-based processes have been shown in several studies. Most of the separation configuration presented in literature are analysed considering membrane properties measured under ideal conditions leading to unrealistic predictions. In order to accelerate the application of innovative materials (i.e: Polymers of intrinsic microporosity, mixed matrix membranes, facilitated transport membranes) in different carbon capture scenarios more realistic conditions needs to be considered during the process design. The effect of real streams conditions, stability issues and membranes characteristics on the process design in terms of separation efficiency, energy requirements and cost will be discussed.

Competitive Sorption of a Binary CH_4/N_2 and a Ternary $CH_4/N_2/CO_2$ Mixture in a Polyimide Model Membrane

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Three molecular simulation techniques to predict the gas sorption isotherms in a glassy polymer membrane in contact with a single- or a mixed-gas reservoir have been tested on a large-scale 6FDA-6FpDA polyimide over a wide range of pressures. Both single- and mixed-gas uptake curves were obtained for CH_4, N_2 and CO_2 over the 0-60 bar range using either a Grand Canonical Monte Carlo (GCMC), an iterative test particle insertion - molecular dynamics (TPI-MD) or an iterative GCMC-MD method. The simulation techniques were first used to obtain the single-gas sorption isotherms and the associated ideal gas sorption selectivities. Further tests were made on a binary 2:1 CH_4/N_2 and a ternary 16:8:1 $CH_4/N_2/CO_2$ gas mixture in equilibrium with the 6FDA-6FpDA matrix. For such mixed-gas feeds, the uptake of each gas in the polymer depends on its gas phase concentration and on its solubility in both the gas mixture and the polymer phases. In the binary mixture, the sorption of N_2 was strongly hindered by that of CH_4. In the ternary mixture, the introduction of the highly-soluble CO_2 at a relatively low partial pressure significantly reduced the sorption of both CH_4 and N_2, although its concentration was insufficient to plasticize the polymer. As such, the mixed-gas CH_4/N_2, CO_2/CH_4 and CO_2/N_2 sorption selectivities were found to differ from their ideal values. Interference effects were characterized by a novel technique which estimates the proportions of molecules of each type of penetrant excluded by competitive sorption for the mixtures under study. The main asset of iterative molecular simulations is their ability to take implicitly into account the interdependence of the different gas concentrations and solubilities as well as the associated changes in the matrices over a large range of pressures and temperatures.

The contribution of energetic and entropic parameters to diffusion selectivity in membrane materials for gas separation

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The synthesis of novel membrane materials with enhanced gas transport properties is essential to promote the exploitation of membrane gas separation technology in emerging fields such as CO_2 capture or H_2 purification. In this quest, (micro-)porous materials play a major role, and in particular, Polymers of Intrinsic Microporosity (PIMs) have been used to design the most recent upper bounds [1] due to their rigid and stiff chains that promote the selectivity for diffusion [2]. Herein, we will present a detailed analysis of the basic transport parameters of several PIMs at different temperatures, with particular focus on the diffusivity of gases in terms of activation energy, entropic selectivity and energetic selectivity. Their performance will be compared with that of other innovative and traditional polymers, having both low and high free volume, to discuss the differences also as a function of their position on the Robeson plots. We will show that the energetic selectivity is a key-parameter governing the materials performance for gas separation applications. For this reason, its optimization must be taken into account during the design and the synthesis of novel polymers for the next-generation membranes. Molecular models will give a deep insight into the polymer structures and, paired to the experimental findings, shed the light on the structure/properties relationships. Experimental and computational approaches will show that the peculiarities of the gas transport in PIMs, and in microporous materials in general, are due to the balanced combination of large interconnected voids and narrow bottlenecks between the free volume elements [3]. Detailed knowledge of the size-selectivity phenomena provides new directions for the synthesis of better performing materials to be used for the preparation of gas separation membranes.

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Separation of raw biogas with PIM based membranes and liquid infused membranes

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Biogas represent an alternative gas fuel where unwanted impurities are removed from CH_4 to avoid corrosion of pipelines or engines and increase its calorific value. The potential of an ultrapermeable benzotriptycene-based polymer of intrinsic microporosity (PIM-TMN-Trip) for the upgrading of raw biogas was investigated. Permeation measurements were performed with model mixtures as well as real biogas from a sewage treatment plant. Permeability and CO_2/CH_4 selectivity for either pure gases or for real biogas up to feed pressure 5 bar were high and lie almost on the recently defined 2019 Robeson upper bound based on ideal permselectivities. Further, a remarkable increase in CO_2/CH_4 selectivity was observed after two weeks of continuous exposure to CO_2 due to a significant decrease of CH_4 permeability. The constant CO_2 permeability and increased selectivity upon ageing suggest that ageing in the presence of CO_2 causes rather a rearrangement than a reduction of the fractional free volume. The mixed gas permeability measurements were performed with high stage-cut in order to mimic a real separation process, and the results confirmed the potential of PIM-TMN-Trip membranes for biogas upgrading. Also we compare price of the membrane module consisting of such functional layer with conventionally available modules for biogas upgrading. On the other hand, we have previously demonstrated that separation membrane units for biogas upgrading based on cheaper thin film composite (TFC) membranes for reverse osmosis can be used under conditions when water condensing layer is formed at hydrophilic polyamide functional layer. The selectivity water swollen barrier is able to remove unwanted impurities such as CO_2 and H_2S due to significantly higher solubility of them in water. The selection of the most suitable thin hydrophilic composite membrane is discussed and other physical or chemical modification of the functional layer were tested.

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Pd-based membranes in an Integrated Membrane Reactor for hydrogen separation

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On an industrial scale, hydrogen is currently mainly produced by natural gas reforming, an endothermic reaction system performed at high temperature (>850 °C) followed by high and low temperature water-gas-shift reactors and final hydrogen purification step(s) [1], where the large amount of CO_2 emissions is the major drawback. Among different technologies related to hydrogen production and purification, membrane-based technologies are considered ideal candidate for substituting conventional systems. The integration of reaction and separation in one step (so-called membrane reactors, MRs) allows obtaining higher conversion degrees, smaller reactor volumes and higher efficiencies compared with conventional systems. Palladium-based membranes have also received a growing interest for production and purification of H_2. Comparing with other membrane gas separation technologies, Pd-based membranes have advantages of high hydrogen flux and exclusive perm-selectivity for H_2 due to the unique permeation mechanism [2]. These characteristics make Pd-based membranes the most suitable device to achieve high hydrogen purity at a reduced cost. H_2SITE has built an integrated Pd-based membrane reactor within INNOMEM project. This actual system, to be proven in operation environment for fuel cell purity requirement, will be presented (Figure 1). H_2SITE systems can be adapted to different feedstocks such as methanol, ethanol and/or ammonia, in distributed applications. Onsite H_2 generation eliminates cost of transportation, compression, and decompression associated to centralized generation, which can amount an average of 80% of the total industry H_2 costs. This technology can be also applied to separate hydrogen from gas blends with concentrations as low as 5%. While hydrogen dedicated infrastructures are built, existing infrastructures can be used for blended hydrogen. Bloomberg New Energy Finance suggest that renewable hydrogen will be produced between 1-2.3 EUR/kg by 2030. However, intercontinental transportation of hydrogen, can double the cost of hydrogen for distances over 1000 km [3].

Acknowledgements

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255. Abstract

3D printed nanofiltration composite membranes with reduced concentration polarisation

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Concentration polarisation (CP) occurs when non-interacting solutes build up near the membrane-feed interface, resulting in a higher solute concertation than that in the bulk solution. This build-up can induce water flux decline, reduce quality of produced water, increase power consumption and operating costs in membrane-based water treatment. There is an extensive literature on methods to reduce the impact of CP through modification of the membranes' surface chemistry and structure. However, these measures have shown limited success since they also negatively impact membrane flux, with limited uptake by industry. Therefore, there is intense interest in methods that would effectively reduce CP without negatively affecting permeance and performance stability over time. Here, we present an innovative approach to minimising concentration polarisation using 3D printed nanofiltration (NF) composite membranes consisting of a NF polydopamine-coated polyvinylidene fluoride (PVDF/PDA) selective layer on a 3D printed asymmetric wavy (patterned) support. The result is a wavy composite membrane with pure water permeance of 14 ± 2 LMH bar^{-1} and molecular weight cut-off of ~550 Da, measured using a crossflow NF setup at TMP= 2 bar and Re= 700, using a range of dyes (rejection > 95% and mass balance >97% for all tests). The CP behaviour of the composite membranes was assessed by filtration of Congo red (CR) dye solution, showing that the wavy pattern significantly reduced the impact of CP compared to flat membranes, with a nearly tripling of the mass transfer coefficient and a 57% decline of the CP factor. CFD showed that these significant performance improvements were due to improved hydrodynamics, with the maximum surface shear stress induced by the wavy structure (1.35 Pa) an order of magnitude higher than that of the flat membranes. These results demonstrate that 3D printing is a viable technology route to reducing concentration polarisation in membrane nanofiltration applications.

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New simplified protocol for the experimental determination and modeling evaluation of intrinsic transport properties of NF/RO membranes

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The development and enhancement of nanofiltration and reverse osmosis membranes is often compromised by the vastly different testing conditions employed by different research groups and by manufacturers. Non-existent standard conditions and non-trivial modeling of the experimental data result in unclear reporting of membrane properties, which ultimately compromises fair comparison between different materials and membranes. In order to support membrane development efforts, standardized testing and modeling protocols for NF/RO membranes should be adopted. This research proposes a new simple while robust procedure that facilitates membrane characterization and the extraction of reliable data. Commercial and non-commercial membranes were rigorously tested and the results were used to validate a new simplified model that describes the mass transfer through NF/RO. This new model is based on the two intrinsic parameters that fully describe membranes: pure water permeability coefficient "A" and solute permeability coefficient "B". Concentration polarization mechanisms affecting the water and salt flux were strongly dependent upon the testing conditions. When these phenomena are not taken into account, significantly different end results and membrane properties may be estimated starting from the same experimental data. Therefore, a new non-dimensional equation was formulated that can be used easily by users to calculate univocal membrane properties starting from simple experimental raw data regardless of the testing conditions, and to evaluate the replicability and the reliability of any new membranes. The model demonstrated in this study may represent a valuable tool to strengthen the already known Open Membrane Database, while also being available for membrane manufacturers.

Super-hydrophobic UiO-66-OPA nanoparticle embedded thin film nanocomposite membrane for organic solvent reverse osmosis

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The organic solvent mixture separation by organic solvent reverse osmosis (OSRO) membrane is a promising method due to its high-energy savings [1]. Therefore, for stable separation, the OSRO membrane must have high stability at high-pressure and in diverse organic solutions and smaller pore sizes of less than 1 nm [2]. Also, the hydrophobicity of the membrane is essential to separate polar-nonpolar or nonpolar-nonpolar organic solvent mixtures for the permeation of the nonpolar organic solvent. Our research group fabricated hydrophobic metal-organic framework (MOF) embedded thin-film nanocomposite (TFN) membranes. Poly(ether ether ketone) (PEEK) polymer which has high stability in organic solvents was cast on the polyethylene terephthalate (PET) non-woven fabric to improve its mechanical strength. The pore size of the membrane was controlled by synthesizing a polyamide layer formed from the reaction of m-phenylenediamine (MPD) and trimesoyl chloride (TMC) on the substrate. However, polyamide is impermeable to non-polar organic solvents because its surface is hydrophilic due to its carboxylate groups, resulting in low permeate (approximately 0.1 LMH bar^{-1}) when separating a nonpolar-nonpolar mixture. Therefore, it is important to use a membrane that shows high hydrophobicity. In this study, zirconium oxide clusters with n-octadecylphosphonic acid (UiO-66-OPA) nanoparticles were embedded into the polyamide layer. The contact angle of the TFN membrane containing UiO-66-OPA was 95.1±7.9˚ which means that the hydrophobicity was improved compared to the membrane without UiO-66-OPA (60.8±5.6˚). Permeation results for polar (ethanol) and non-polar (toluene) organic solvents performed using the dead-end system were achieved at 4.32 and 3.59 L m^{-2} h^{-1} at 30 bar, respectively. This means that hydrophobic membranes can be used in separation processes in various industries such as petrochemicals, pharmaceuticals, and cosmetics to separate non-polar solution mixtures.

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Ultra-permeable MOF-303 hollow fiber nanofiltration membranes fabricated by a novel two-step in-situ growth technique

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Metal-organic frameworks (MOFs) are a class of nanoporous crystalline materials that hold great promise for molecular separation. However, the preparation of defect-free MOF membranes and their structural stability in aqueous media are two main technical challenges that pose a big obstacle to their practical realization for water purification. MOF-303 is a new class of water-stable MOFs with outstanding performance firstly developed for water harvesting [1]. High water sorption capacity and great hydrolysis resistance endowed by the strong azolate-based linker make MOF-303 an appropriate candidate for water separation application. Recently, a new class of MOF-303 membrane was grown on alumina flat-disc which demonstrated good potential for water desalination [2]. However, to avoid the formation of non-selective defects, a thick membrane (> 4 µm) was fabricated leading to very low water permeance (<0.8 LMH bar^{-1}). In this study, we proposed a novel two-step in-situ growth approach to fabricate MOF-303 hollow fiber (HF) membranes with promising water permeance and size-exclusion ability. Taking the advantage of metal sites available on alumina HF support, we fabricated thin/defect-free MOF-303 membranes with a crystallographically preferred orientation that demonstrated promising water permeance in the range of 5-7 LMHbar^{-1} and size sieving (MWCO ≤ 269 g.mol^{-1}). This outstanding water permeance is mainly ascribed to two main factors: (i) the hydrophilic, one-dimensional (1D) pores of MOF-303 (0.6 nm) oriented preferentially and (ii) the lower thickness of the selective layer. Indeed, alumina HF substrate provides abundant Al-based metal clusters to form a seed layer of MOF-303 crystals in the first step of in-situ growth followed by the second in-situ growth to further grow MOF-303 crystals without any defects, as confirmed by SEM images and rejection test. Importantly, the developed MOF-303 HF membranes showed promising water permeance and separation factor for alcohol dehydration indicating their great potential for water-based separation.

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Experimental design of a lab-scale nanofiltration system for the treatment of cooling tower blow down water of thermal power plants

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Cooling towers of thermal power plants are responsible for large amounts of freshwater abstractions. Due to water scarcity, process optimisation is necessary for treating and reusing cooling tower blow down (CTBD) water, aspiring to achieve near zero liquid discharge. This work presents an experimental design of a lab-scale nanofiltration (NF) system for the removal of bivalent ions (Ca^{2+}, Mg^{2+} and SO_4^{2-}) from CTBD water that would allow the reuse of treated water as partial make-up of the cooling tower with the minimum specific energy consumption (SEC). Experiments with different conditions of transmembrane pressure (TMP, 1.5–8 bar) and feed flow rate (2–34 L/min) were carried out with commercial spiral wound (2.5 inches diameter) and hollow fiber (8 inches diameter) NF membranes using real CTBD water. During the lab-scale experiments, parameters such as pressure, temperature, conductivity, turbidity, flow rates and velocity, SEC and major ion concentrations for the NF permeate and concentrate streams were monitored, including the parameters for biological and particulate fouling. Special attention was given to the study of saturation indices for measuring the scaling tendency of the sparingly soluble salts, such as calcium carbonate and barium sulphate. The results suggested that, as expected, with an increase in TMP there was an increase in recovery and concentrate conductivity. With the spiral wound module, recovery reached 80% at TMP of 6 bar and feed flow of 1.9 L/min, while with the hollow fiber module a recovery of 60% was achieved at 2.4 bar with a feed of 16.7 L/min. The rejection of Ca^{2+} and Mg^{2+} ranged between 70% and 80%, while sulfates' rejection was around 95%. The conclusions may be applied as a tool for the scaling-up of the CTBD treatment process to the pilot scale in conjunction with other parameters, such as chloride removal and SEC optimisation.

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A New Library of Loose Polyester Nanofiltration Membranes with High Permeability and Antifouling Properties

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Polyamide thin film composite (TFC) membranes are the most commonly used nanofiltration and reverse osmosis membranes to treat and desalinate wastewater. Even though polyamide membranes perform remarkably well, they unfortunately have low chlorine resistance, poor thermal stability, and high fouling propensity. In this work, we made new nanofiltration polyester TFC membranes through the interfacial polymerization of terephthaloyl chloride (TPC) and sulfonated kraft lignin (SKL). We also compared our new polyester TFC membranes with a traditional polyamide TFC membrane made by copolymerizing piperazine (PIP) and trimesoyl chloride (TMC). Zeta-potential measurements showed that our new polyester TFC membranes had higher negative surface charges than the conventional polyamide TFC membrane. Higher negative surface charges favor the rejection of negatively-charged solutes such as reactive red, reactive black, and methyl orange, as well as most foulants such as sodium alginate and bovine serum albumin. The new polyester TFC membrane prepared with 5 wt% SKL and 0.2 wt% TPC had a 66.7 LMH ($L.m^{-1}.h^{-1}$) water flux and dye rejections of 100 % (reactive red), 99.1 % (reactive black), and 87.78 % (methyl orange). This corresponds to a more than 5 times increase in water flux and almost the same dye rejections of the conventional polyamide membrane. We may hypothesize that, compared with highly reactive monomers such as PIP and TMC, the slow rate of interfacial polymerization between SKL and TPC increased the free volume of the polyester structure, leading to higher membrane permeability but keeping a similar dye rejection performance. Our results could be used to develop non-polyamide membranes that can potentially compete with loose nanofiltration membranes available in the market in terms of separation performance and antifouling properties.

Advances in membranes and monoliths for biopharmaceutical product capture and purification

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This presentation will focus on recent developments in membranes and monoliths as alternatives to column chromatography for product capture and purification in a wide variety of different applications including protein therapeutics, vaccines, oligonucleotides, and gene therapy. Novel nonwoven membrane manufacturing approaches enable the production of electro-spun, spun-bond, or melt-blown fabrics with micron- and sub-micron diameter fibers for high surface areas, but large porosities (80-85%) that result in flow permeabilities more than an order of magnitude greater than those of process chromatography columns packed with resins. This allows for process operations with high superficial velocity (residence times < 1 minute) with low pressure drops. These processes can involve rapid cyclic bind-and-elute steps using small volume devices, flow-through operations for polishing, and modular cassette membrane systems for scale-up. Our laboratory has developed a variety of ion exchange and multi-modal ligand salt-tolerant nonwoven membranes that exhibit dynamic binding capacities that compete well with resins but can be operated at residence times of a few seconds rather than minutes as required in column chromatography. The high dynamic binding capacities are achieved by UV- and heat-induced grafting of hydrophilic coatings that, upon modification by addition of charged groups, expand at low ionic strength and contract at high ionic strength. This results in high binding capacities and high elution yields of bound product. The hydrophilic coatings reduce binding of host cell proteins and other contaminants. We have succeeded in using these membranes for product capture applications involving monoclonal and single-chain antibodies, globular proteins, and viral vectors (AAV). They also work well in a flow-through mode for host cell protein (HCP) and DNA removal, enabling end-to-end downstream purification processes using only membrane devices. Because of the low cost of production of these nonwoven fabrics, these membranes exhibit great promise as single-use devices.

Membrane adsorbers for the valorization of aqueous waste streams

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Many waste streams in the dairy industry are currently discarded and valuable resources, such as lactose, minerals and proteins are lost because cost effective technologies to selectively recover these components are not available. Electrospun membrane adsorbers offer a promising separation platform technology to selectively and cost-effectively harvest these valuable components, because electrospinning allows: (1) tuning the functionalization of the electrospun membranes by blending polymers, by embedding functionalized particles during the spinning process or by chemical post functionalization, (2) tailoring the permeability and selectivity by adjusting the bed height, porosity and fiber diameter such that non-binding and coarser impurities easily elute through the bed while the desired components can bind to the (functionalized) electrospun fibers, (3) easy production and linear scale-up [1]. Here we report the fabrication, characterization and performance evaluation of electrospun sulfonated poly(ether-ether-ketone) (SPEEK) membrane adsorbers. The sulfonated groups enable selective binding of Lysozyme at neutral conditions where other bigger proteins are eluted through the membrane. Contrary to slow diffusion controlled (chromatographic) bead adsorption the adsorption on the electrospun fibers is convectively controlled and thereby much faster. The static adsorption capacity was 69 mg Lysozyme/g membrane. Dynamic adsorption experiments of a stack of 58 membranes (0.5 mm bed height) showed a high water permeability of 144 L/($m^2 \cdot h \cdot bar$) (> 100 bed volumes per hour) with a dynamic adsorption capacity of 18 mg Lysozyme/g membrane at 10% breakthrough. This low hydraulic resistance allows using higher bed height or arranging more beds in series (merry-go-round principle) which will increase the dynamic capacity at 10% breakthrough enormously [2]. The current research focusses on further tailoring the porosity of the electrospun membrane adsorbers to optimize the water permeability and dynamic adsorption capacity.

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Mimicking Nature using immobilized Rubisco on polyethyleneimine/dopamine codeposited membranes for CO_2 capture and conversion

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CO_2 capture and valorization is an emerging need to achieve reduction of global CO_2 emissions. Using amine-based solvents to capture post-combustion CO_2 gas leads to energy intensive processes and use of toxic solvents. Benign water-based solvents are an attractive alternative to avoid additional environmental concerns [1]. Despite the poorer performance of water-based solvents compared to amine-based solvents, the incorporation of enzymes have shown improvements in its CO_2 capture performance [2]. In this work, the enzyme ribulose-1,5-biphosphate carboxylase/oxygenase or Rubisco is proposed as the biocatalyst to facilitate the conversion of CO_2 into high-value products. This enzyme is responsible for converting inorganic CO_2 into organic acid during photosynthesis. However, in spite of being the most abundant enzyme in Nature, it has a slow turnover rate and poor CO_2/O_2 specificity [3]. In addition, its application can be limited by its short service life. In this study, Rubisco is covalently immobilized on porous membranes to improve its stability. Dopamine (DA) and polyethyleneimine (PEI) were used for surface modification to improve enzyme compatibility, while glutaraldehyde was used for covalent immobilization on the coated membrane. The effect of coated membrane on retained enzyme activity, reusability, storage stability and CO_2 conversion performance were investigated. Initial SEM results confirmed successful coating on the porous membranes using DA and PEI. On the other hand, the presence of new peaks at 1500-1800 cm-1 and 2800-3750 cm-1 in ATR-FTIR spectra confirm the presence of the DA/PEI coating and Rubisco immobilization, respectively. With this study, preliminary steps are taken in exploring the potential of biocatalytic membrane contactors for CO_2 valorization into organic compounds.

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Hydrogel-based, single-batch use membrane chromatography for highly productive, low risk, and scalable process chromatography

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Alternative stationary phases are being developed to solve issues of bead chromatography processes, namely high diffusional resistance leading to long processing cycles (4-6 h). Targeted life-time of 75 -150 bind-and-elute cycles, taking sometimes years to be achieved, requiring cleaning and respective validation, storage, sometimes even column un- and repacking, all introducing cost and increasingly bioburden risks. Chromatographic membranes, due to intrinsically high mass transfer rates, overcome these limitations, enabling residence times of seconds and full life-time utilization (75-150 cycles) in 1-2 shifts. Here, we present a novel chromatographic membrane platform based on a functionalized, self-structured hydrogel with high specific binding capacities. Its material composition, a detailed microscopic analysis of its functional structure (Kryo-SEM, CSLM) as well as the manufacturing process principles of the membrane will be correlated to its performance characteristics. The membrane exhibits short diffusional path lengths (2-3 µm) providing fast mass-transfer and thus high dynamic binding capacities even for large proteins. Large convective pores (approx. 4 µm) transport those target molecules to the hydrogel interphase, ensuring low fouling propensity and ease of cleanability, as well as high permeability, providing the basis for uncompromised scalability. Supported by modelling approaches we will introduce to the mass transfer concept of this novel membrane material and show residence time dependent binding capacities for different functionalities (affinity, IEX, MM) and target molecules exemplifying its capabilities in direct comparison to state-of-the-art chromatography resins. Focus will be set, however, on a protein-A affinity membrane, with average binding capacities of around 40 g/L at residence times of 12 seconds. In addition, we provide data showing that this technology reduces development costs of new therapeutics and that the approach of using a new membrane for each batch can compete with economical commercial processes while eliminating bioburden risks associated to the long re-use of packed bed columns.

Metal-phenolic network for sustainable fabrication of cellulose membranes

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Separation applying membrane technology has recently revolutionized various industries because of energy-saving and environmental advantages. Nowadays, regulations on the environment and orientation of sustainable development has been increasingly strict. Therefore, in membrane fabrication, innovative research to substitute petro-derived conventional polymers and toxic solvents with natural polymers and environmentally benign solvents is necessary to plow the way through a green path. In this work, a sustainable fabrication of cellulose membrane with its characterization and advantages for organic solvent nanofiltration (OSN) will be discussed in detail. Cellulose acetate was used to prepare cellulose membrane by NIPS technique using methyl lactate - a promising candidate for green solvent. The fabricated membrane was processed with thermal treatment followed by deacetylation by an alkaline solution to convert cellulose acetate into cellulose membrane, which is stable in strong organic media such as DMF, NMP, DMSO and THF, etc. Furthermore, a novel 2-step coating with combination of polyphenol and metal salt was applied on top of cellulose membrane in order to significantly improve OSN performance of as prepared membrane. The rejection performance of polypropylene glycol (PPG) in various organic solvents were recorded with an impressive increase from around 35% to 100% when testing pristine and coating membrane, respectively. Additionally, various performance was obtained with different coating membranes using other metal salts for one type of feed, not to mention producing consistent trends in typical organic solvents, suggesting that the proposed fabrication can achieve appropriate application in membrane technology.

References

Metal-phenolic network, cellulose membrane, green solvent, sustainability

High throughput ion exchange membrane chromatography in downstream bioprocessing

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The separation and purification of valuable biomolecules such as antibodies and proteins, play an increasingly important role in biopharmaceutical applications supporting the development and production of therapeutics, immunodiagnostics and vaccines [1]. The drastic increase in market demands, such as recent surge in vaccine productions related to the Covid-19 pandemic, has induced great challenges on biopharmaceutical manufacturing, particularly in downstream processing [2]. To this end, membrane chromatography is recognized as a potential solution with enhanced process throughput, scalability and production continuousness [1]. In particular, the success of ion exchange membrane chromatography (IEMC) as a polishing step to remove impurities in bioseparation has been demonstrated in laboratory and preparative scales [3]. Nevertheless, the guidelines for designing high throughput IEMCs in bioprocessing are still unclear. In this study, based on the review of recent development in the literature and our research, we aim to identify the key performance-determining factors in the design of high throughput IEMC systems. Although some of the modern laboratory-made membranes with polymeric chains of surface charge exhibited much higher binding capacity that is equivalent or beyond that of traditional resins [4,5], a trade-off was identified considering the significant loss of process throughput (i.e., permeability) due to the grafted layer on the surface, e.g., up to 40%. We thus highlight the recent advancements in nanofibrous membranes with 3D structure and high throughput as compared to conventional type. The roles of membrane characteristics and flow dynamics in affecting the protein binding performance of the membrane, selectivity and process throughput are critically analyzed. Membranes of different types in terms of the effects of the structural parameters and surface properties on the protein binding performance, highlighting the importance roles of specific surface area and charge properties. Overall, a comprehensive analysis was provided on the design principles of next generation high throughput IEMC systems.

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NEW NON-POROUS MEMBRANES FOR FLUE GAS PURIFICATION

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A new and unique apparatus for testing of flat sheet dense membranes with humid mixture air-SO_2/CO_2 was constructed [1]. A polymer of intrinsic porosity with tetramethyltetrahydronaphthalene and bicyclic triptycene units (PIM-TMN-Trip) shows excellent separation properties for CO_2/N_2 as well as for SO_2/N_2 gas pairs. Determined permeability of CO_2 (with values ranging from $16.5{\cdot}10^3$-$18{\cdot}10^3$ Barrer, caused by adging oth material) was somewhat lower compared to the literature [2]. Mixed gas selectivity corresponds to the reported ideal selectivity with values located between 2008 Robeson's upper bound [2] and recently redefined CO_2/N_2 upper bound for pure gases [3]. The permeability of SO_2 in a model mixture air-SO_2/CO_2 was very high ($28{\cdot}10^3$ -$30{\cdot}10^3$ Barrer) and the SO_2/CO_2 mixed gas selectivity was low (ca. 1.8) but comparable with other novel membranes which have shown good separation properties for CO_2 separation [4]. The effect of feed pressure on CO_2 or SO_2 permeability was found negligible, but the stage cut strongly increases due to the very high permeability of the membranes. Obtained results in mixed gas separation conditions show promising potential of PIM-TMN-Trip membranes for efficient flue gas purification.

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Carbon Nanomembrane (CNM)-Composite Membranes for Demanding Water Filtration

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Carbon Nanomembranes (CNMs) are a unique class of molecular thin, two-dimensional sheet materials with a high density of intrinsic pores in the sub-nanometre range [1]. It has been previously shown that water passes with an extremely high flux through CNMs whereas dissolved species like ions or organic compounds are blocked almost entirely [2]. In order to make these unique properties of nanometre-thin CNMs accessible for technical applications, we have developed a composite membrane with the CNM as active layer free-standing over micron-sized pores in a polyethylene terephthalate (PET) support film. We are currently able to fabricate this composite membrane in sheets with a size of up to 30 x 30 cm² or on 25 mm wide rolls. With this laboratory-sized production, it is possible to demonstrate demanding separation tasks in small simple experiments. These include the extraction of water from solutions by forward osmosis e.g. for mild concentration of beverages. Furthermore, we have shown that CNM-composite membranes are suitable as pervaporation membranes by effectively extracting water from ethanol-water mixtures at room temperature using only dry air on the permeate side. This was demonstrated by concentrating rosé wine ino red wine. In the next step, we are preparing for a pilot production with a capacity of several 10,000 m²/year in order to demonstrate the performance of CNM-composite membranes in real processes.

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Preparation of Aamine-Intercalated Graphene Oxide Membranes and Their Application for the Removal of Synthetic Dyes in Water

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Graphene oxide (GO) are ideal material for the fabrication of high performance membranes. The GO membrane properties can be improved by chemical modification, being interlayer spacing and chemical composition key parameters in application for water purification [1-2]. Synthetic dyes are pollutants present in many industrial wastewaters. Due to its toxicological and environmental effects, the study of their removal from water has increased recently, since conventional techniques have difficulties in their treatment [3]. In this work, GO membranes (0.04 mg cm^{-2}) supported on Nylon have been prepared by pressure-assisted filtration. Membranes with chemical modification were prepared, using GO and amine solution. Amines of different molecular weights (MW) have been used: ethylendiamine (EDA), 1,3-diaminopropane (DAP), and 1,4-diaminobutane (DAB), with solution concentrations between 0.001–0.5 M. Membranes were characterized by SEM, XRD, and XPS, and were tested in dye filtration using a cross-flow device (4 cm2) at 2 bar, using 2 mLmin^{-1} of Disperse Blue 3 (DB, 120 ppm). Amine intercalation was analyzed by XPS, with the appearance of the C-N bond (285.4 eV). A reducing effect caused by amine intercalation was also noted, with a decrease in the atomic amount of oxygen from 30 to 20 %. XRD analysis showed an increase in the interlayer spacing of the membranes, mainly related to the higher amine concentration and the higher MW of the amine. Thus, an increase in the interlayer spacing from 8 to 11 Å was achieved. Amine intercalation in the GO membrane led to a significant increase in flux, from 1 to about 3 L h^{-1} m^{-2} bar^{-1} while the rejection of DB remained above 99 % in all cases. Flux reached a maximum for membrane prepared with 0.01 M of EDA, indicating that amine excess could block the channels, and for the higher amine MW used, DAP and DAB.

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Transforming Ti_3C_2Tx MXene's Intrinsic Hydrophilicity into Superhydrophobicity for Efficient Photothermal Membrane Desalination

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Owing to its 100% theoretical salt rejection capability, membrane distillation (MD) has emerged as a promising seawater desalination approach to address freshwater scarcity. Ideal MD requires high vapor permeate flux established by cross-membrane temperature gradient (ΔT) and excellent membrane durability. However, it's difficult to maintain constant ΔT owing to inherent heat loss at feedwater side resulting from continuous water-to-vapor transition and prevent wetting transition-induced membrane fouling and scaling. Here, we develop a Ti_3C_2Tx MXene-engineered membrane that imparts efficient localized photothermal effect and strong water-repellency, achieving significant boost in freshwater production rate and stability. In addition to photothermal effect that circumvents heat loss, high electrically conductive Ti_3C_2Tx MXene also allows for self-assembly of uniform hierarchical polymeric nanospheres on its surface via electrostatic spraying, transforming intrinsic hydrophilicity into superhydrophobicity. This interfacial engineering renders energy-efficient and hypersaline-stable photothermal membrane distillation with a high water production rate under one sun irradiation.

Synthesis and Characterisation of Zn/Ag Modified Graphitic Nitride Nanocomposites and Its Use in the Degradation of Organic Pollutants in Water

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Herein, this research work focuses on utilizing of layered graphitic carbon nitride (g-C_3N_4) for water treatment. Knowing that South Africa is a water scarce country with limited drinking water therefore, strategies on water recycling are a necessity. This project has adopted photocatalytic nanocomposites for the degradation of pollutants in wastewater. Photocatalytic zinc oxide (ZnO), silver oxide (Ag_2O), graphitic carbon nitride (g-C_3N_4) nanocomposites; g-C_3N_4/ZnO, g-C_3N_4/Ag_2O, and g-C_3N_4/ZnO/Ag_2O were synthesized via the hydrothermal synthesis and chemical precipitation reaction. The functional groups of the prepared materials were confirmed via the Fourier Transform Infra-Red (FTIR) Spectroscopy for the unmodified g-C_3N_4 and the Ag_2O and ZnO modified nanocomposites. The X-Ray Diffraction (XRD) analysis also confirmed the presence of these nanoparticles embedded onto the g-C_3N_4 nanosheets. Remarkably, the original lattice structure of the g-C_3N_4 nanosheets were maintained as the nanoparticles were embedded. However, newly formed diffractions were observed as the new nanoparticles were successfully embedded. The Scanning Electron Microscope coupled with Energy Dispersive X-Ray Analysis (SEM-EDX) further confirmed the presence of C, Zn and Ag indicative of the presence of the g-C_3N_4, g-C_3N_4/ZnO, g-C_3N_4/Ag_2O, and g-C_3N_4/ZnO/Ag_2O. Furthermore, thermogravimetric analysis (TGA) results demonstrated thermal stability of the materials. The band gap of the nanocomposites (g-C_3N_4/ZnO, 2.8 eV) was determined via Ultra-violet Differential Reflectance Spectroscopy (UV–Vis DRS) demonstrated a reduction as compared to the pure ZnO (3.3 eV). Lastly, the g-C_3N_4 modified nanocomposites excited at 300-350 nm emitted light at between 400-450nm, suggesting photocatalytic capability of the modified nanocomposites. The prepared nanocomposites demonstrated a high photocatalytic performance on methylene blue with an efficiency of above 80% within 1 hour of irradiation. These nanocomposites were later impregnated onto carrageenan which has sulphate groups which will act as electron pool. These nanocomposites will enhance the surface properties and the photocatalytic behavior of biopolymeric membranes for water treatment applications.

Study of the relationship between the stability of film-forming dispersions and the properties of biopolymeric films and membranes.

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Usually mechanical, structural and transport properties are studied to evaluate the effectiveness of membranes as separation systems or packaging films as barrier systems.[1] In the latter case, optical properties are also important because ultra violet (UV-light) protection is strictly required.[2] Less attention is, however, paid to the properties of film forming dispersions, which characterization was poorly addressed, especially regarding its correlation with produced films or membranes. In literature, the miscibility of polymers was largely studied on solid-state films,[3] stability is evaluated in terms of zeta potential,[4,5] but there are not correlation with dispersion stability. In this work, we propose a methodology based on multiple light scattering (MLS) analysis to characterized film-forming dispersions stability with the aim to study whether and how their characteristics affect the morphology and optical properties of the films or membranes produced.[6] To do that Turbiscan by Formulaction, instrument based on MLS analysis, has been used to do a complete in-depth characterization of film-forming dispersions, by visualization of the main phenomena of destabilization for long time.[7] Samples of different film-forming dispersions, based on biopolymer, were characterized by Turbiscan, to identify possible phase separation and to evaluate the degree of miscibility among components already in the dispersed phase. A stability index of dispersions (TSI, Turbiscan Stability Index) has been calculated by comparing backscattering and transmission profiles during the time to compare different dispersions. Temperature effect has been also evaluated. Starting from these dispersions, films and membranes have been produced with different techniques and characterized. The morphology was analyzed by optical microscope and the degree of homogeneity was compared with TSI dispersion values. Mechanical properties and permeability were also correlated to the dispersion stability and TSI values. Turbiscan-based analysis of dispersions was useful as a preliminary step to forecast the final film quality.

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Nanoparticles for membrane performance enhancement

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Nanoparticle addition into polymeric membrane structures is a common practice aiming towards performance enhancement such as increased salt rejection, flux, mechanical and thermal properties. The general term mixed matrix membranes refers typically to the incorporation of inorganic matter into polymer matrices in a uniform and controlled manner in an effort to combine the best of the two worlds. A vast number of nanomaterials have been used incuding porous and non-porous such as zeolites, nanocarbons, metal oxides and metal organic frameworks. Main challenges towards a successful development of the targeted membrane is nanoparticle dispersion, the interface adhesion and particle wetting, the nanoparticle functionality as well as the minimization of defects.

The current presentation will demonstrate the use of nanoparticles in membranes for both gas and water treatment applications focusing in carbon based materials and their functionalization, and superhydrophobic inorganic nanoparticles.

Several paths can be followed for implementing nanoparticles which involve their incorporation during membrane formation either by adding them in the polymeric dope or the non-solvent bath.

Another interesting approach is to introduce the nanoparticles as a coating on top of a membrane structure in an attempt to affect the interface between the membrane feed and separating layer. The latter although brings more resistance to the membrane structure appears to have beneficial effects in specific applications such as membrane distillation (Fig. 1).

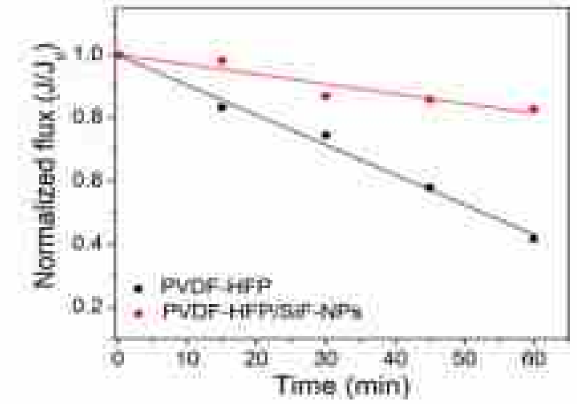

Fig. 1. Membrane performance in BSA fouling test-normalized flux (J/Jo) *vs* Time.

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Molecular characterization of CO_2/CH_4 and CO_2/N_2 gas separation under very high temperatures and pressures in hybrid organic-inorganic network membranes

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When new materials are being developed, atomistic simulations, such as molecular dynamics (MD) and Monte Carlo (MC), can be used prior to experiments in order to pre-screen a large number of candidate structures under various operating conditions. This is even more critical for high temperatures and pressures since these are difficult to implement in a laboratory and potentially hazardous. In addition, mixed-gas measurements are much more complicated than pure-gas conditions. Within this context, molecular modelling is not aimed at replacing the experiments, but at selecting the most promising structures and avoiding losing time on less interesting ones. It also provides a detailed molecular understanding of the materials and their transport properties, which is difficult to obtain experimentally. This work illustrates this approach by comparing at 300°C and 400°C the CO_2/CH_4 and CO_2/N_2 sieving properties of a series of hybrid network membranes based on inorganic silsesquioxanes hyper-cross-linked with small organic PMDA or 6FDA imides. The inorganic precursors are the octa(aminopropyl)silsesquioxane (POSS), which degrades above 300°C, and the octa(aminophenyl)silsesquioxane (OAPS), which is expected to resist well above 400°C. As such, the polyPOSS-imides were tested at 300°C only, while the polyOAPS-imides were tested at both 300°C and 400°C. Following an extensive screening phase on 22 structures, six polyOAPS-imides and two polyPOSS-imides were further simulated in order to assess their N_2, CH_4 and CO_2 permselectivities under such harsh conditions. The feed gas pressure was set to 60 bar in all cases. The models were first analysed under single-gas conditions, but to be closer to the real processes, the networks that had the best CO_2/CH_4 and CO_2/N_2 ideal permselectivities were also tested with binary-gas 90%/10% CH_4/CO_2 and N_2/CO_2 feeds. Two polyOAPS-imide model networks were able to maintain their CO_2/CH_4 and CO_2/N_2 sieving abilities above 2 at 400°C which are outstanding performances for polymer-based membranes.

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The Effect of Solution Casting Temperature and Ultrasound Treatment on PEBAX MH-1657/ZIF-8 Mixed Matrix Membranes Morphology and Performance

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Abstract:

Mixed Matrixes Membranes (MMMs) are composite membranes made by combining a filler (dispersed phase) and a polymer matrix (the continuous phase). By using two materials with different transport properties, these membranes have the potential to synergistically combine the easy processability of polymers and the superior gas-separation performance of filler materials, and therefore provide separation properties surpassing the Robeson upper bound. One of the key challenges in creating effective MMMs lies at the polymer-filler interface wherein performance hindering defects can occur, such as chain rigidification, pore blockage or voids. The fabrication route and material selection are key in minimising these defects.

In this work, MMMs were created using PEBAX MH-1657 as polymer and ZIF-8 as filler. The effect of casting plate temperature, varying from −5 °C to 50 °C, and the effect of ultrasound treatment (time and method) have been investigated, aiming to reduce defect formations hence improving the performance of the MMMs. SEM images, permeation, XRD and DSC experiments were used to study and compare the membranes morphology and performance. The results indicated that the MMMs maintained their permeabilities and selectivities at all tested casting temperatures. For, the neat PEBAX membranes, the casting temperature induced a larger impact, due to the phase separation between the polyamide and polyether oxide phases at high temperatures, causing a reduction in permeability due to the higher crystallinity degree, confirmed by DSC and XRD experiments. The MMMs fabricated with the lower ultrasound time exhibited a large amount of aggregation with large particle size causing channelling. At long ultrasound time, a well-dispersed filler with small particle diameters was observed, providing better membrane performance. Overall, defect-free membranes were successfully fabricated, leading to improved performance, with the best membrane resulting from the longest ultrasound time reaching the Robeson bound upper limits.

Rheology of Polytriazole/ZIF-8 Solutions and Dynamics of Mixed-Matrix Composite Films.

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Mixed matrix membranes are expected to reach high selectivity and permeability, combining polymers properties and metal-organic fillers, overcoming the permeability-selectivity trade-off in gas separation.[1] However, the problem with mixed matrix membranes is the poor adhesion between the filler and the polymer; this creates cavities around particles, providing a preferred route for the permeate, indicating an increasing permeability without reaching the expected selectivity. Meanwhile, excessively strong interactions increase the rigidity of the polymer around the filler affecting the permeability. Quantification of the polymer/filler adhesion is essential, but it is usually confirmed only after the membrane performance testing.[2] Solution rheology is a tool for identifying polymer-filler pairs with good interaction in this work. The investigated system was polytriazole with and without hydroxyl functionalization (PTA-OH and PTA) and ZIF-8. ZIF-8/PTA-OH forms stable gels even with small filler content, while analogous systems with PTA do not form gel.[3] Since a significant increase in viscosity was observed even with 3% filler, rheology is highly sensitive for identifying the best polymer/filler pairs. The investigations were supported by spectroscopic methods, electron microscopy, and dynamic mechanical analysis of the final membrane. While small ZIF contents lead to sensitive changes in the solution viscosity, only minor changes in the FTIR and NMR spectra were detected for the same system. The good adhesion between PTA-OH and ZIF-8 could be seen by SEM, confirming the formation of cavities in membranes prepared with PTA, while they were practically absent in membranes prepared with gelling PTA-OH/ZIF-8 systems. The separation performance of the optimized membranes is under investigation using different gases such as CO_2, CH_4, and N_2. To sum up, the methods described in this work can be extended as useful tools in the development of composites such as mixed-matrix membranes applied to a large variety of currently available metal–organic frameworks and polymers.

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In Silico Approach to Screen Nanostructured Materials for Mixed Matrix Membranes Adsorbers in Hemodialysis

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Hemodialysis (HD) is the life-supporting treatment for 2.6 million patients with end stage kidney disease (ESKD), affecting 9.7 million people worldwide. HD replaces kidney functionality, based on the exchange of uremic toxins (UT) through a semipermeable membrane with a buffered solution, called dialysate. The lack of access to affordable treatment resulted in 1.2 million deaths in 2017. The treatment is discontinuous, delivered 2-3 sessions per week, in hospital. The ultrapure water consumption (100 L per session) represents the bottleneck for a wearable artificial kidney (WAK). Therefore, dialysate regeneration would boost accessibility of hemodialysis, improving patients' life expectancy and lifestyle. Current technology for WAKs involves enzymatic or electrochemical conversion of urea, that is the most abundant and hard-to-remove compound, but issues of patient's safety were raised. A technology involving a capture mechanism of UTs is intrinsically safe. The fabrication of mixed matrix membranes, a composite formed by adsorbing particles onto a porous polymeric support, revealed to guarantee optimal fluid dynamic condition and low energy consumption [1-3]. Nonetheless, conventional adsorbers used so far showed to poorly capture urea and usually discovery and screening of new materials are strongly empirical. Our work aims to develop a computational screening tool for existing and hypothetical crystals, to assist the selection of new sorbents, scoring new candidates for the removal of urea from water. The pipeline is based on the in-silico characterization of the crystals calculating topological and physiochemical descriptors. Interaction energies between UTs and frameworks are used as a scoring function, in terms of binding strengths and selectivity toward UTs. A quantitative structure-property relationship is devised for describing UTs adsorption, for the first time. This in-house developed pipeline is a fast, versatile and open-source tool and it will be used in future to drive the design of a new generation of materials for hemodialysis.

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Ion-exchange membranes for electrodialysis, reverse electrodialysis and piezo dialysis

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Ion exchange membranes have been applied to electrodialysis (ED) and reverse electrodialysis (RED). ED and RED stacks consist of alternately stacked anion exchange membranes (AEMs) and cation exchange membranes (CEMs) with flow channels for solution supply. To increase desalination performance of ED systems and power output of RED systems, there have been some reports on fabrication of profiled IEMs (PF-IEMs) developed by several techniques, such as molding method and 3D printing [1, 2]. Here, we fabricated novel PF-IEMs with "ORIGAMI" structure that fabricate PF-IEMs just by bending one sheet of a flat IEM to form PF pattern on homogeneous IEMs with a nearly uniform membrane thickness. The PF-IEMs had low membrane resistance and high mechanical strength. The voltage-current curves indicated that the stack using PF-IEMs have 31% lower inner resistance and 145% higher power output than that using flat-IEMs. The stack using PF-IEMs also indicated 18% lower pressure drop at the low-concentration flow channel than that using flat-IEMs.

A charged-mosaic (CM) membrane is made up of parallel arrays of negatively-charged (N) domains and positively-charged (P) domains passing through the membrane. The CM membrane has high permselectivity for electrolytes, and has been applied to desalination in piezo dialysis [3]. Here, CM membranes with high electrolyte permselectivity were prepared by a fabric prepared from P and N charged fibers, and also by ion track graft polymerization. In the ion track graft polymerization, after irradiating commercial anion-exchange membranes (AEM) with 560-MeV ^{129}Xe ions from a cyclotron, graft polymerization of N charged monomer on the AEM was performed to fabricate cation-exchange nano-channels inside the anion-exchange matrix. The fabric structured CM membranes showed negative osmosis, and CM membranes prepared by ion track graft polymerization succeeded in desalination up to 1500ppm at pressure of 0.3MPa.

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Influence of membrane structure-dependent water transport on conductivity-permselectivity trade-off and salt/water selectivity in electrodialysis

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Water transport, including osmosis and electroosmosis, is an important phenomenon in electrodialysis (ED) but is generally ignored in many studies on the analysis of ion transport. Having a fundamental understanding of the influence of water transport on ion transport during ED is of help for the optimization of the design of ion exchange membranes (IEMs) and the development of novel ED-related processes. In this study we systematically investigated the effect of membrane structure-dependent water transport on ED performance and explored the potential of using porous IEMs in ED. A 1-D model, which is based on the extended Nernst–Planck equation and duly considers the membrane structure-dependent water flux, was developed to analyse mass (ion and water) transport in IEMs during ED. Results show that the electroosmotic flux increased with increasing the water volume fraction, which further leads to the increase in counterion flux and the decrease in co-ion flux. Osmotic water flux exhibited a similar trend to electroosmotic flux with changing water volume fraction. The existence of osmotic water transport further enhanced the change of counterion and co-ion fluxes, which leads to the increase in both membrane conductivity and permselectivity. This suggests that osmotic water transport can break the trade-off between conductivity and permselectivity of IEMs during ED. On the other hand, osmotic water transport leads to the decrease in specific salt flux (i.e., the ratio of salt flux over water flux) despite the increase in salt flux, suggesting the decrease in salt/water selectivity. The effect of osmotic water transport is more significant for porous IEMs due to higher osmotic water flux compared to dense IEMs. This work provides new insights into the use of porous IEMs in osmotic electrodialysis for potential applications.

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Flexible operation of Chlor-Alkali Electrolysis using switchable Oxygen Depolarized Cathodes

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The worldwide energy sector is facing the challenge to reduce carbon dioxide emissions, mitigate global warming, and revolutionize energy generation. With increasing proportion of renewable energy production, strong fluctuations in supplied energy come along. In our work, we aim to reduce the energy demand of chlor-alkali-electrolysis and adapt the process to flexible production conditions. A new type of oxygen depolarized cathodes (ODC) operates flexibly, while reducing the energy consumption of chlorine-alkali-electrolysis by 30 %. Bifunctional, so-called switchable ODCs (sODC) enable an energy-saving oxygen-consuming mode and an energy-intensive hydrogen-evolving mode. Thus, in times of large energy amounts, the oxygen mode is employed whereas in times of low energy quantities the hydrogen mode is applied. Experiments on lab-scale focused on the system's stability for the two cathode reactions and compared the cell potential of the sODC to a conventional ODC without ability for switching. The stable cell potential difference between hydrogen and oxygen mode of 1.23V for moderate current densities is in accordance with literature. A comparison of the oxygen mode of the switchable and the conventional ODC demonstrate the high potential of the sODC with a difference of 0.07V only. Long-term experiments with up to 1,000 switching cycles, including short-term oxygen and hydrogen modes at 6.7kA/m^2, interrupted by a nitrogen flush, aimed for mimicking a realistic application of more than 500 days with regular switching. The long-term experiments revealed a potential increase of up to 6.7% over the whole course of switching cycles, of which the cause is examined in more detail in our study. This high potential of the sODC is supported by intensive economic studies showing that the sODC in chlor-alkali electrolysis can reduce operational costs by 6.5% and accommodates the expected strong fluctuations in future renewable energy supply.

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Layer-by-layer modified electrospun interfacial layer of bipolar membranes for enhanced water dissociation

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Lactic acid, an important material in the food industry, is often produced by fermentation. However, during fermentation mostly lactate salts are formed instead of lactic acid. Electrodialysis with bipolar membranes has the potential to convert lactate salts into lactic acid without the introduction of other chemical reagents by utilizing the water dissociation at the interface of the anion and cation-exchange layer of the bipolar membrane (BPM). Tailoring this interface layer (IL) of the BPM is thus essential to promote high water dissociation rates. Electrospinning allows larger control over the BPM interface thickness, morphology and composition compared to conventional casting techniques. In this work electrospinning is used to tailor the interface of the BPM to increase the compatibility and the surface area between the two ion-exchange layers resulting in increased water dissociation rates. To allow water dissociation to occur via both acid and base catalyzed mechanisms, polymeric water dissociation catalysts with a range of pKa's are introduced in the electrospun IL by a layer-by-layer (LbL) modification approach with a polyethyleneimine (PEI)/polyacrylic acid (PAA) polyelectrolyte couple. The BPM interface layer was fabricated by electrospinning the cation and anion exchange nanofibers simultaneously, resulting in an entangled structure with a high active surface area. The IL was subsequently modified using the LbL coating with PEI-PAA. The LbL assembly was characterized using optical reflectometry and showed an increase in layer mass per adsorption step. Furthermore, the adsorbed amount per layer was strongly dependent on the coating conditions (ionic strength and pH). This shows that the amount of catalytic sites in the IL of the BPM can be controlled by the amount of coating layers and the coating conditions. This work demonstrates the potential of bipolar membranes with enhanced performance due to the incorporation of catalytic sites with different pKa's compared to conventional laminated bipolar membranes.

Monovalent Ion Selective Membranes for Desalination by mMCD

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The increasing world-wide demand for clean water [1], the rising fresh water scarcity [2] and saltwater intrusion into fresh water resources are the driving forces to find innovative solutions for desalination and water purification. In many cases partial desalination e.g. removal of monovalent ions is sufficient and complete desalination as by reverse osmosis is not always needed. In addition fully desalinated water often has to be re-mineralized before its use. Membrane capacitive deionisation (MCDI) is a cost effective and suitable technology for this purpose [3]. However, the selective removal of monovalent ions is not possible with the current state of MCDI. The aim of the joint project innovatION is the development of monovalent ion selective membranes to be used in monovalent MCDI (mMCDI), allowing the selective removal of monovalent ions such as sodium, chlorine and nitrate from water sources exceeding the thresholds for drinking water. Different approaches for the preparation of monovalent ion selective membranes like pore-filled membranes, membranes based on interpenetrating networks as well as modification of nanofiltration membranes are introduced. Basic properties such as conductivity, membrane resistance and permselectivity of these membranes will be presented and discussed in detail. Furthermore, results from mMCDI experiments conducted on a 3-cell lab-scale mMCDI are presented.

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Membrane strategy at AstraZeneca – Implementing membrane technology for a sustainable future

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AstraZeneca is committed to have zero carbon emissions from operations across the world by 2025 and ensure the entire value chain is carbon negative by 2030. Drug substance manufacturing has been identified as a major contributor to the release of greenhouse gases, hence our aim is to contribute to the ambition carbon zero by leading a sustainability program in which membrane technology plays a key role. In 2020, the work-up group in process engineering focused on introducing a membrane laboratory capability to evaluate opportunities to i) recycle waste streams from our processes ii) investigate organic solvent nanofiltration for product purification and ultimately develop lower environmental impact processes. Initial assessment has confirmed the potential of membrane technology as a key separation to achieve sustainability targets and drive the carbon footprint reduction. Hence, to develop membrane technology into our processes, we have outlined a membrane strategy to establish membrane separation as business as usual by 2025. Among the areas covered in our strategy, solvent recovery using membranes could have a significant impact on our manufacturing processes. For instance, the solvent used across the manufacturing of one of our portfolio medicines is estimated to be ~ 24,000 tonnes per annum for 100 tonnes per annum of active pharmaceutical ingredient. If a recycling opportunity is implemented for one of the manufacturing stages, there is the potential to save 1,300 tonnes per annum of solvent and decrease the carbon footprint by 5%. Furthermore, we have implemented an alternative purification step for an API part of our new modalities portfolio by using ceramic membranes at laboratory scale. By developing a ceramic ultrafiltration step we have been able to achieve the target API purity saving up to 444 L of solvent for Kg of API, which is 86% less solvent used when compared with the current process.

Nanofiltration of organic solvents through a PIM-1 membrane: a non-equilibrium molecular simulation study

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The depletion of energy resources and environmental constraints are forcing manufacturers to resort to more sustainable separation and purification processes. In this context, organic solvent nanofiltration (OSN) is receiving more and more attention as a low-energy process, an alternative to solvent-consuming purification techniques (liquid/liquid extraction, chromatography) and suitable for processing large volumes as well as heat-sensitive products. However, its development on an industrial scale still remains limited due to the lack of understanding of the mechanisms ruling molecular separations in non-aqueous media. The aim of this work was to rationalize the molecular mechanisms controlling transport of organic solvents through a polymer-with-intrinsic-porosity (PIM-1) thanks to non-equilibrium molecular dynamics (NEMD) simulations. A microscopic model of an OSN PIM-1 membrane [1] was brought into contact with common organic solvents such as methanol, ethanol, acetone and toluene (pure solvents and solvent mixtures) and an external pressure gradient was applied to force them to cross the membrane. NEMD simulations allowed us to determine the permeability and selectivity of the PIM-1 membrane and to understand the microscopic phenomena that control their separation performance towards the various organic solvents under consideration. The simulation results showed good agreement with the experimental data, which made it possible to validate the microscopic interpretations of the OSN process.

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Liquid Phase Peptide Synthesis via One-Pot Nanostar-Sieving (PEPSTAR)

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Peptides, an important class of polymers, mainly used as therapeutics, are generally synthesised using solid phase methods. While solid phase peptide synthesis provides for facile manipulation of intermediates retained in the solid support, this technology faces challenges for large scale synthesis, where its non-quantitative coupling and deprotection leads to error sequences. In contrast liquid phase peptide synthesis, offers high peptide purity and scalability but its development is hampered by inefficient intermediate separations and solubility issues. Liquid phase peptide synthesis via nanostar-sieving (PEPSTAR) is a platform which synthesises peptides in solution with facile intermediate separations. Amino acids (AA) are coupled iteratively onto a n-armed, star-shaped macromolecule, forming peptide-nanostar intermediates. After coupling, the unreacted AA is quenched and subsequently proceeded to N-terminal deprotection. The bulky intermediates are then 'sieved' out from the debris and quenched AA all together via organic solvent nanofiltration (OSN) thus omitting the post-coupling isolation step. This synthetic cycle is repeated until the desired peptide length is achieved. Standard Fmoc peptide chemistry is applied throughout the synthetic cycle. The use of nanostar greatly enhances the molecular sieving efficiency by the >3-fold mass difference between the nanostar and the unreacted building blocks. Most importantly, real-time reaction monitoring can be undertaken by LC-MS with high accuracy. OSN plays a pivotal role in synthesis efficiency. A polymer-based membrane made of crosslinked polybenzimidazole (PBI) was proven to be durable and to have a high separation factor which remained consistent throughout many synthesis cycles [1]. In this work we demonstrate the successful synthesis of Enkephalin-type model peptides (~5-10 mers) via nanostar-sieving technology. Nanostars of different molecular weight (2-10 KDa) and structure are evaluated. The products are of higher (or equal) purity than peptides produced by a solid phase vendor, while using less equivalents of AA during coupling thus bringing the cost of materials down.

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Crosslinked polyethersulfone membranes for organic solvent nanofiltration in polar aprotic and halogenated solvents

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Solvent-resistant nanofiltration (SRNF) is a promising pressure-driven technology for molecular separation in organic solutions that could potentially replace many established downstream processing technologies. Polymeric membranes are cheap and easy to fabricate and moduled but typically suffer from low chemical stability in organic solvents, particularly in polar aprotic and halogenated solvents. Therefore, much effort has been sought to develop polymeric OSN membranes that are stable in organic solvents. Here we present A facile method for manufacturing polyethersulfone (PES) membranes that are stable in halogenated and polar aprotic solvents. The organic solvent stability was achieved by dissolving a multidentate aromatic amine as a crosslinker in the membrane dope solution. Then, after casting the membrane by conventional non-solvent-induced phase inversion, the membranes underwent simple diazotization/dediazonization to effectively crosslink the PES matrix. The crosslinking mechanism and the membrane properties were investigated. The membranes showed high stability in all studied solvents, including polar aprotic and halogenated ones. Their properties were easily adjusted from ultra- to nanofiltration by changing the PES concentration in the dope solution. Specifically, membranes based on 21%, 24%, and 27% PES in the casting solution showed approximately 50%, 75%, and 90% rejection of a 1 kDa solute in different solvents, respectively. The performance was stable during one-week filtration time in dimethylformamide and chloroform, verifying the membranes' high solvent stability. Overall, this study prepared membranes that were stable in many types of organic solvents and performed comparably to contemporary organic solvent nanofiltration membranes while showing better accessibility and scalability.

Recycling of plastic waste: removal of additives using solvent resistant nano/ultra-filtration membranes

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The worldwide production and use of plastics has grown rapidly over the last decades. The problem of recycling still is a major challenge, as there are both technological and economic issues that restrain the progress in this field. Global introduction of waste utilization techniques to the polymer market is currently not fully developed, but has an enormous potential. In this study, crosslinked polyimide membranes were studied to recycle plastic waste by removing additives using solvent resistant nano/ultra-filtration from different types of dissolved plastic. Process performance was investigated under different operating parameters, such as feed temperature and concentration, viscosity, and pressures to find an optimal condition to separate the dissolved polymers from the solvent and the additives. Brominated flame retardants (BFRs) are widely used in polymer materials in e.g. electronics, particularly in polystyrene (PS), acrylic-butadiene-styrene plastics (ABS), and high impact polystyrene (HIPS). Three types of polystyrene with different molecular weights and 5,10,15,20-Tetrakis(3,5-di-tert-butylphenyl)porphyrin (TBPP) as a model compound for BFRs were dissolved in different solvents to check membrane performances. More than 99% of the polymer could thus be recycled by ultrafiltration, and pure solvent streams were obtained by using nanofiltration to recover the additives.

Recovery of bioactive compounds from food wastes by membrane processes

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The valorization of wastes and wastewaters from the agro-food industry is a challenge and, at the same time, a key factor in the development of new strategies to achieve a circular economy. Food industry generates a large amount of wastewaters and solid wastes that cause a significant environmental problem due to their high organic load and phytotoxicity. However, they are also rich in bioactive compounds, especially phenolic compounds, which have shown great antioxidant and anti-inflammatory capacity, being highly appreciated by the pharmaceutical, cosmetic and food industries. In this work, different alternatives based on membrane technology are presented for the recovery and purification of phenolic compounds from wastewater and solid residues from olive processing [1-2]. In the case of solid wastes, a previous extraction with water or ethanol/water mixtures has been performed before the purification of the extract with membrane processes [3].

The membrane processes of ultrafiltration (UF), nanofiltration (NF) and forward osmosis (FO) were considered both in aqueous and organic solvent environments. Different membrane materials and molecular weight cut-offs were tested. The experimental results highlighted that the interaction between phenolic compounds and membrane materials is of great importance to explain permeate flux and rejection in the three membrane processes. For instance, severe fouling was observed in the case of hydrophilic UF membranes due to this interaction. The combination of membrane processes selected to obtain a polyphenols enriched stream depended on the type of wastewater treated: fermentation brine from table olive production, olive oil washing wastewater or aqueous and hydroalcoholic extracts obtained from solid wastes. The results can be extrapolated to other agro-food sectors, such as wine and pickle industries.

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Granular Anaerobic submerged Membrane Bioreactor for energy recovery and domestic wastewater treatment

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Granular Anaerobic Membrane Bioreactor (G-AnMBR) is a hybrid anaerobic biotechnology combining anaerobic digestion by granular sludge and membrane ultrafiltration. G-AnMBR have a great potential to compete conventional aerobic treatment for domestic wastewater treatment (DWWT) at ambient temperature by providing a high-quality effluent with less energy consumption and energy recovery through biogas production. Validation and optimization of the process have still to be done since full-scale implementation for DWWT is not yet achieved [1]. This study first aims to highlight that membrane submerged directly into a granular anaerobic bioreactor improves the anaerobic treatment efficiencies [2]. A G-AnMBR and an UASB were operated in parallel during 120 days at 25°C with low-strength synthetic wastewater (HRT = 13 h, OLR = 0.8 kgCOD.m^{-3}.d^{-1}). Following the 12 months of acclimation and 4 months of operation it has been demonstrated that: • G-AnMBR overcomes the usual long start-up period and biomass washout compared to conventional UASB reactor. • G-AnMBR performed higher organics and suspended solids removal (93% COD, 98% TOC and 100% MLSS) in comparison to the UASB (79% COD, 86% TOC and 99% MLSS) due to the UF membrane separation that retains all particulate and colloidal matter and macromolecules. • Higher quantity of methane was produced in the G-AnMBR (0,28 L CH$_4$/kg COD removed) highlighted that the organic matter retained by the membrane are later converted into biogas thanks to a longer contact time. This work proves that G-AnMBR can achieve an excellent water quality with an organic removal rate as high as aerobic treatment for domestic WW treatment at ambient temperature while producing biogas and recovering energy. Then, it provides a better understanding of the impact of the submerged membrane within the anaerobic tank through the anaerobic granular sludge.

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Laser-Induced Graphene Composite Conductive Membranes for Degradation of Emerging Contaminants

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Emerging contaminants, such as Pharmaceutical and Personal Care Products (PPCPs) have shown their adverse effect at very low concentrations. Conventional wastewater treatment failed to remove these PPCPs, and also, the traditional low-pressure membranes are not effective. However, the electroconductive membranes have the potential to degrade these PPCPs through in-situ ROS generation. Laser-induced graphene (LIG) is a three-dimensional porous electrically conductive graphene material printed on the polymer substrates with CO¬2 laser in a single step.1,2 A single-step conversion of metal-doped PES membranes to LIG filters and further UF membrane has been demonstrated with excellent catalytic and anti-biofouling properties for emerging contaminants removal and reducing biofouling. The fabricated membranes were tested for different parameters, such as flux and rejection of the BSA along with the removal of the pollutants (antibiotics). These electrically conductive membranes also achieved 6-log removal of mix culture of bacteria with applied voltage along with the generation of OH radicals.3 The "catalytic mechanisms of these metal-doped LIG membranes will be explained, including the 3D texture effect of LIG. These membranes (single-step laser printed) have great potential in desalination and wastewater treatment applications.

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Optimization of an integrated solar powered hollow fiber module for membrane distillation in desalination

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Nowadays, desalination carbon footprint has become a crucial matter considering the continuous implementation of those systems. An interesting solution to this challenge is renewable energy utilization with membrane distillation. Recently, experimental and modelling studies of systems combining vacuum membrane distillation (VMD) and solar collectors have been evaluated. [1,2,3]. However, heat losses, space and solar limitations are some of the drawbacks of the studied systems. The integration of a non-conventional solar collector in a module containing a bundle of hollow fibers in a cylindrical geometry for VMD might result in an attractive solution. This hollow fiber solar collector membrane distillation (HF-SCMD) module has numerous potential advantages as compactness, high contact area, low heat loss at the permeate side and a low operating temperature. The choice of operating conditions and module design of this innovative module is a key aspect in order to implement and operate it in remote regions for small communities. This study aims optimizing the module design and the operating conditions to obtain the lowest specific energy consumption (SEC) of HF-SCMD, and thus the lowest need for photovoltaic panels for sea water desalination. In order to achieve this objective, a model was generated by using Matlab to calculate the permeate flux and radial and longitudinal temperature profiles of a HF-SCMD module. After an experimental validation, the model was coupled with an optimization process (Python code) in order to minimize the SEC by considering some design parameters (module length, diameter, bundle porosity) for a chosen hollow fiber, and operating conditions (feed inlet temperature, flow rate, permeate pressure). The optimization takes into account the entire process. This study allows to determine the appropriate geometry and operational mode of a HF-SCMD module for the targeted application. Experimental tests are in progress with a prototype based on a 2.1 m^2 module.

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Production and Performance Evaluation of Thin Film Nanocomposite (TFN) Reverse Osmosis (RO) Membranes Containing Boron Carbon Nitride Quantum Dots (BCNQDs)

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Desalination with reverse osmosis (RO) membranes produced by interfacial polymerization is an effective technology to obtain drinking and irrigational water. Thin-film nanocomposite (TFN) membranes promise significant advantages in terms of high flux, high selectivity, and resistance to chlorine. Producing defect-free, reproducible and homogeneous TFN RO membranes is directly related to the nanomaterial and production method. Carbon quantum dots (CQDs) are fluorescent, semiconducting nanocrystals with sizes between 2-20 nm, which can be coated with hydrophilic or hydrophobic groups, are easily synthesized, and have low-cost advantages. The fact that these materials are both cheap, biocompatible, and easily synthesized reveals that they are the most prone to commercialization when used in membrane production. Boron nitride carbon quantum dots (BCNQDs) produced by using nitrogen (N) and boron (B) increase the amount of hydroxyl with the amount of B, and the amine groups with the amount of N [1]. The water flux increases with the addition of these groups, which make the membrane surface more hydrophilic. Other hand, carboxylic groups from citric acid provide a more negative charge on the surface, improving salt removal. In this work, we investigated the effect of BNCQDs incorporation on polyamide TFN RO membrane production and performance. Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), roughness, and contact angle analyzes were performed for membrane surface characterization. To determine the filtration performance, the membranes were tested with 2000 ppm NaCl solution at 15 bar using a cross-flow cell.

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Membranes from biopolymers: opportunities and challenges

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The need for renewable resources and environmentally friendly materials has enhanced the research on biopolymers with improved properties. An overview of the development of biopolymeric membranes, their limitations and the requirements for obtaining innovative products and/or processes will be addressed.

Polysaccharide membranes have been produced using mainly alginate and chitosan since they are biodegradable and widely available. However, intensive research is being carried out on the production of microbial polymers as they represent an advantageous alternative to biopolymers extracted from animals or plants. The glycerol-rich by-product from biodiesel production was employed to produce negatively charged exopolysaccharides which were used to obtain dense membranes. These have shown a high hydrophilic character and good gas barrier properties being insoluble in most common solvents (ethanol, dichloromethane, acetone, toluene and ethyl acetate). Therefore, they have been applied in dehydration processes (bioethanol and biogas) [1]. Polyhydroxyalkanoates (PHAs), which are microbial polyesters and Cyrene™ a non-toxic solvent, were used recently to produce dense membranes that were successfully applied for the separation of an organic/organic azeotropic mixture by pervaporation [2]. The development of new cellulose derivatives is also expected to provide a more sustainable alternative to conventional synthetic polymers. Although plant cellulose is the most important source, various bacteria can produce cellulose as an alternative source. Recently, a dicarboxymethyl cellulose polymer (DCMC) was developed and patented [3]. This cellulose derivative has a wide pH working window due to its low pKa, making it suitable for the adsorption of several cationic species (dyes, proteins, micropollutants) [4]. The development of DCMC porous membranes will couple filtration and adsorption, thus cutting production costs and contributing to a greener process.

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Polyelectrolyte Complexes as Versatile Building Blocks for Advanced Functional Membranes

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Polyelectrolytes, charged polymers, tend to have a natural solubility in water. However, when oppositely charged polyelectrolytes are brought together they can interact to form an insoluble material, a polyelectrolyte complex. The properties of this formed material, such as the density of ionic crosslinks, an excess of charge and the degree of swelling can be controlled to a great extent by carefully choosing the used polyelectrolytes and the conditions (e.g. pH, Salinity) under which complexation takes place. In this talk, we highlight how this excellent control over the material properties make polyelectrolyte complexes very relevant as membrane materials for which the separation properties can be very precisely tuned. We do so by highlighting a number of recent breakthroughs that allow the processing of these versatile polyelectrolyte complexes into advanced functional membranes. The most established approach, layer-by-layer deposition of polyelectrolyte multilayers (PEMs) recently saw such a breakthrough with the development of asymmetric PEM coatings, allowing the formation of dense and active separation layers of just 4-5 nm in thickness [1]. Such asymmetric PEM based membranes show excellent performance in the removal of organic pollutants at high permeances, while the degree of ion retention can be precisely controlled. But polyelectrolyte multilayers can be more than just the separation layer, they can also form complete porous or dense membranes. Indeed, an Aqueous Phase Separation (APS) process where a switch in pH or salinity induces polyelectrolyte complexation, allows for the completely water based production of membranes ranging from microfiltration to (organic solvent) nanofiltration [2]. Moreover, hot-pressing of pre-formed polyelectrolyte complexes leads to the preparation of novel ion exchange membranes with very relevant ionic selectivities, including a difficult to obtain monovalent to monovalent selectivity of Na^+/K^+ [3]. Interestingly, dense polyelectrolyte membranes have very low gas permeabilities, offering their alternative use as fully recyclable oxygen barrier coatings.

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Fully green membranes prepared combining the bio-polymer PHBHV with the green solvent CyreneTM

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To fully respond to the requirements related to the reduction of waste generation and in accordance with the basic principles of the Green Chemistry, this work aimed to develop innovative fully sustainable membranes exploiting raw bio-based materials (polymer, solvent and additive) for targeted applications [1]. For the first time, the bio-polymer poly(hydroxybutyrate-co-hydroxyvalerate) (PHBHV) was combined with the green solvent dihydrolevoglucosenone (Cyrene™) for the preparation of membranes exhibiting different structures [2]. Bio-based additives, such as epoxidized broccoli oil (EBO), were also employed to tune membrane properties and morphology. The membranes were prepared by phase-inversion techniques evaluating the effect of different variables such as: polymer concentration, evaporation time and additives concentration. Asymmetric membranes with suitable mechanical properties were successfully obtained by introducing a pre-evaporation (EIPS) step before the coagulation bath. Besides the improvement of the mechanical properties, this EIPS step also strongly influenced the membrane morphology, including its cross-sectional microstructure and overall porosity. It was, thus, possible to produce more diversified membrane architectures, from dense to porous ones. The different morphologies obtained resulted in membranes permeable to water (up to 350 L/(m^2 h bar), with a pore size in the UF/MF range, and in asymmetric membranes displaying a dense top layer. These latter ones, thanks to the solvent resistance properties of PHBHV, were applied in pervaporation for the separation of a MeOH/MTBE organic mixture at its azeotropic point. The membranes resulted selective for MeOH as a consequence of their hydrophilic nature and of the small dimension of the alcohol molecule, reaching a selectivity of about 30. Porous and dense membranes showed also interesting stability performance when tested under specific degradation environments for long periods of time, suggesting possible applications for the filtration of aqueous solutions.

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Interfacial Polymerization kinetics: new insights on film formation using in-situ microscopy and particle-tracking

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Thin-film polyamide (PA) films fabricated via interfacial polymerization (IP) form the core of membrane-based water purification. Despite widespread commercial application, the exact details of reaction kinetics and film formation are not fully understood. Studying reaction kinetics is critical for controlling the process and designing better membranes for different applications. However, studies mostly rely on post-fabrication characterization due to the challenging nature of IP that make it difficult to monitor. Recently, in-situ techniques have evolved, enabling direct monitoring of IP in terms of heat and mass transfer and reaction dynamics. Here, we study reaction kinetics and film formation during IP via microscopy-based particle tracking. Fluorescent particles located in the aqueous and organic phase are observed using confocal microscopy during an IP reaction performed in a microfluidic device. Reaction parameters that affect the polymer formation rate and/or the system's susceptibility to instability are systematically varied. Particle trajectories are calculated using image analysis, as well as the mean squared displacement (MSD), which are then used to analyze the motion to determine whether it is non-Brownian and directed towards a specific direction. A directed flow implies the triggering of a hydrodynamic instability which drives a flow. There are various instability mechanisms that may occur during IP due to the rapid and exothermic nature of the reaction. The flow created during film formation may cause its deformation and ultimately contribute to the resultant crumpled film morphology. Therefore, studying the flow during IP with varied reaction parameters can link between synthesis conditions, reaction kinetics, and film morphology. Such measurements are important steps in the process of acquiring fundamental understanding of IP and mechanisms that affect the evolvement of different film morphologies.

Lignin as a renewable and biodegradable resource for polyelectrolyte multilayer based nanofiltration membranes

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Polyelectrolyte multilayer membranes (PEMMs) have gained a lot of attention in the drinking and wastewater treatment industries as nanofiltration membranes due to their ability to selectively remove ions, while being able retaining micropollutants and small solutes.[1] Currently, these membranes are made from fossil-based polyelectrolytes that result in plastic waste when the membranes are put out of use. With the inevitable depletion of fossil-bassed resources and increasing amount of plastic pollution in the environment, it is necessary to investigate renewable and biodegradable resources as sustainable alternatives. One promising alternative is lignin. Lignin is an abundant biopolymer originating from vascular plants that is currently being produced as a waste product from the pulping process in the paper industry. Recent studies have proven that negatively charged lignosulfonates originating from the sulfite pulping process in combination with cationically modified Kraft lignin, from the Kraft pulping process can be used in a layer-by-layer self assembly to form stable multilayers.[2] In this work, we present lignin as sustainable alternative to the conventional fossil-based polyelectrolytes for the fabrication of PEMMs. Here, the layer-by-layer self-assembly process was used to coat porous hollow fibre support membranes with lignin polyelectrolytes to obtain all-lignin PEMMs. The PEMMs showed loose nanofiltration properties with a pure water permeability of 23 L/(bar h m^2), a low divalent salt retention of 19% and a molecular weight cut-off (MWCO) of approximately 1 kDa. The lignin PEMMs proved to be stable over 24 h in highly saline solutions up to 5 M of NaCl and the lignin was shown to retain its biodegradable properties after forming a PEMM. The results thus clearly indicate that PEMMs with promising separation properties can be produced using renewable and biodegradable resources such as lignin.

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Functional additives for industrial applications by means of nanoprecipitation process

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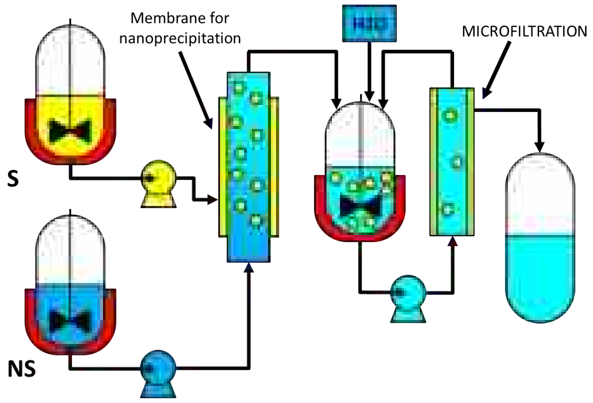
Functional additives are continuously developed with targeted functionalities to bring value in composite products for demanding applications and new processes are required to design particles with innovative characteristics. In this context, nanoprecipitation process can represent a powerful approach to transform functional materials in beads and capsules form. Nanoprecipitation process, patented by Fessi et al. in 1989 [1], is based on the variation of the quality of the solvent in which the main composition of a functional material is dissolved. Such variation can be achieved by altering the pH, salt concentration, solubility conditions, or the addition of a nonsolvent phase. In particular, the addition of non-solvent decreases solvent ability in dissolving the solute, promoting its precipitation.



This approach was applied by SAES Group Research Labs with the precious support of ITM team to design a new process on pre-industrial scale to prepare polymer beads for industrial applications. The process was tested on lab scale and then scaled up to pre-industrial level where new kind of functional beads are prepared for flexible packaging application. Specifically, an alcohol solution of an ethylene-vinyl alcohol copolymer (EVOH) was processed through a membrane assisted nanoprecipitation process to prepare beads able to provide high barrier performance to limit oxygen permeation in thin coating and frames configurations [2]. EVOH beads were characterized in terms of particles size, stability, composition and their integration in different prototypes will be presented as innovative approach to meet demanding requirements in industrial applications.

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The potentiality of Membrane Nanoprecipitation for nanoparticles production

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Membrane Nanoprecipitation is emerging as valuable method for the production of polymer nanoparticles, especially for hydrophobic materials. More recently, the powerful of the process was demonstrated for the production of uniform hydrogel nanoparticles based on polyvinyl alcohol (PVA) in a continuous process. The key role of phase composition and operating conditions that permits to the membrane to act as a high throughput contactor was identified. Moreover, the ability of the membrane to finely control the mixing process makes it suitable for the development of nanoparticles based on different materials. Here, we introduce the case of integration of membrane dispersion technology with reaction-based precipitation and non-solvent nanoprecipitation for the production and stabilization of iron nanoparticles. Magnetic nanoparticles were synthesized by coprecipitation of iron salts in alkaline medium by using reaction-based membrane nanoprecipitation. The influence of reactants concentration on the particle size distribution and structure was investigated to achieve a homogeneous supersaturation environment and control the nucleation and growth process. In order to overcome the aggregation tendency of magnetic nanoparticles due to the strong magnetic dipole-dipole attractions between particles, membrane nanoprecipitation was integrated with the reaction-based membrane nanoprecipitation for polymer coating of iron nanoparticles. A dissolved polymer (chitosan) was precipitated from a solution by supersaturation onto existing iron nanoparticles in suspension. A preliminary study of the effect of polymer concentration, solvent/non-solvent ratio and composition of the non-solvent phase was required to optimize the production of chitosan nanoparticles by membrane nanoprecipitation. The nanoprecipitation method for polymer coating of iron nanoparticles was compared with in-situ iron nanoparticles functionalization to identify the most suitable method for achieving continuous polymer coating. This study aims to broaden the application of membrane nanoprecipitataion as a competitive substitute method of conventional techniques used for nanoparticles production.

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Membrane-assisted nanoemulsification: From fundamentals to biomedical applications of hydrophobic deep eutectic solvents based nanoemulsions

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Hydrophobic deep eutectic solvents (DESs) are a recent class of designer green solvents, alternative to ionic liquids, offering low cost and negligible toxicity. To potentiate bio-based applications, wherein polar media is encountered, the low solubility of these hydrophobic solvents in water was tackled by dispersing them as nanoemulsions using energy-efficient membrane emulsification technique [1]. DES-in-water nanoemulsions were produced sustainably using Nuclepore track-etched isoporous polycarbonate membranes with varied pore sizes. Under optimum conditions, stable nanoemulsions of 39.0±0.4 nm and polydispersity index of 0.2 were formulated using an 80 nm pore size membrane. A detour from the principles of direct membrane emulsification was witnessed as the membrane pore size did not control, rather assisted in the formulation of DES-in-water nanoemulsions. To further explore the phenomenon of "membrane-assisted nanoemulsification", laser-drilled microengineered membranes of 9µm isopores were fabricated and tested. Interestingly, menthol/decanoic acid-based (58.7±0.4 nm) and menthol/thymol-based (81.2±0.9 nm) hydrophobic therapeutic DESs-in-water nanoemulsions were produced [1,2]. Unique observations such as self-assembly of hydrophobic DESs into nano-droplets, reduced interfacial tension (7.5 mN/m) between water and DES, and the hydrophobic DESs wetting behaviour to both hydrophilic and hydrophobic membrane surfaces were witnessed. Along with Tween 80 (surfactant), the DESs serving as co-surfactants to further stabilise the nanoemulsions were hypothesised and a dimensionless analysis was explored to describe the behaviour of fluids. From the application perspective, antimicrobial and in-vitro drug delivery applications were unravelled. In contrast to DESs or their individual components, the optimised DES-in-water nanoemulsions exhibited increased bioavailability and enhanced synergetic antimicrobial effects against Escherichia coli, Staphylococcus aureus and superior potency against Cutibacterium acnes (acne inducing bacteria). Furthermore, as hydrophobic DES demonstrated enhanced drug solubilisation capabilities, the drug-loaded DES was encapsulated into nanoemulsions and drug release studies were performed. Lastly, cytotoxicity studies of the nanoemulsions against various human cell lines validated its potential for biomedical applications.

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Micro- and nanoparticles based on natural lipid developed by membrane emulsification and nanoprecipitation

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Solid lipid particles (SLPs) are promising carrier systems for the controlled release and protection of bioactive compounds from external agents. SLPs are characterized by several advantages, including encapsulation of both hydrophilic and hydrophobic compounds, low toxicity and good biocompatibility. Among manufacturing methods of micro-nanostructured particles, membrane emulsification (ME) and nanoprecipitation (MN) are considered processes that enable the development of particles according to green chemistry principles [1, 2]. In this work, a hydrophilic SPG (Shirasu Porous Glass) tubular membrane (pore size of 1.1 micron) was used to explore for the first time the production of solid lipid micro- and nanoparticles based on cocoa butter by ME and MN, respectively. For MN, ethanol was used as solvent for cocoa butter, while water was used as non-solvent. The formation of nanoparticles in the shell or lumen side promoted by permeation of the organic solution into the aqueous phase and vice versa was studied to evaluate the influence of operational configurations on the particle size. Regarding ME, the melted lipid was mixed with ethyl acetate (dispersed phase), this strategy permitted the development of SLPs in water without temperature control. The different mechanisms of particle formation in the two membrane processes allowed the production of microparticles by ME and nanoparticles by MN with the same membrane pore size. Uniform SLPs smaller than 300 nm were produced using MN, which proved to be an energy-efficient method for continuous and scalable process. The ME enabled the production of highly concentrated and uniform microparticles with an average size of 3.6 times the membrane pore size. Comparing the two processes, MN showed great potential for large-scale production of nanoparticles. However, the process is characterized by lower mass productivity. To overcome this bottleneck, the present work addressed the recovery of SLPs by combining MN with the microfiltration process.

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Real-time monitoring of nanoemulsion formation: Recovery of valuable biomolecules from a microalgal biorefinery

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Current research efforts to valorise microalgal biomass have promisingly resulted in the recovery of biomolecules with myriad applications [1]. One efficient way to extract these valuable products is by using emulsions which are widely used for different biological [2] and biorefinery applications [3]. In this case study, a protein-enriched Spirulina microalgae extract, provided by our commercial partner, contained undesirable chlorophyll pigments that needed to be removed. Our strategy was to encapsulate the pigments in emulsions, formed using energy-efficient membrane emulsification technique. This allowed for the removal of undesirable chlorophylls from the valuable extract. The membrane emulsification process was optimised through the development of a platform that enables the real-time monitoring of droplet formation and coalescence. The platform consists of an ultrathin membrane (surface modified silicon wafers), containing precisely drilled micro or nanopores (using focused ion beam lithography), packaged within a fluidic system that supports pressure, flow, and real-time observation under high-resolution fluorescence microscope. Fluid pressures and flow dynamics within the system were controlled by computer-driven syringe pumps. This platform was used to study the effect of membrane morphology parameters (pore pitch and diameter) and operating parameters (pressure and flowrates) in real-time on the formation, growth and stability of the resulting emulsions. O/W emulsions were produced using optimized surfactant, Tween 80 (2% (w/v)) dissolved in aqueous microalgal extract as continuous phase and mineral oil as dispersed phase (several concentrations). The oil droplets selectively encapsulated the chlorophyll pigments thereby decolorizing the extract, as confirmed by UV spectroscopy. To remove pigments from the system, high-speed centrifugation was carried out to promote droplet coalescence, eventually leading to the separation of chlorophyll-enriched oil phase. Therefore, dynamic visualization with the developed platform promotes the understanding of role of parameters in droplet formation as it was critical to have control on droplet size to boost encapsulation.

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Challenges and proposed solutions for nanofiltration

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The transition to a sustainable future requires the implementation of more effective separation technologies with low carbon footprint. Membrane technology like nanofiltration can play an increasing role in the chemical, petrochemical and pharmaceutical industry, if suitable membranes and adapted processes will be available. The main requirements for a broader application are (solvent and temperature) stability, high selectivity, processability and scalability. Developing materials and membranes has been the main goal of our lab. We will discuss some of the recent achievements we have demonstrated, advantages, challenges, and perspectives of application. The innovation of our approaches consists of proposing new polymeric materials and building blocks, using manufacture processes that can be translated to continuous machines. We use mainly non-solvent induced phase separation for integrated asymmetric membranes, followed or not by interfacial polymerization. Recently demonstrated approaches integrated asymmetric include thermally crosslinked polytriazole membranes, converted/recovered polyetherketones and polyimides. Membranes with shape chiral selectivity are mainly prepared using different building blocks (e.g., macrocycles, fluorinated monomers) for interfacial polymerization. While their application for instance in oil fractionation and separations in organic solvents have been our main interest, membranes responsive to stimuli are being also investigated for specific smaller scale applications.

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Gold Nanoparticles-Dynamic Constitutional Frameworks Bioconjugates for Enzyme Activation and CO_2 capture

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It is challenging to perform macromolecular modifications on the surface of gold nanoparticles (AuNps) while maintaining their stability in aqueous environments. Interestingly, dynamic combinatorial frameworks (DCFs)-able to adapt and change under different stimuli [1]-interacts on the surface of AuNps to form stable cross linked AuNPs-DCFs. Carbonic anhydrase is known for its catalytic activity in the reversible hydration of carbon dioxide, which increases its interest for environmental and biomedical applications [2]. We apply this new matrix to the immobilization of bCA by exploring different strategies. The conformation of CA is preserved and the catalytic activity is significantly increased during binding to DCFs. CA assembled to AuNps and DCFs induced an additional increase in activity: the reactivity of dynamic connections with bCA active sites plus their ability to hydrolyze substrates by themselves. Cross linked gold-dynamic constitutional frameworks immobilizing bCA shows significant high temperature resistance. Our results are very promising for membrane applications in catalytic CO_2 hydration processes with high activation and stability.

Keywords: dynamic constitutional frameworks, gold nanoparticles, bCA activation, CO_2.

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On the order and orientation in liquid crystalline polymer membranes for gas separation

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Despite that polymeric membranes are often used for gas separations, most of the used polymers are not organized at a molecular level, which results in a polymer functionality trade-off between permeability and selectivity. Using the self-assembly of polymerizable liquid crystals (LC) to prepare membranes ensures control over the supramolecular organization and alignment of the building blocks at a molecular level providing an additional tool to break the standard trade-off relationship. Here, we demonstrate the importance of molecular order and orientation in free-standing LC polymer membranes for gas permeation [1]. Robust free-standing LC membranes were fabricated that have various, distinct morphologies (isotropic, nematic, and smectic C) that differ in type and degree of molecular order while using the same chemical composition. Single gas permeation data show that when going from a randomly ordered to the highly ordered smectic C morphology the permeability decreases with increasing molecular order while the ideal gas selectivity of He and CO_2 over N_2 increases tremendously (36-fold for He/N_2 and 21-fold for CO_2/N_2). The calculated diffusion coefficients showed a tenfold decrease when going from the randomly ordered membranes to the highly ordered smectic C membranes. It is proposed that with increasing molecular order the free volume elements in the membrane become smaller which hinders gasses with larger kinetic diameters (N_2) more than gasses with smaller kinetic diameters (He, CO_2), inducing selectivity. Apart from the supramolecular organization also the orientation direction of the LC molecules is decisive for the permeation and selectivity performance. Smectic C membranes with a homeotropic orientation show superior selectivity's over Smectic C membranes with a planar orientation. These results strongly highlight the importance of molecular order and orientation direction in polymer gas separation membranes as a tool to increase the permeation selectivity.

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Photodegradation of tetracycline using g-C_3N_4/Nb_2O_5/HPEI/PES nanocomposite membrane

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Tetracycline is an effective antibiotic that is extensively used for treating bacterial infections found in animals and humans due to its low and cost broad spectrum of activity [1]. As an emerging pollutant it is not regulated meaning there are no set limits for its presence in water. Moreover, complete removal of tetracycline is not achieved by the existing traditional wastewater treatments [2]. Hence, there is a great need to explore new technologies that can effectively remove this pharmaceutical pollutant in water systems. Therefore, this study is focused on the development of a photocatalytic membrane for the degradation of tetracycline in water system. Graphitic carbon nitride/niobium oxide (g-C_3N_4/Nb_2O_5) heterostructure within a hyperbranched polyethyleneimine (HPEI) template, was prepared via the in-situ hydrothermal method for the first time. This heterostructure was embedded on a polyethersulfone (PES) membrane using the phase inversion technique. Characterisation techniques such as scanning electron microscopy (SEM) and contact angle analyses were used to investigate the morphological features and hydrophilicity of the prepared membranes. Liquid chromatography-mass spectrometry (LC-MS) analysis was used to determine the performance, pathway, and mechanism of the tetracycline degradation. The nanocomposite membranes had large macrovoids and reduced pore size with improved hydrophilicity. Tetracycline was transformed into seven innocuous by-products, with 2,3-butadienoate and methoxyacetylene appearing at 83 m/z and 59 m/z, as the most stable fragments.

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PVDF/Graphene/catalyst intelligent composite membrane with multifunction for water resource and renewable energy application

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Due to not only rapid population growth but also rapid social development, the world is facing energy shortage and damage to environments such as deficiency of electricity and water pollution. The membrane is one of the solutions, which is a relatively ripening technology, but it still has some limits, such as fouling during long-term operation. Hence, in this study, a PVDF/Graphene/catalyst multifunctional composite membrane was fabricated, with a photocatalytic effect, piezoelectric effect, photo-thermal, and conductivity. By adding a traditional catalyst to a polymer, the membrane can carry out the photocatalytic effect and degrade pollutants in water. By adding graphene and using a wet phase conversion process, the crystal phase of PVDF can be induced to convert from α phase to β phase. Membranes will have a piezoelectric effect and convert effectively wave energy to electrical energy. At the same time, good piezoelectric performance can also improve photocatalytic efficiency. The membrane has an output voltage of 12 V (±6 V) for 2 Hz simulated impact and a dye degradation rate of 35% for 1500 rpm stirring. In addition, the stacked graphene and the polymer formed a pore structure with a photo-thermal effect and can be used as a solar evaporator to obtain freshwater. With the electrical energy stored by the piezoelectric effect, it can reach the continuous distillation process. Finally, the membrane in this study has good conductivity, which can observe the resistance variation when the membrane was fouling and identify the degree of fouling effectively. Thus, the composite membrane was used as a water power generation device, which can simultaneously obtain freshwater, generate electricity, degrade the pollutant and monitor the degree of fouling. This study proposes a new and effective solution for resource scarcity and renewable energy.

Gas separation performance of MMMs containing (PIM-1)-functionalized GO derivatives: unveiling the role of functionalization and nanoholes in gas diffusivity and physical aging

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A strategy to reduce the physical aging in PIM-1 membranes by incorporating novel functionalized graphene oxide (GO) fillers is reported. PIM-1 was covalently attached both to as-synthesized GO and to GO reacted with (3-aminopropyl)triethoxysilane (APTS-GO), leading to homogeneous dispersion of the nanofillers in the mixed matrix membranes (MMMs). It was found that the aging rate decreased with increasing content of (PIM-1)-functionalized GO, as evidenced by the smaller decrease in gas permeability over time. The best performance was achieved by a membrane containing 10 wt% of (PIM-1)-functionalized APTS-GO. This membrane maintained 85% of the initial CO_2 permeability after 150 days (drop of only 310 Barrer), which represents about nine-fold less CO_2 permeability drop as compared to pure PIM-1 [1]. However, the addition of GO-like nanofillers results in a reduction of gas diffusivity due to the non-porous nature of GO. Therefore, nanopores were created in the basal plane of gas-impermeable GO by chemical etching reactions, and the resulting holey flakes were further functionalized with PIM-1 moieties to aid their dispersion in PIM-1. It was found that nanopores barely promoted gas transport through the graphene-like nanofiller for fresh membranes (tested right after preparation). However, 150-days-aged hybrid PIM-1/holey GO membranes exhibitted higher CO_2 permeability and CO_2/CH_4 selectivity than the purely polymeric aged membrane; the addition of 1wt.% of (PIM-1)-functionalized holey GO nanofillers led to membranes that maintained up to 70% of their initial CO_2 permeability (only 53% was kept for pure PIM-1 after the same 150-days period [2]. The gas permeability of the nanofiller was rationalized with the aid of the Maxwell–Wagner–Sillars equation.

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Super hydrophilic membrane integrated with Polyaniline-Graphene oxide nano fillers for treatment of textile effluents

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Water pollution from the fashion industries containing dyes has become a major source of water pollution. These anthropogenic contaminated waters directly enter irrigation and drinking water systems, causing irreversible environmental damage to human health. Nanomembrane technology has attracted extensive attention to remove these toxic chemicals but new approaches are still required for improving removal efficiency and control the channel size. The work deals with the fabrication of a novel hybrid polyvinylidene fluoride (PVDF)-polyaniline (PANI) membrane with graphene oxide (GO). Incorporation of PANI-GO as a nanofiller has significantly improved antifouling properties and a solvent content of the fabricated membrane. Besides, pure water flux also increases from 112 to 454 mL m^{-2} h^{-1} indicating the hydrophilic nature of the nanocomposite membrane. Among various compositions, the nanocomposites membrane with 0.1%w/v GO demonstrated a maximum of 98% dye rejection at 0.1 MPa operating pressure. After multiple testing of the membrane, the flux recovery ratio reached about 94% and dyes rejection improved with the addition of PANI-GO. The removal efficiency of the composite membrane for Allura red is 98% and for methyl orange is 95%. Based on the above results the PVDF/PANI/GO membranes are recommended for practical use in wastewater treatment, particularly for anionic dyes removal from textile effluents.

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Clay composite membranes for salinity gradient batteries

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Sodium chloride gradient batteries are attractive urban energy storage solutions thanks to their ability to easily be scaled up and their use of abundant, safe, and cheap materials. However, large scale deployment of this technology is currently hampered by the lack of low-cost ion exchange membranes that have both excellent permselectivity and high ionic conductivity. This problem can be tackled by using composite membranes that combine a cheaper hydrocarbon matrix than the state-of-the-art perfluorosulfonated polymers with the attractive properties of inorganic fillers. In this work, microcomposite membranes made of sulfonated poly(ether ether ketone) (SPEEK) and 1 to 20 wt.% K30 montmorillonite (MMT) and aluminum pillared MMT (Al-pil MMT) are considered. The obtained membranes were heterogeneous microcomposite membranes due to the immiscibility of the non-functionalized clays in the SPEEK matrix. The addition of inorganic clays with 2D geometries enables the formation of percolating sodium diffusing pathways with reduced tortuosity. As a result, the conductivity of the membranes increases with an increasing clay loading fraction, reaching up to 1.4 times that of the pure SPEEK with 20 wt% K30 MMT. The permselectivity the SPEEK membrane also improves with the addition of set amounts of K30 MMT, while the Al-pil MMT composites suffer from a slightly reduced permselectivity (by no more than 3%) due to their higher water uptake. This is due to the larger hydrophilic interlayer spacing in the Al-pil MMT which creates more free volumes in the membrane. These results show that the addition of unmodified clays to a polymer-based cation exchange membranes is a promising method to lower the permselectivity-conductivity trade-off.

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CO_2 separation by imide/imine organic cages as fillers in mixed matrix membranes

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The development of new membranes with improved separation performance represents an ongoing challenge in the gas separation field. A way to boost the performance of neat membranes is to incorporate intrinsically porous materials (IPMs) into the polymeric matrix, combining synergically the scalability and processability of polymers with the high gas separation performance of the filler. Organic cages represent a type of IPMs formed by single molecules with three-dimensional cavities and a well-defined pore structure in the solid state. One of their main advantages is that they are soluble in organic solvents and therefore more easily processed via solution-casting to yield mixed matrix membranes (MMMs). In this work we describe a series of different polyimine cages and their corresponding MMMs in the solvent-processable poly(ether ether ketone) PEEK-WC.[1] Pure gas permeability measurements of the neat polymer and the MMMs are carried out to investigate the effect of the fillers on the gas transport parameters. The results showed that the use of imine based cages can tailor the gas transport properties of the pure polymer membrane, increasing either the permeability or the selectivity. In most cases, the increase in permeability is mainly due to the increase in diffusivity, while the enhanced permselectivity is mostly a result of an increase in the size-selectivity of the membrane, i.e., the cages favor the transport of the smaller gas molecules over that of the larger molecules. Further studies will focus on novel imide/imine-based organic cages, comparing pure gas permeability measurements and mixed gas permeation tests, in order to validate the potential of the most promising membranes for the separation of industrially relevant gas mixtures, such as CO_2/CH_4 and CO_2/N_2.

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Thin film nanocomposite membranes of super glassy PIM-1 and GO-based fillers for gas separation

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Loss of free volume over time (i.e. aging), which takes place at a much faster rate in thin films, is the main hurdle towards the commercial use of super glassy polymers for gas separation membranes. In this work we have investigated the use amine-functionalized reduced holey graphene oxide (rHGO) in PIM-1 thin films, and have obtained a nanocomposite membrane (1wt.% of rHGO-Tris (4-aminophenyl)amine) whose CO_2 permeance after 1 year of aging is very close to that of the fresh membrane (tested right after preparation) at ~ 800 GPU. Lower concentrations of filler (e.g. 0.1 wt%) also show a high CO_2 permeance (double that of purely PIM-1) at 846 ± 37 GPU after 1 year of aging, but they aged quite rapidly; CO_2 permeance values of the fresh thin film nanocomposite (TFN) membranes at filler loading of 0.1 wt.%. of rHGO- Tris(4-aminophenyl)amine and rHGO-N,N,N’,N’-Tetrakis(4-aminophenyl)-1,4-phenylenediamine were 3,351 ± 662 and 3,200 ± 842 GPU, respectively. The aging behaviour has been also investigated in thick membranes of several tens of micrometers (up to 2 years) for filler loadings of 0.1 wt. % and the gas separation performance has shown similar tendencies to that of thin films; leading to higher CO_2 permeability without sacrificing CO_2/CH_4 selectivity.

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Comparison of Solar Powered MCDI and RO Desalination for Mediterranean Agriculture

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In the North-African region, water scarcity and water salinization are being exacerbated by climate change conditions and agriculture is one of the economic sectors mostly affected [1, 2]. In order to provide an innovative and robust solution for groundwater desalination, an all-in-one "smart cube" for irrigation was developed within the EU-PRIMA research project SmaCuMed in the field of sustainable irrigation [3]. The smart cube is fully operated on solar energy and consists of a membrane capacitive deionization unit (MCDI), a low-pressure reverse osmosis plant (LPRO), photovoltaic panels (PV, 7.7 kW_p) with battery system (24 kWh) and a cloud-based IoT-system for monitoring and controlling, integrated with soil and water sensors (humidity, salinity, conductivity, pH) as well as a weather station. First results state the comparison of performance of the two different desalination technologies, while removing 33% of salt with the MCDI unit and 65% with the LPRO. The specific energy consumption (SEC) of the two technologies for the production of 3 m^3 of desalinated water every second day resulted in SEC_(MCDI) = 1.62 kWh/m^3 (running time: 150 min) and SEC_(LPRO) = 2.71 kWh/m^3 (running time: 95 min). In further trial tests, the desalination targets of 33% and 65% will be inverted for a direct comparison of the SEC values. Long term tests showed no decrease in permeability and performance for both desalination technologies after six months of operation, and SEC values indicated competitiveness with conventional LPRO usage. Furthermore, the focus on testing the novel MCDI technology was to find optimum operational parameters for its application in agricultural use. Hereby, most energy efficient desalination was achieved with constant current charge and discharge cycles, with additional zero voltage discharge in between. The pilot test with the smart cube showed the feasibility of MCDI use for irrigation of Argan trees in Moroccan semi-arid regions.

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The behavior of heterogeneous cation- and anion-exchange membranes under DC polarization

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Heterogeneous cation- (hCEM) and anion-exchange membranes (hAEM) play an essential role in electromembrane separation processes, such as electrodialysis or electrodeionization. Their properties (ionic selectivity, conductivity, etc.) intrinsically determine the quality of the separation process. Although the theories describing the ionic transport across both membrane types are mostly the same, the experimental observations show that they exhibit different behavior when under DC polarization. This is especially true when a high degree of desalination is reached. In this contribution, we will compare some of the properties and the behavior of heterogeneous membranes (Ralex membranes provided by Membrain a.s., CZ) with a focus on water splitting and convective structures occurring on the membranes. The experimental studies exploit novel techniques, such as computed tomography for the 3D reconstruction of the membranes [2] or PIV analysis to describe the developed convective flow fields around the membranes both qualitatively and quantitatively [1]. Our experimental results [3] show that the hAEM is more conductive than the hCEM. This feature is not only observed on the slope of the measured current-voltage curves but also from the analysis by computed tomography, which shows higher content of the active ion-exchange resin in hAEM. High-degree desalination is accompanied by forming a strong electroconvection superimposed by natural convection at hCEM. No water splitting was observed at this type of membrane. At hAEM, similar desalination conditions resulted in the onset of water splitting reaction manifested by profound pH changes of the electrolytes adjacent to the studied membrane. The proceeding water-splitting reaction significantly attenuates the electroconvection. The natural convection, however, still provides an intensive ionic transfer mechanism.

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Production of boric acid from seawater desalination brines using electrodialysis with bipolar membranes

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Boron is essential for many sectors of the European Union's (EU) industry, such as glass, ceramics, and Fertilizers. However, the EU's import dependence on borates is around 100%, hence it is considered a raw material with high supply risk. As the EU's natural reserves are deficient in boron and other critical raw materials, attention has turned to the oceans where almost all elements can be found. Considering a circular economy approach, the concept of brine mining emerges as a sustainable solution to supply the EU's industry since the concentration of elements in brines from seawater desalination plants (SWDP) is higher than in seawater. The Horizon 2020 Sea4Value project proposes a multi-mineral brine mining process to recover boron and other critical raw materials (such as magnesium and lithium, among others) from SWDP brines. The process comprises 3 stages: pre-treatment, concentration, and recovery. This work investigates the recovery of boron using electrodialysis with bipolar membranes (EDBM) in the final stage of the process. Initial tests feeding the EDBM set-up with synthetic borax solution (4 g/L of boron) at pH 12 resulted in a concentration factor of 2 for boric acid. Previous experiments with nanofiltration (pre-treatment) and boron selective resin columns (concentration) were performed using synthetic SWDP brine to determine the composition of the feed solution for the EDBM set-up. Since a solution of HCl 4% was used for columns elution, initial results showed interference of chloride ions in the production of boric acid. Therefore, a two-stage electrodialysis (ED) process was proposed after the columns elution and before the EDBM step. The first ED stage, at acid pH, was used to remove the chloride and other anions since below pH 9 boron was presented as neutral species ($B(OH)_3$). The second ED stage, at pH 12, was used for increasing the boron concentration.

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Nitrogen compounds removal from brackish water by electrodialysis in fixed electric potential and dynamic current density operations

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Electrodialysis (ED) is a mature technology for water desalination and has been applied for nitrogen compounds removal from brackish water especially in fixed electric potential [1]. A recent study [2] used dynamic current density (DCD) in ED for ammonium recovery resulting in reduced energy consumption. To verify the potential of DCD, an ED batch system was operated in DCD and fixed electric potential to compare the removal of ammonium, nitrite and nitrate (100 mg/L) from brackish water. During DCD experiments, the current density was dynamically adjusted in relation to a percentage (40, 60, 80, 100, and 120 %) of the limiting current density (LCD) according to the decreasing ion concentration in the diluate. During the fixed mode, the electric potential was set 5 - 25 V [3]. The effect of the current density on the removal factor and specific energy consumption (SEC) was assessed by considering the current efficiency (CE) and the water quality regarding the nitrogen compounds. The results showed that an increase of electric potential at fixed or DCD operation increase the removal and molar flux of nitrogen ions. During fixed electric potential at 15 V and DCD at 80 % of LCD, the removal and molar flux are almost similar, reaching a removal of >90 % for all compounds and compliance with the guideline limits for ammonium and nitrate concentration in drinking water. It was demonstrated that the SEC of the process increased at higher fixed electric potential or DCD while no significant (<5 %) difference in removal and molar flux were observed. Overall, the CE during DCD was higher than during fixed electric potential operation. In total, ED proved to be a suitable treatment technique to produce drinking water due to the selective removal of the studied ions both in fixed electrical potential and DCD operation.

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Evaluation of power generation performance of reverse electrodialysis using ion exchange membranes with different properties

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The energy generated by mixing of two salt solutions with different concentrations is called salinity gradient energy (SGE). SGE can be converted into electricity using an ion exchange membrane (IEM). This technology is called reverse electrodialysis (RED) power generation. There are various types of IEM used for RED stack, including standard, monovalent ion selective, and low-resistance membranes. However, the effect of IEM properties on RED power generation has not fully understood. In this study, we evaluated RED performance using 5 kinds of IEMs with different membrane properties to investigate the effect of different membrane properties on RED power generation. In addition, the power output of a large RED stack using these IEM was predicted. Neosepta® CSE/ASE, Neosepta® CMX/AMX, Neosepta® CIMS/ACS-8T, C-2/A-2 (Astom Corp., Japan), and Fumasep® FKS-20/FAS-20 (FUMATECH BWT) were used as cation exchange membrane/anion exchange membrane pairs. The number of cells was 40 pairs, the membrane area was 88 cm^2 (total membrane area 7040 cm^2), and the intermembrane distance was 200 µm with spacers. In the case of using real seawater and surface water from a water purification plant, C-2/A-2 and FKS-20/FAS-20, which have lower membrane surface resistances, showed larger power density than standard membranes (CSE/ASE and CMX/AMX). On the other hand, CIMS/ACS-8T, a monovalent ion selective membrane, showed the identical power density as the standard membranes. These results indicate that membrane resistance is more important than monovalent ion selectivity for RED performance. Compared to the results obtained with a large RED stack (total membrane area 179.4 m^2) using CIMS/ACS-8T [1], the large RED stacks using C-2/A-2 and FKS-20/FAS-20 are expected to show 250 W and 231 W of power output, respectively.

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Pervaporation vs vacuum distillation in beer dealcoholization

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In addition to its aroma and flavour, beer is also a wealth of organic and inorganic compounds that possess properties modulating the work of the body. Some come from malt, some from hop extract, and some are produced during fermentation. One method of obtaining alcohol-free beer is to remove ethanol from the finished product. It is important not to lose or transform components responsible for beer's taste, aroma and colour together with ethanol. Still, it is also crucial not to lose substances of health-promoting characters, such as, e.g. phenolic acids and flavonoids. In this study, we compared the quality of products obtained after ethanol removal by vacuum distillation and pervaporation. The membranes used in pervaporation were commercial and additionally impregnated with compounds of natural origin in the presence of supercritical carbon dioxide. The colour, concentration of ethanol, selected phenolic acids, maltol, and antioxidant activity was determined in the liquid exhausted after the distillation process and in the retentates after the pervaporation process. The quality of retentates was satisfactory and characterized by a better aroma and flavour than that of exhausted liquids after vacuum distillation. Significant losses were noted (because of transport to permeate) only in the case of 4-vinyl guaiacol, a derivative of ferulic acid and one of substances responsible for beer flavour and aroma. It was indicated that the impregnation of pervaporation membranes with some compounds improves the quality of non-alcoholic beer.

Reactive pervaporation for valorisation of glycerol as glycerol carbonate

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Glycerol carbonate (GC) production plays an indispensable role in the valorisation of glycerol, which is a by-product of the production of biodiesels [1, 2]. The transesterification reaction of glycerol with dimethyl carbonate (DMC) produces glycerol carbonate and methanol (MeOH) as by-product. The purification of GC requires significant energy and capital cost [3] since DMC and MeOH can form an azeotropic mixture at different concentrations under different pressures because excess DMC is generally added in this reaction for higher glycerol conversion. In addition, this transesterification reaction is a reversible reaction limited by the equilibrium and it is typically performed at high temperature (100 °C). In order to further reduce energy demands and achieve sustainable development, reactive pervaporation at low temperature (60 °C) is proposed in this work as a better choice compared to traditional distillation [4]. Reactive pervaporation is a hybrid process combining reaction and separation in one single setup. While the reaction is taking place, MeOH is separated by a MeOH-selective membrane, shifting the reaction equilibrium to a higher glycerol carbonate yield. Results show that at 60 °C, not only a higher conversion rate of glycerol can be obtained by reactive pervaporation, but also the amount of DMC used can be reduced, thus reducing significant energy and capital cost in the separation and purification stage. Thus, reactive pervaporation is presented as a potential technology that allows advancing towards a greener production of glycerol carbonate.

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Modelling thermodynamic and kinetic coupling in multicomponent organophilic pervaporation: Extending binary models for multicomponent systems

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The increased environmental hazards associated with the use of fossil fuels have encouraged attention towards biofuels as a supplementary energy source. However, the biofuel industry is still plagued by the challenges of cost-effective and sustainable recovery of these fuels from complex fermentation broths. Organophilic pervaporation has the potential to emerge as a sustainable, cost effective and process integrated downstream recovery process for many fermentation and industrial applications. However, membrane transport in pervaporation is a function of both operating conditions as well as feed composition, which makes understanding sorption and permeation through the membrane tricky due to the presence of thermodynamic and kinetic interactions or "coupling" between the various components and the membrane. In our study, we investigated the intercomponent coupling encountered in pervaporation for preferential recovery of bio-butanol from multicomponent Acetone-Butanol-Ethanol (ABE) model fermentation mixtures. For this binary semi-empirical model like NRTL model and solution diffusion model were modified and extended to multicomponent systems. The phenomenological parameters were estimated by a numerical estimation scheme and interpreted in terms of temperature and concentration-dependent plasticization or swelling of the membrane polymer matrix by the permeating components. On the other hand, Response Surface Methodology was also used to estimate sorption and permeation over an experimental range relevant to fermentation mixtures regression coefficients and interaction parameters determined from the statistical model. Coefficients of determination of fit and ANOVA were then used to evaluate the models. Finally, it was found coupling was most favorable at lower butanol to second organic component molar ratio and experienced a minima w.r.t temperature. Interaction parameters involving acetone were also more significant than those involving ethanol. In conclusion, it was observed that for optimized extraction of bio-butanol the effect of solvent ratios and temperatures should be carefully modeled before any large-scale endeavor.

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Application of pervaporation for the removal of water from the postreaction mixture

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Pervaporation is a membrane separation method used nowadays to the dehydration, removal of volatile organic solvents from water, separation of organic-organic liquid solvent mixtures and desalination. Hydrophilic pervaporation can be used as the method for water removal from reaction mixtures shifting the reaction equilibrium towards the main product. 2,2-Dimethyl-1,3-dioxolane-4-methanol (DDM) is synthesized in the reaction of glycerol with acetone in the presence of highly selective catalysts. The occurrence of water in the postreaction mixture limits the yield of the synthesis. The aim of the research was to evaluate the efficiency of polymeric hydrophilic membranes in the removal of water from the mixtures containing 15 wt% of DDM, 3-15wt% of water, and acetone glycerol in the mass ratio 10:1. Additional experiments were performed using the acetone to glycerol ratios in the range from 8:1 to 1:1. The temperature of experiments was kept constant at 40oC. The commercial hydrophilic membranes provided by DeltaMem (PVA based polymeric membranes) were used. Results showed that membranes were selective to water with traces of acetone (up to 3 wt%) found in permeate. Moreover, there was no DDM nor glycerin in permeate. The best separation results were obtained using PervapTM 4100 HF membrane (separation coefficient over 500). However, the permeate flux through this membrane was lower than throught PervapTM 4100 one (79 g/(m2h1) vs. 106 g/(m2h1) for in contact with mixture containing 10 wt% of water). The change of the acetone:glycerol ratio from 10:1 to 1:1 resulted in the diminution of the water flux from 79 g/(m2h1) to 40 g/(m2h1). The results proved the applicability of the pervaporation process for the dehydration of the postreaction mixture. Further plans include the use of ceramic membrane instead of polymeric ones, experiments with other substrates (ethylene glycol and propylene glycol) and modeling of the dehydration process.

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Development and characterization of porous calcium copper titanate membranes for water treatment

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Pharmaceuticals compounds are categorized as emerging and persistent organic pollutants (POP) released into aquatic ecosystems. In fact, some compounds are not efficiently removed with the conventional methods. Therefore, advanced oxidation processes (AOP) is capable of effective degradation of POPs. Electrocatalytic membrane reactor is an alternative approach due to high efficiency, low energy consumption and non-selectively attack by losing electrons at anode or by high oxidizing of reactive radical substance OH. In addition, SO_4 radicals have a strong oxidation potential and show long life-time compared to OH, therefore research interest has grown in the generation of SO_4 by adding peroxymonosulfate as active agent. The performance of dense anodic electrode membrane raises major limitations of internal mass transfer which may be resolved by using porous electrode membrane to increase the surface area of electro active sites. In this study, we are interested in the perovskite oxides due to their unique structure properties, high chemical stability and excellent optical characteristics. A porous membrane of calcium copper titanate ($CaCu_3Ti_4O_{12}$(CCTO)) was synthesized with ball milling and used in a continuous system for degradation of paracetamol. In order to optimize the properties of membrane, pellets with different percentages (20-30 and 40%) of polymethyl methacrylate (PMMA) used as pore-forming agent were prepared and characterized. X-ray diffraction, scanning electron microscopy and Raman spectroscopy showed the presence of grain and grain boundary structure with two different phases CCTO and CuO. Electrochemical activity of the membrane was investigated with cyclic voltammetry measurement and impedance spectroscopy. The CCTO with 30 wt% PMMA showed the highest oxidation-reduction peak and has the best conductivity. CCTO nanocomposites were used as anode in an electrochemical cell (210 mL) with peroxymonosulfate as agent active (0.5 mM) to treat 10 ppm of paracetamol in aqueous solution. Paracetamol was fully degraded after 90 minutes using CCTO-30 with a kinetic higher than other pellets. The stability and the species responsible for degradation were investigated. The acute toxicity (Microtox) of potential by-products following the degradation was also investigated. Finally, CCTO-30 has been used for the electrocatalytic membrane reactors investigations.

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Direct hollow fibre nanofiltration for greywater reclamation: Applicability and performance of polyelectrolyte multilayer (PEM) nanofiltration membranes for wastewater reclamation and micropollutant removal at a full-scale treatment plant

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Over the past decades, global population growth, socio-economic development, and climate change, among others, have severely increased the pressure on global freshwater resources. Due to this increase, it is projected that one-third to half of the world's population will face water scarcity by 2050 [1]. One viable method to increase supply to meet local water demands is the reclamation of wastewater. However, the occurrence of hardly biodegradable emerging contaminants, such as micropollutants, require the addition of advanced post-treatment technologies to utilise the reclaimed wastewater fully. Recently, a new wastewater reclamation system opened in Helsingborg, Sweden, with the capacity to treat wastewater of 2300 p.e. in the Oceanhammen district. This project was recently awarded with "Wastewater Project of the Year" by Global Water intelligence. The reclamation system uses source-separated sanitation to treat greywater extensively, allowing 80% of the produced water to be reclaimed. First, greywater is treated using conventional activated sludge processes. Following this, direct hollow fibre nanofiltration is implemented to remove residual contaminants. These membranes offer a more sustainable process compared to conventional spiral wound membranes. Our current research investigates the biological removal and retention of 22 micropollutants (consisting of pharmaceuticals, personal care, and household products). Biodegradation of the selected micropollutants ranged from near complete removal of parabens (>88 %) to limited removal of hard to biodegrade contaminants, such as diclofenac (22%). A broad group of micropollutants were efficiently retained. The highest retention was observed for galaxolide (89%), while the lowest retention was observed for metformin (~30%). Complete process removal ranged from 91% for galaxolide to ~25% for metformin, indicating its viability as an advanced recovery system. Concentrate treatment by recirculation is currently being investigated to further enhance the sustainability of the treatment plant by sending the stream to the aerobic biological process to improve micropollutant removal.

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Chemical, physical and biological processes in highly efficient wastewater treatment by living electro-encapsulated self-forming membrane

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This contribution reports on a new concept of aerobic membrane bioreactors for highly efficient wastewater treatment based on a novel living electro and encapsulated self-forming dynamic biomembrane (e-ESFDM). The biological filtering membrane is intercalated between two woven polyester fabrics, as supports, that assist the formation and protect the biomembrane. The innovative architecture of the e-ESFDM in combination with electrocoagulation processes resulted in an efficient and cost-effective wastewater treatment and an unprecedented control of the membrane fouling The performance of the novel e-ESFDMBR was compared to a yet highly efficient ESFDMBR, where the electric field was not present. The ESFD-based reactors both showed comparable results in the removal of organic matter, in terms of COD and DOC. On the other hand, e-ESFDMBR exceeded the performance of the ESFDMBR in the reduction of nitrogen- and phosphorous-containing pollutants, responsible for eutrophication processes in the environment, and recalcitrant molecules, such as humic-like substances. In addition, an extremely low fouling rate was observed for the e-ESFDM bioreactor. Insights on the chemo-physical and biological processes involved in the novel MBR were provided by investigations on the fate of wastewater nutrients. The microbiological diversity found in reactor mixed liquor, ESFDM layer and treated wastewater was investigated isolating and identifying the microbial species as well as by Next-generation sequencing (NGS).

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Inorganic photocatalytic membrane reactors for wastewater purification

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The rapid growth of the world population and industries determined the production of a large amount of wastewater and solid waste with severe environmental pollution. The availability of safe and sufficient water is inextricably linked to the wastewater production. Textile wastewater and pharmaceutical residue wastewater are difficult to degrade due to the presence of highly stable contained organic compounds [1]. Different physical, chemical, and biological treatment technologies have been developed that present various advantages but at the same time cannot completely decompose organic pollutants. In the last decade, advanced oxidation processes (AOPs) have been growing due to their ability to degrade the organic pollutants in aqueous systems. AOPs can generate radicals that can completely convert refractory organic pollutants into harmless species (e.g. carbon dioxide and water) [2]. Despite these advantages, the large-scale application AOPs is still limited because of: (1) fast-backward reaction, and (2) the difficulty of using visible light (limiting the efficiency in using the solar energy). The combination of the membrane separation with the photocatalysis process represents an efficient, low cost and eco-friendly technology with a great potential in wastewater treatment. In this work, inorganic photocatalytic membranes have been prepared and characterized. Their morphology has been observed by SEM analyses. The photocatalytic activity of the prepared membranes has been evaluated by monitoring the photodegradation of some chemical species used as recalcitrant model pollutants. The experimental results demonstrated as photocatalytic membranes exhibit an excellent potential for the degradation of organic pollutants.

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Engineering Superior Multi-Functionalized Holey Graphene toward Ultra-Permeable Membrane for Emulsified Oil/water Separation

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The direct discharging of industrial oily wastewater into the environment with inadequate treatment is causing pollution issues to aqueous system. Membrane filtration process is considered one of the most effective solutions for treatment of oily wastewater, due to its low cost, energy efficiency and sustainability. In this work, we report the fabrication of novel hydrophilic multi-functionalized holey graphene (HG) membranes for oil-in-water emulsion separation using a facile vacuum filtration approach. Different loadings of the coating were utilized to optimize the membrane fabrication process. The fabricated membranes were thoroughly analyzed, and the performance of membranes were assessed in terms of flux and oil removal %. The modified HG membrane has high water flux with more than 8-fold, compared to graphene membrane. While the efficiency of oil-in-water emulsion separation was enhanced from 74 to 97% for control and modified membrane, respectively. This eco-friendly, facile, and versatile coating-based HG membrane may open a golden opportunity to be applied for the efficient treatment of industrial oily wastewater.

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Incorporation of nano-powdered activated carbon into porous polyethersulfone membranes via reverse filtration for preparation of adsorber cum filtration membranes

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Water contaminations by inorganic and organic micropollutants (MPs) remains a serious concern worldwide. Adsorption and membrane separation are widely applicable and comparatively cost-effective; nevertheless, they remain with limitations. This study aims to prepare porous adsorber membranes where the adsorption capacity and filter size selectivity will be combined. Different commercial ultra- and microfiltration membranes and two different nano powdered activated carbon (NPAC) materials are used. Reverse filtration method is employed to load the NPAC in the bottom/support layer of the membranes, optionally followed by additional fixation in a polymer coating on the pore walls. When reverse filtration was performed at a concentration of 10 mg/L NPAC and a total volume of 800 mL was filtered through 10.2 cm2 membrane, NPAC was successfully immobilized within the matrix and on pore walls of the membranes (Fig. 1). 4-Nitrophenol (PNP) was used as a model pollutant due to its ease of analysis and because of abundant reference data. Capacity of the NPAC towards the removal of PNP was investigated using static adsorption isotherm experiments in order to obtain reference data. Based on the preliminary investigation, optimum adsorption capacity (35.08 mg/g) of the adsorbent was attained at a PNP concentration of 10 mg/L, NPAC dose of 10 mg, and volume of 80 mL (pH of 6.58). PNP solutions at different concentrations (1 - 10 mg/L) were then filtered through the UF membranes at different trans-membrane pressures in order to evaluate the adsorption of the PNP onto the modified membranes under flow-through condition in terms of binding capacity and solute break-through. Functionalization of NPAC is also considered to tune the simultaneous removal of different MPs and colloidal particles. Adsorber membranes with a high rejection cum adsorption as well as good reusability performance, without significantly influencing the flux and anti-fouling performance of the original membrane, are anticipated.

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Towards Totally-Green Membrane Preparation Using Polylactic Acid (PLA) as a Biopolymer and Dimethyl isosorbide (DMI) as a Green Solvent

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In the last decade, the priority given to the valorisation of natural resources and the mitigation of climate change lies in reducing carbon dioxide (CO_2) emissions. This can be achieved through the transition of fossil chemicals to carbon-neutral alternatives according to the circular economy strategy (reuse, recycling, and waste reduction). In this scenario, membrane preparation strategies based on the use of green solvents and bio-polymers derived by bio-based platform chemicals are developing rapidly [1,2]. From these premises, the aim of the present study was to prepare, for the first time, entirely green porous membranes by combining the use of polylactic acid (PLA) as a biopolymer and dimethyl isosorbide (DMI) as a sugar-based green solvent. The main goal was to address the challenge of developing sustainable membranes from biomass. The membranes were produced via nonsolvent and vapor-induced phase separation (VIPS-NIPS) at different exposure times to humidity (55 RH%) such as 0, 2.5 and 5 minutes. The thermodynamic aspects of the interactions between polymer and solvent and non-solvent were determined in terms of solubility parameters, polymer-solvent distance, and relative energy difference (RED). The influence of the viscosity, polymer concentrations, and the presence of pore formers was properly investigated. The so-obtained membranes were characterized in terms of morphology, pore size, porosity, thickness, contact angle, and ATFR-IR. Filtration tests were also carried out in terms of pure water permeability (PWP) and the stability of the membranes was determined. The data collected confirmed that the membranes are characterized by finger-like structures with a wide range of applications from microfiltration (MF) to ultrafiltration (UF) processes. The performance was also evaluated in terms of rejection and permeability recovery ratio (PRR) using methylene blue (MB) in water solution.

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Polyolefin membranes fabricated with bio-based solvents: From plastic waste to value-added materials

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The utilization of non-renewable solvents and plastic pollution are two sustainability challenges that can be addressed from the membrane field. In recent years, alternative raw materials such as bio-based solvents and plastic waste have been proposed for membrane fabrication. In this work, two bio-based and renewable solvents are employed to dissolve polypropylene (PP) and low-density polyethylene (LDPE). The thermal properties and phase separation behavior of solutions prepared with LDPE and PP were studied, from which phase diagrams were obtained. Liquid-liquid phase separation and spherulitic morphology were observed for the studied systems. Polypropylene membranes were obtained by thermally induced phase separation (TIPS) process employing commercial polymer and analogous plastic waste. The influence of the quenching media during TIPS and polymer content was evaluated in terms of morphology, mechanical and thermal properties, and water contact angle on the resulting membranes. Their underoil contact angle (above 150°) allowed them to be used for water-in-toluene emulsions separation, where around 95% water rejection and an average of 99.97% toluene purity were obtained.

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New routes for 3D printing of membranes with controlled properties

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Additive manufacturing or its more conventional name 3D Printing is a relatively new but increasingly more popular method for generating complex structures [1]. In membrane manufacturing, there is a lack of methods to generate structures more complex than flat sheets or hollow fibers. Therefore, 3D printing has been seen as a viable new method for creating new membranes with complex topology and shape.[2,3] However, 3D printing for membrane fabrications has been limited by either the resolution or the processing speed. The current study presents a novel integration of a phase separation technique within a conventional 3D-printer to overcome these challenges. Membranes with controllable characteristics in the range of microfiltration and ultrafiltration were prepared using different synthesis temperatures, irradiation times and resin to solvent concentrations. The suitability of the method was validated by synthesizing the membranes with pore size and porosity ranging from <108 nm to 666 nm and 61-80%, respectively. The rejection potential of the membranes was tested with solutions of 600 nm nano-clay, 100-50 nm magnetite and 5 nm hemoglobin and rejections of >99, >99 and 34.1%±1.1%, respectively were observed. The permeability of the membranes was dependent upon the membrane characteristics and varied from 10 L/(m^2 h bar) to 444 L/(m^2 h bar). The method does not only allow controlling the microscale features and physical shape of the membranes but also uses less harsh chemicals. Therefore, this process can make membrane fabrication greener than conventional phase inversion techniques. Furthermore, this technology opens up application of a completely new range of polymers for membrane manufacturing that is not possible today.

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New sustainable routes for gas separation membranes: an experimental and modeling assessment of polyhydroxyalkanoates.

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The gas separation performance of biopolymers is still scarcely characterized, because of their poor thermomechanical properties and high crystallinity which is associated to low permeability. In this work we assessed the separation performance of copolymers beloning to the polyhydroxyalkanoates family. Such materials have good mechanical properties, combined with a renewable origin, biodegradability and biocompatibility. Furthermore, we evaluated the use of alternative green solvents for the fabrication of the bio-membranes, finding that dimethyl carbonate allows the production of polymer films with transport properties similar to those obtained with the more toxic chloroform. Membranes based on Poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) show a size-sieving gas separation behaviour, and strong energetic interactions with CO_2 that induce a marked solubility-driven CO_2/N_2 and CO_2/CH_4 selectivity. The experimental campaign was augmented with molecular simulations, that served to expand the range of investigation to ten different copolymers based on poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) and poly(3-hydroxybutyrate-co-4-hydroxybutyrate) (PHBB) with composition of the co-monomers ranging from 0 to 100%. Molecular models of the amorphous part of each copolymer were simulated using Molecular Dynamics (MD) and the solubility and diffusivity of different gases calculated using Widom insertions and mean square displacement. The molecular screening allows to evaluate the separation performance of CO_2/CH_4 mixtures of different copolymer formulations in silico, reducing the number of experimental tests required and accelerating the adoption of alternative sustainable materials in gas separation processes.

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Continuous rotary membrane emulsification

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Membrane emulsification (ME) can generate monodisperse droplets in which the disperse phase (DP) is extruded, dropwise, into the continuous phase (CP) through a microporous membrane. ME requires lower energy to operate than traditional top-down emulsification methods (such as high-pressure homogenisation). However, its characteristic low concentration output limits large-scale use: Cross-flow membrane emulsification (XME), in which droplets are generated when the DP permeates through a membrane into a cross flowing CP, has been demonstrated industrially as a continuous process, but results in low emulsions with lower DP concentration compared to industrial homogenisers. In rotary membrane emulsification (RME), the DP is extruded from a rotating tubular membrane into the surrounding CP, with the DP concentrating to a desired level before terminating the process. However, as this is a semi-continuous process, scale up is challenging. Here, we present the novel design of a continuous rotary membrane emulsification (cRME) system, allowing decoupling droplet generation from CP flowrate. The decoupling results in higher productivity and greater control compared to traditional crossflow and rotational membrane emulsification processes. The cRME system consists of a stainless-steel tubular membrane and a 3-D printed flow cell. A design of experiment (DoE) investigated the influence on droplet formation of CP flow, membrane rotational speed and emulsion composition, using a Pickering emulsion consisting of 1 wt% keratin solution and varying concentrations of oxidised cellulose nanofibrils (OCNF) as a model system. Experiments showed that CP flowrate had a negligible effect on droplet diameters in a wide range (between 78 and 241 µm), with uniformity index as low as 0.14, i.e. high monodispersity. Continuous operation, the possibility of designing emulsions and increased productivity represent marked advances over conventional cross-flow and rotational membrane emulsification systems, opening the way to a wide range of applications in the food, drug, personal care and agricultural manufacturing.

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Continuous rotary membrane emulsification for the production of sustainable Pickering emulsions, E. E. Ekanem, A. Wilson, J. L. Scott, K. J. Edler and D. Mattia, Chemical Engineering Science 2022 Vol. 249 Pages 117328, DOI: https://doi.org/10.1016/j.ces.2021.117328

Cationic and anionic biopolymer platforms in thin-film composite biocatalytic gas-liquid membrane contactor for carbon capture process: Does it matter?

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Gas-liquid membrane contactor (GLMC) is one of the viable membrane technologies used in post-combustion carbon capture process due to its similar separation principle with conventional absorption column. Common liquid absorbent used in GLMC is monoethanolamine (MEA) due to its high CO_2 affinity and absorption capacity. However, this compound is prone to oxidation and has energy-intensive solvent regeneration process. Alternative solvents, such as carbonate salts, have been proposed to replace amine-based absorbent. Unfortunately, these solvents have slower CO_2 absorption rate and limited CO_2 affinity, making them unattractive for practical application [1]. Incorporating biocatalyst in GLMC could potentially solve the problems when using these alternative solvents to replace amine-based absorbent [2]. In this work, we prepared thin-film composite biocatalytic GLMC with two different bio-derived polyelectrolytes coated on top of porous hydrophobic support. The type of porous hydrophobic support, polyelectrolyte coating concentration, membrane coating method, immobilization parameters and process conditions were studied. The works showed that the coating thickness, the type of polyelectrolytes coating and the type of porous hydrophobic support greatly influenced the CO_2 capture performance. The immobilized biocatalyst onto anionic polyelectrolytes coating was able to improve the overall mass transfer coefficient (Koverall) compared to the cationic polyelectrolytes coating. In conclusion, anionic polyelectrolyte coating is more suitable to be implemented as a biocatalyst immobilization platform in thin-film composite biocataltytic GLMC for CO_2 capture process.

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Hollow Fiber Membrane Contactor System for the Recovery of Dissolved Biogas from Anaerobic Effluents

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Biogas produced in anaerobic digestion process has been considered as a renewable energy source which is derived from wastes. To recover biomethane from the anaerobic effluent and thereby maximize the energy recovery from anaerobic processes, a membrane contactor (MC) was employed as a mass transfer equipment for the CH_4 recovery. First of all, we developed a series of hollow fiber membranes using various materials and methodology. The tests revealed that our membranes performed better than commercial polypropylene membranes owing to their high porosity and hydrophobicity. The long-term stability of membranes was also successfully demonstrated with real anaerobic membrane bioreactor effluent. Energy balance between electrical energy obtained from the recovered CH_4 and energies consumed by vacuum and liquid pumps for the operation of membrane contactor were also investigated. Results revealed that a combination of a high strip gas flow rate and slightly low vacuum condition closed to the atmospheric pressure can provide the highest net energy at 0.178 MJ/m^3. In the following studies, the factors affecting the net energy production were further investigated and the strategies to control membrane fouling were proposed. Moreover, we prepared a wetting- and fouling-resistant hollow fiber membrane that can operate with realistic anaerobic effluents. Our membrane could sustain 7 days of continuous operation with UASB effluent, which contains a large amount of foulants, with a performance loss of only ~10%. We observed that the fouling layers were much richer in proteins rather than substances similar to humic and fulvic acids and microbial byproducts. Besides, we calculated that our wetting/fouling-resistant membranes provide a net energy benefit for the MC process (when the recovered CH_4 is retrofitted to the process as fuel). Thus, our results suggest that the MC-based CH_4 recovery process is a viable alternative for industrial practices in case the demonstrated custom-designed membranes are employed.

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Biogas upgrade with gas-liquid membrane contactors

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Biogas, as product of anaerobic digestion of organic substances, belongs to the RES with zero carbon footprint. Its utilization is typically performed by combustion in ICE towards heat and power production. However, due to the fact that biogas plants are typically located at considerable distance from residential areas, the cogenerated heat cannot utilized optimally, thereby reducing the overall efficiency of the process. A potential alternative route is to upgrade biogas to biomethane, which as an energy carrier, can be injected into the existing gas grid and transferred at long distances. This could also contribute in securing the gas and energy supply of Europe, one of the main EU's strategic targets in our days. Conventional technologies for upgrading biogas to biomethane include absorption, adsorption, cryogenic or membranes. A potential alternative hybrid technology, which combines the advantages of the membrane with that of absorption technology is Membrane Gas Absorption (MGA), i.e., absorption in gas-liquid membrane contactors instead of packed columns. This is a very promising new technology, with interesting advantages and further developmental potential, which has been increasingly studied in the last few years, especially for CO_2 capture applications [1]. In the current work the biogas to biomethane process is experimentally studied using commercial 3MTM Liqui-Cel membrane contactors and amine-based solvents. The experimental results revealed the high efficiency and adaptability of the process. High quality biomethane with customized properties (partial to complete H2S and CO_2 removal) can be effectively produced by a proper selection of the process operating parameters. Specifically, low gas residence times, high G/L ratios and MDEA as solvent favor selective H_2S removal, while high gas residence times, low G/L ratios and DEA as solvent favor combined H_2S and CO_2 removal. In all cases, high H_2S and/or CO_2 removal efficiencies, with bio-CH_4 recoveries >99%, were obtained.

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Zein nanoparticles preparation and recovery by membrane processes

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Biopolymeric nanoparticles (NPs) have drawn attention in different areas of application such as biomedical, drug delivery, as well as bio-filler in composite film. Zein, a prolamin from corn, represents a potential alternative bio-based material due to its outstanding properties such as good film-forming, biocompatibility, biodegradability and non-toxicity [1]. In this study, zein nanoparticles (ZNPs) were prepared by membrane nanoprecipitation as a simple method to produce stable NPs suspensions without the addition of stabilizing agents, by non-solvent induced phase separation. A systematic study regarding the preparation of ZNPs by nanoprecipitation in a conventional batch system and membrane process will be illustrated. The Ouzo diagram was built to identify and compare the stable region where uniform ZNPs are obtained from both processes. The investigation was pursued to account for the influence of the concentration of zein and solvent/non-solvent volumetric ratio on the size and size distribution of NPs measured by Dynamic Light Scattering (DLS). The size of ZNPs increased from 100 to 400 nm when increasing the zein concentration from 0.5 to 5 %w/v. Low values of Polydispersity Index (PDI < 0.2) were obtained among all the zein concentrations range. The size and the shape of the ZNPs have been monitored by Transmission Electron Microscopy (TEM). Membrane filtration methods (i.e., ultra-diafiltration) were optimized in order to increase the concentration of nanoparticles suspension without forming aggregates and results were compared with freeze-drying.

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Microstructured TiO_2-Al/Cu/Mg ceramic catalytic membranes for the degradation of dye laden wastewater

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Wastewater remediation using metal oxide based photocatalysis has been touted as a viable method in combating water scarcity [1]. However, recovery of these nano-structured catalyst, which are typically in powder form is an issue which requires close attention. Multiple attempts have been carried out to immobilise such photocatalyst onto various substrates with varying success [2]. In this work, a ternary TiO_2-Al/Cu/Mg composite photocatalyst was immobilized onto a micro structured silicon dioxide pellet. Traditional photocatalysts such as TiO_2 can synergistically be improve when coupled with other similar metal oxides such as Al, Cu and Mg by reducing the band gap and inhibit recombination rates. Meanwhile, silicon dioxide was chosen as the material of choice to fabricate the substrate pellets for their transparency when sintered at temperatures of 1400 ºC. A transparent substrate would allow more immobilized photocatalyst to be activated during photocatalytic reaction. The dense but microchanneled nature of the silicone dioxide pellets also offers high porosity and large number of active sites for photocatalyst loading. To prepare the pellets, a slurry of silicone powder, polymethyl methacrylate (PMMA) and n-methyl-2-pyrollidone (NMP) was ball milled for 48 h and subsequently degassed. It is then poured into molds and then sintered at 1400 ºC to form the pellets. The porous pellets were then used as the substrate to impregnate TiO2-Al/Cu/Mg composite photocatalyst via co-precipitation and subsequently calcined at 500 ºC for 3 h. The catalyst loaded pellets were tested under visible light conditions to degrade rhodamine B (RhB) and methylene blue (MB) as model pollutants. The prepared photocatalytic pellets exhibited characteristic peaks at 25.2, 38.0 and 54.7 in XRD analysis while porosimetry analysis indicated a surface area of 135 m^2/g. It is expected that the photocatalytic pellets can photodegrade the model pollutants while providing a facile way to recycle them for repeated use.

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Micro-patterned PVDF membranes and magnetically induced membrane vibration system for efficient wastewater treatment

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Polyvinylidene difluoride (PVDF) based membranes have been used extensively for wastewater treatment due to their chemical and mechanical stability. However, membrane fouling remains a critical factor limiting the widespread use of membrane processes. To mitigate membrane fouling, patterned PVDF membranes were prepared via spray-modified non-solvent induced phase separation (s-NIPS) which resulted in membranes with significantly larger pattern heights of 750 ± 100 µm. Patterned membrane modules were then subjected to magnetically induced membrane vibration (MMV) applied perpendicular to the direction of the pattern to reduce the foulants deposition and delay the irreversible fouling. The anti-fouling characteristics of patterned and vibrating membranes were studied in a submerged membrane bioreactor (MBR). Mere patterning resulted in about 70-300 % decrease in membrane fouling rate and a doubled critical flux as compared to the flat counterpart. The introduction of membrane vibration further improved the membrane anti-fouling potential by prevention of foulant deposition through improved hydrodynamic conditions near the membrane surface and efficient removal of foulant deposition. Introducing an axial vibration at 5 Hz of frequency dropped the fouling rate by an additional 40-60 % and the critical flux for irreversible fouling was almost tripled from 15 L m^{-2} h^{-1} for the flat non-vibrating membrane to 40 L m^{-2} h^{-1} for s-NIPS patterned vibrating membranes. A computational fluid dynamics (CFD) simulation with mesh motion at the patterned membrane interface confirmed the formation of vortices due to vibration, which are convected away from the membrane surface. The obtained CFD results provided a sound basis for understanding the effect of membrane topography combined with vibration. These overall results suggest enhanced fouling reduction without the need for intense air scouring and frequent cleaning which can result in energy- and cost-effective filtration during MBR operation

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Development of Nanofiltration membranes incorporating Artificial Water Channel

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Membrane-based technologies have a wide role in water purification and desalination. Artificial Water Chanel (AWCs) are synthetic aquaporins that can offer high water permeation with high selectivity. This work demonstrates that self-assembled imidazole-quartet (I-quartet) can be incorporated into polyamide Nanofiltration membrane in order to increase the performances of the membranes. Polyamide Nanofiltration membranes were fabricated via interfacial polymerization with AWCs and tested in desalination for Brackish water. An optimization was done to explore the best combination between AWCs and Piperazine monomer to achieve a maximum of performances in a defect-free polyamide membrane. The performance of the elaborated membranes was evaluated in cross-flow filtration, Nano filtration condition (12-10 bar) of brackish feed streams. As expected, the optimized AWCs membrane achieved an insane improvement, the permeability dramatically increases in the bioinspired membranes resulting in more than twice the permeability of the reference membranes (44 $L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$) while the NaCl rejection was maintained above 62%.

Synthesis of hybrid PES membranes via electron beam irradiation for selective removal of 17ß-estradiol from water

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The existence of estrogenic hormones in the aquatic environment poses a threat for human health since the traditional methods are not able to efficiently remove them. The selective removal of 17ß-estradiol (E2) was performed by synthetizing hybrid polyethersulfone (PES) adsorber membranes. The novel hybrid membranes were prepared by embedding molecularly imprinted polymer (MIP) particles in the membrane matrices by a phase inversion process. The hybrid membranes were irradiated with electron beam irradiation to immobilize the particles in the membrane scaffold [1]. Soxhlet extraction and dynamic adsorption experiments proved that irradiation was a key factor for immobilizing and fixing the particles in the membrane scaffold. The hybrid membranes were tested in batch-wise and dead-end filtration experiments. The static and dynamic adsorption capacity and selectivity were measured, and adsorption isotherm models were developed. The irradiated hybrid PES membranes exhibited a significantly higher dynamic adsorption loading compared to the reference PES membrane, with a value of 13mg/g and 1.4 mg/g, respectively. Furthermore, the hybrid membranes were able to selectively remove E2 from aqueous solution also in the presence of competitive molecules. The selectivity factor measured for the hybrid membranes were 6.2 for the adsorption of E2 over bisphenol-a and 12.5 for the adsorption of E2 over citalopram. Isotherm experiments were investigated, and Langmuir model fitted best with the experimental adsorption data. Maximum adsorption loading for hybrid membranes towards E2 was predicted to be 22 mg/g via Langmuir model. In addition, a simple regeneration process was proposed, and the hybrid membranes were able to be reused without the loss of adsorption capacity for at least 10 cycles.

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Chiral polymeric membranes prepared from polymeric blends with inherent optical activity

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Many chiral drugs and food supplements were developed and are still sold as racemic mixtures of both enantiomers. However, human metabolism is a complicated set of biochemical reactions involving many chiral recognition sites, which recognize only one of the two enantiomers found in racemic drugs, while the other one is found to be inactive or even toxic. Therefore, it is very important to use enantiomerically pure drugs to avoid undesired side effect caused by administration of racemic mixtures. Yet, enantioseparation of racemic substances on larger than laboratory scale has been a difficult challenge and membrane technology is a promising candidate to overcome this issue. In this work, we present a polymeric membrane with inherent chiral properties for enantioselective recognition and separation of racemic amino acids and similarly structured racemic compounds. Chiral polyimide based on enantiomers of 1,2-diaminocyclohexane was blended with various types of polymers to obtain film-forming polymeric solutions [1]. We show that combination of hydrophobic chiral polyimide with highly hydrophilic polyether-block-amide (PEBAX® 1657) allows for flexible polymeric membrane films with good permeability and excellent selectivity towards L-Tryptophan. Pertraction experiments reveal more than 99 % enantiomeric excess both in retentate and permeate compartments of our diffusion cell at the end of the experiment, owing to the high sorption selectivity and capacity of the membrane.

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Synthesis of novel organic fillers for efficient Mixed Matrix Membranes (MMMs)

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Mixed matrix membranes (MMMs) are prepared via the dispersion of an insoluble component (filler) into a soluble organic polymer (matrix), and combine the benefits derived by the processability of the organic support and the increased separation perfomance given by the fillers. Typical MMMs are prepared mixing a commercially available polymer, such as Matrimid® 9725, whereas common fillers are represented by inorganic and crystalline materials, such as Metal Organic Frameworks (MOFs) that exhibit a high porosity, which boosts the permeability of a gas, and a relatively easy functionalisation of their backbones, which improves the selectivity. Despite this combination seems ideal, the compatibility between the organic matrix and the inorganic fillers is often poor. This is a recurring problem, especially evident at the interface between the two components.[1] The most obvious solution is to substitute crystalline and inorganic fillers with amorphous and organic ones, which is likely to enhance the adhesion between fillers and matrix in a “like dissolves like” fashion.[2] Recently, we reported the synthesis of a series of novel hypercrosslinked Polymers of Intrinsic Microporosity (PIMs), employed for carbon capture and for the separation of CO_2 from N_2 and CH_4.[3] These materials showed a similar adsorption and gas separation performance as some of the best MOFs, which were complemented by an easier systematic functionalisation of their backbone. This talk focuses on their use as fillers for MMMs, in combination with Matrimid® 9725 as the soluble matrix, with the aim to simultaneously increase permeability, selectivity and interface compatibility of novel mixed matrix membranes. The starting hydrocarbon structure is highly microporous but also non-polar. Its functionalisation permits the creation of either basic (amines) or acidic sites (sulfonic groups). The difference in polarity, along with the altered hydrophilicity/hydrophobicity, helps the tuning of the permselectivity for different gases.

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What is the physical meaning of the time lag of mixed matrix membranes?

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The time-lag method is commonly used for the characterization of gas separation membranes. It allows the determination of the diffusion, permeability, and solubility coefficients from a single dynamic gas permeation experiment. This method is also used to characterize mixed-matrix membranes (MMMs). Recently, we considered ideal MMMs consisting of a different number of repeatable layers.[1] The single repeatable layer had uniformly dispersed impermeable particles of the same shape, orientation and size in a continuous polymer phase. Assuming an ideal polymer/particle interface, we numerically simulated the time-lag experiments by solving the governing partial differential equation. The solution allowed us to investigate the effect of the number of repeatable layers, i.e. MMM thickness (l), on the simulated effective time lag (TL), which revealed that TL is not directly proportional to l^2. Consequently, the effective diffusivity (De,TL)determined from the simulated time-lag experiment depended on the number of repeatable layers. It was different from the constant effective diffusivity (De) determined by the ratio of the effective permeability (Pe) and solubility (Se) of the MMM. The difference between De, TL and De decreased with the increase of repeatable layers and practically disappeared for MMMs with five or more repeatable layers [1]. This study extended the analysis to MMMs with permeable cuboid particles. We considered two groups of MMMs with particles: 1) more permeable and 2) less permeable than the continuous polymer phase. Within each group, we considered how the difference between De, TL and De is affected by varying the solubility and diffusivity of particles while maintaining their product, i.e., the particle permeability, constant. We also investigated the effect of the relative size of cuboids (i.e., volume fraction of dispersed particles in MMM) by independently varying their relative thickness and the projected area normal to the main direction for permeation.

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Benzimidazole-dichlorimidazole zeolitic imidazolate frameworks based on ZIF-7 and their application in mixed matrix membranes for CO_2/N_2 separation

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This study investigates the synthesis of a hybrid zeolitic imidazolate framework (ZIF) based on a solid solution of zinc benzimidazolate and zinc dichloroimidazolate, and its incorporation into a polymeric matrix. ZIF-7/COK-17 (the names of the end-members) has been obtained with a rhombohedral sod structure by combining in-situ two ligands under mild conditions. The hybrid ZIF outperforms its single linker parent ZIFs thanks to its open structure and high CO_2 uptake. According to the temperature used during the synthesis, two different particle sizes have been achieved, 250 nm and 450 nm, at 0°C and room temperature, respectively. This novel ZIF has been further incorporated within a commercial polymer: PEBAX MH-1657, a copolymer based on polyamide and polyether oxide. The effect of ZIF size has been investigated by studying the separation performance (CO_2 versus N_2) and morphology of MMMs. The addition of hybrid ZIFs enhances the separation performances with an increase in CO_2 permeability as well as CO_2/N_2 selectivity. It induces a decrease of diffusivity coefficient and an increase of the solubility coefficients for CO_2 regardless of the particle size. However, the MMM with the smaller ZIF particles exhibits the best performance, with a selectivity of around 200 for CO_2/N_2 (four-fold increment compared to pure polymer) and a CO_2 permeability of 64, which exceeds the current 2008 Robeson upper bound.

Preparation of polymer/porous coordination polymer mixed matrix membrane for separation of hexane isomers

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Mixed matrix membranes (MMM) are regarded as promising separation technology for liquid and gas separations. However, finding a suitable type of filler to offer high permeance and selectivity remains a significant challenge. Herein, we report a porous coordination polymer (PCP) {[Zn(1,3-H2BDP)2(TPA)2]·DMF} (1) incorporated into two different polymers: tertbutyl-polybenzimidazole (PBI-BuI) and polyethersulfone (PES) polymers, to prepare MMMs by the phase inversion method. The loadings of PCP1 as a filler were varied between 0-8 wt.% to give MMM0-MMM8. The influence of PCP1 on the morphology of membranes was investigated by SEM, EDS, PXRD, TGA, and contact angle measurement. Both EDS and PXRD confirmed the successful incorporation of PCP1 onto membranes. SEM analysis indicated an increase in surface pores (pore sizes) of membranes with the PCP1 increase from MMM0 to MMM8. The contact angle measurements also showed improved hydrophilicity as the PCP1 loadings increases from MMM0 to MMM8. Furthermore, the membrane performance was evaluated in terms of flux and selectivity using a dead-end filtration cell and GC, respectively. As a result, the flux of hexanes showed a decline as the PCP1 loadings increased, and the selectivity showed a preference for linear hexane (nHx) over 2,3-dimethylbutane (2,3DMB) hexane with the selectivity coefficients between 1.0 and 1.2 in all the membranes (MMM0-MMM8).

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Dendritic polymer functionalized carbon nanodiscs as additives for water treatment membranes

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Water use has been increasing over time, driven by a combination of population growth, socioeconomic development, and changing consumption patterns. These factors led to the shortage and uneffective management of water resources, which raises the need to find new ways of water recovery. Water treatment technologies based on membranes tend to be established as the main technology for the desalination of brackish and seawater, for the treatment of drinking water as well as for the treatment of various wastes to recover clean water. In the present study, carbon-based nanoparticles were added to the PVA matrix in order to improve membranes properties and then the nanocomposite membranes performance in water treatment was evaluated. Since carbon-based nanoparticles’ dispersion in challenging in aqueous solutions [1], the following modification process was carried out in order to render them water soluble: two guanidinylated derivatives of hyperbranched polyethyleneimine with molecular weights of 5 and 25 kDa (GPEI5K and GPEI25K), were used as functionalising agents on oxidized carbon nanodiscs (oxCNDs). Nanocomposite PVA membranes were then prepared containing 0.1, 0.25 and 0.5 wt% of the above mentioned nanoadditives. Subsequently, the membranes of about 60-70 µm in thickness were crosslinked using glutaraldehyde, rendering them water stable.[2] The hybrid nanomaterials, oxCNDs@GPEIs, were physicochemically characterized using a variety of techniques such as 1H and 13C NMR, FT-IR, SEM, TEM, RAMAN and XRD. Furthermore, the prepared membranes were studied in regards to their water permeability and their anti-fouling properties. They were also characterized by SEM, FTIR, XRD and mechanical properties. Finally, the antibacterial properties of these membranes were assessed against Gram-positive bacteria, Escherichia coli. The developed membranes exhibit improved mechanical anti-fouling and antibacterial properties. Based on the above results, it was found that the nanocomposite membrane with a loading of 0.25% oxCNDs@GPEI-25kDa exhibited overall better antibacterial and rejection performance.

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Application of Mg(Al)O mixed metal oxides (MMO) nanoparticles in the preparation of sustainable polymeric membranes

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Today, developing processes to efficiently reduce water pollution has become crucial. Among several methods being developed, membrane technology provides many advantages, including high efficiency, low energy cost, and ease of use [1]. Although polymeric membranes are undoubtedly important in water and wastewater treatment, their fouling, on the other hand, is still a major drawback, as it reduces flux during filtration. Therefore, Mixed Matrix Membranes (MMM) with improved selectivity, permeability, and antifouling properties, are becoming a field of interest. Another common challenge in membrane preparation is substituting classically used hazardous solvents, such as dimethylacetamide (DMAc) or N-methyl pyrrolidone (NMP), with non-toxic alternatives. One of the intriguing nanoparticles (NPs) that could be exploited in membrane modification are mixed metal oxides (MMO) generated from layered double hydroxides (LDH) precursors. Thanks to their unique properties such as hydrophilic nature [2], very low cytotoxicity, high thermal stability, large surface area, and low cost, these NPs are garnering increasing attention in the environmental field. Mg(Al)O MMO NPs were chosen as nanofillers for MMM preparation following encouraging results as low-cost adsorbents in prior work [3]. In this study, Mg(Al)O MMO NPs were prepared through one-step pyrolysis of MgAl-LDH precursors. Then, using Rhodiasolv® PolarClean as an alternative green solvent, homogenous dope solutions were obtained at 80°C. The membranes were fabricated by non-solvent induced phase separation (NIPS) technique using water as a coagulation bath. The effects of various concentrations of NPs (0.1 - 1 wt.%) and additives such as PEG200, PVPK17, and PVPK90 were investigated. The as-prepared Mg(Al)O@PES membranes were characterized in terms of morphology, porosity, thickness, pore size and distribution, contact angle, and pure water permeability. Furthermore, rejection and antifouling ability were investigated using Acid Red 4 as a feed solution. The optimum Mg(Al)O loading was determined between 0.5 and 1 wt.%.

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Development of electrospun G-C_3N_4 NT/CDs polyacrylonitrile (PAN) nanocomposite membrane for improved microfiltration performance

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In the present work, the effect of graphitic carbon nitride nanotubes/carbon dots G-C_3N_4 NT/CDs nanoparticles (NP) on membrane properties and the microfiltration performance of electrospun polyacrylonitrile (PAN) membranes under different hot-pressing condition was systematically investigated. Firstly, the G-C_3N_4 NT/CDs nanoparticle was synthesized and characterized using scanning electron microscopy (SEM) and transmittance electron microscopy, and XRD. The different weight percentage of NPs (0.5, 1 wt%) containing 12 wt% PAN solutions in N, N-dimethylformamide was prepared. The electrospinning was carried out at 24 kV and a flow rate of 0.5 mL per hour. The characteristics of the fabricated membranes were assessed using FTIR, SEM, and atomic force microscopy. The nanocomposite electrospun membrane containing optimum content of NPs (1 wt%) showed a threefold increase in permeability during filtration of pure water. Finally, the MBR operation was conducted with the optimum nanocomposite electrospun membrane and showed improved biofouling potential (~37%) according to TMP values. The results of this research demonstrated the hot-pressed electrospun nanofiber membranes may promising for microfiltration applications with high performance and low degree of biofouling.

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Removal of heavy metals from surface water using chitosan/ZIF-8 mixed-matrix membranes

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This study presents the synthesis of a nanocomposite thin-film (NFC) RO membrane of chitosan with the zeolitic imidazolate framework (ZIF-8) at low cost, on 2 types of PAN supports, namely wet (PAN-w) and dry (PAN-d). The membranes were characterized by SEM, XRD and FTIR analyses, the results indicated that ZIF-8 was successfully introduced into chitosan. The PAN-w present the high performance. 3% CS and 15% ZIF-8 MMMs were the optimal concentration and a membrane thickness of 250 µm. Water contact angle analysis showed that all membranes are hydrophilic and there was a significant change in the hydrophilicity of wet and dry supported membranes. Synthetic waters at 15 ppm containing the ions As^{+3}, Cd^{+2}, Cr^{+3}, Ni^{+}, Pb^{+2} and Sb^{+3} presented through the MMM CS/ZIF-8 at a pressure of 30 bars, show the rejection ions respectively; 13%, 88%, 98%, 96.5%, 85% and Sb: 24%. The permeate flow was around 134 L.m-2.h-1. However, a higher permeability 4.6 L/m-2.h-1/bar. The other synthetic solutions, each containing one ion, were filtered under the same working condition as the previous one and the rejection rate had increased significantly for the Cd^{+2}, Pb^{+2} and Ni^{+2} ions respectively 97; 96; 97% and Cr^{+3} has not changed remains 98.5%. The permeate flux decreased slightly for Cd^{+2}, Ni^{+2}, Pb^{+2} respectively 111; 124; 108 L/m2h and strong decrease was observed at Cr^{+3} ion 99 $L/m^2.h$, and the permeability varies from 3.4 - 4 $L/m^2h/bar$, compared to commercial membranes with TFC which guarantees the economic profitability of the process of filtration under an applied driving pressure of 28.97 bar and the energy consumption was brought to 2.10-3 kWh.m-3.

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The Micropollutant Removal Potential of Ultrafiltration Membranes Prepared with Bismuth Metal in Addition to Their Antifouling Properties

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The need for clean water resources is increasing day by day, making conventional water treatment technologies inadequate. At this point, membrane technologies emerge as a solution. One of the biggest factors affecting the widespread use of this technology is membrane fouling. In addition, the control of micropollutants, which has been a major problem in water pollution in recent years, is possible with membrane processes [1]. By preventing biofouling and reducing energy consumption, barriers to membrane technology can be removed and smart water management in cities can be made possible. For the solution of biofouling, biofilm formation must be prevented in the first place and a membrane resistant to biofouling must be produced. In order to reduce biofouling, polysulfone (PSF) membranes were made by adding Bismuth metal to the dope solution. Bismuth containing antibiofouling membranes were produced by phase inversion method with casting solution which contained different concentrations of selected metal to observe maximum performance of antibiofouling and organic micropollutants removal. A series of filtration and characterization studies including Fourier Transform Infrared Spectrophotometer (FTIR), Scanning Electron Microscope (SEM), etc. were carried out to determine the micropollutant removal. According to the results from this unique study, the membrane containing Bismuth metal played an effective role in the removal of micropollutants.

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Membrane Integration in Biorefinery Processes

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This lecture discusses the integration of membrane operations in biorefinery processes, aiming the valorisation of bioresources following a circularity approach. Several case studies will be presented and discussed:

1 - the biorefining of microalgae, integrating their harvesting by ultrafiltration / low-shear centrifugation and the reuse of cultivation media, together with the recovery of various valuable fractions such as proteins, lipids and pigments;

2 - the recovery of valuable plant sterols during the processing of vegetable edible oils, combining their enzymatic biotransformation into sterol esters and their recovery using solvent resistant nanofiltration.

Critical issues related with process integration will be addressed as well as a techno-economical analysis of the processes developed.

A Multifunctional Electrochemical Membrane System for Chemical-Free and Low-Energy Regulation of Solution pH

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A proper pH environment is essential for a wide variety of industries and applications especially related to water treatment. pH adjustment is commonly accomplished by dosing strong acid and base. The electrochemical processes triggering water splitting also contribute to the local solution pH variation. However, these methods demonstrate disadvantages associated with environment and energy. Here, we designed a multifunctional electrochemical membrane system (EMS) with one piece of filtration membrane inserted into an electrochemical cell. When electrical field was applied, hydroxyl and proton ions were produced from reduction and oxidation reactions at cathode and anode, respectively. The membrane posed a resistance for the transport of hydroxyl and proton ions and prevented their mixing in the cell. The EMS can be also operated in a filtration mode, which could simultaneously regulate permeate and feed pH and accomplish water filtration. In both non-filtration and filtration modes, EMS could achieve effective control of solution pH over a wide range by exerting different voltages without dosing any chemicals. Under the voltage of 1.2 V, the solution pH could reach and be maintained at 10.7 and 3.3 in cathodic and anodic channels, respectively. Furthermore, it was experimentally demonstrated that the system only consumed extremely low energy. The EMS provides a chemical-free and low-energy option for effective pH adjustment and addresses shortcomings of conventional methods. Meanwhile, the combination with the filtration membrane makes this system more versatile and promising.

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Forward Osmosis-Membrane Distillation hybrid technology: Opportunities, challenges, and benchmarking

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Energy-efficient and cost-effective water desalination systems can significantly boost freshwater reserves without stressing the limited supply of energy. Most of the existing desalination systems are powered by non-renewable energy sources such as fossil fuel power plants. The sustainability of any desalination technology depends primarily on the type and amount of energy it utilizes and the product recovery. In recent years, Forward Osmosis (FO) and Membrane Distillation (MD) have drawn the attention of the scientific community because of their low energy requirements and the potential of operation using a low-grade waste heat or a renewable source such as solar or geothermal energy. Despite of the numerous potential features of FO and MD, there are still some limitations that may negatively affect their performance in terms of water-energy nexus. In this critical review, the hybrid forward osmosis-membrane distillation (FO-MD) system was the main target, with the energy demand and product quality in focus. We began by exploring the detrimental downsides of the standalone FO and MD technologies and their limited performance. Through this exploration, we demonstrate the importance of combining these technologies into a hybrid FO-MD system and the resulting strengths of this combined hybrid process. The promising applications of this hybrid technology in different areas and their advantages was also explored. Moreover, the performance of FO-MD hybrid system was compared with other hybrid systems like forward osmosis-reverse osmosis (FO-RO), forward osmosis-nanofiltration (FO-NF) and forward osmosis-electrodialysis (FO-ED). We outlined how FO-MD hybrid system could outperform other hybrid technologies when a suitable draw solution was used and a low-grade waste-energy source was utilized. Finally, it was concluded that the FO-MD hybrid system is a sustainable technology in regards to the water-energy nexus and a solution to global water scarcity could be found in this technology.

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Gas Separation Polymeric Membranes for Xenon Recovery

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Xenon appears as one of the most valuable anesthetics nowadays. The main reasons are: lack of metabolism and low toxicity, easy and absolutely controllable patient evocation etc. However, high price of xenon makes its utilization for anesthesia unavailable for the majority of the patients. Xenon recovery from patient's expiration during the medical procedures and reuse can become a way of lowering the price. The aim of this work is screening and research of effective polymeric membrane materials for hospital-based xenon recovery for reutilization in the medical procedures using both xenon-selective and xenon-barrier membranes. Xenon permeability data is rare and data of mixtures separation is unavailable in the literature. For the first step of the research an attempt of permeability and Xe/O_2 selectivity estimation was made for a set of polymers. Correlation uses available permeability data for different gases from the literature and own data base. The result of this work shows the classes of prospective materials with potentially high separation parameters. Among them: xenon-selective rubbers, high oxygen permeable perfluoropolymers with relatively high selectivity, polymers with high fractional free volume (PIMs, polyacetylenes). By the aging of the polymer matrix selectivity $\alpha id(Xe/O_2)$ usually decreases. In the first set of experiments MDK membrane with polydimethylsiloxane-based copolymer as selective layer was used for xenon and oxygen permeation. The results show the tendency of selectivity growth with temperature decrease: $\alpha id(Xe/O_2)$ was 2.96 at 80°C and 3.73 at 5°C while $\alpha mix(Xe/O_2)$ changed from 3.07 to 3.89 over the same temperature range. The experimental ideal selectivity at 35°C is 3.07 while the selectivity value predicted with correlations at this temperature is 3.04. Accumulation of experimental results and further modeling of xenon-containing gas mixtures membrane separation process will show technical and economic indicators for xenon reutilization in medical sector.

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Controlling L-Serine Antisolvent Crystallization Using Hydrophobic Polymeric Membranes

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In the current changing chemical industry, efforts are made to develop less energy consuming processes, more compact and environmentally friendly. Membranes have proven to be capable of controlling both energy and mass transfer of fluids, and are playing an important role in that evolution. Specifically, in antisolvent crystallization, random kinetic and thermodynamic aspects need to be limited in order to avoid undesired crystal size and shape [1]. In this work, a narrow crystal size distribution of L-serine using membrane-assisted antisolvent crystallization (MAAC) was obtained. Two commercial membranes made of polyvinylidene fluoride (PVDF) and polypropylene (PP), with a water contact angle of 130° and 150° respectively, were used. These membranes succeeded in controlling the transmembrane transport of antisolvent under different solution velocities in both sides of the membrane at ambient conditions. In all cases, membranes provided a narrow crystal size distribution, which was significantly better than either the commercially available L-serine, or the crystals resulting from batch antisolvent crystallization or drop-by-drop. Thanks to the quantification of both the evolution of L-serine and ethanol (the antisolvent) along the operating time, an intriguing dynamic of MAAC was observed. The increase of the antisolvent or the crystallizing solution velocities did not necessarily increase the antisolvent transmembrane flux as reported in [2]; instead, it has proven that an excess of one or another at a specific spacetime inside the membrane module can result in wetting or the system blockage, originating from the membrane, module, or tubing. This study invites us to use the principles of mass transfer in porous membranes to describe and gain understanding of the resulting crystal quality [3].

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Investigation of Innovative Hybrid Membrane Bioreactor (If-Mbr) Treatment Technology For Domestic Wastewater

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In recent years, because of the increase in the amount of water to be treated, research on new treatment processes that requires less space and very efficient in terms of economics and effluent quality has intensified. Membrane technologies are at the forefront of these technologies. Membrane Bioreactor (MBR) systems, which can be used instead of conventional treatment systems in domestic wastewater treatment plants, have created a widespread field of study because they are both economical and easy to operate. Due to issues such as the reduction in wastewater treatment plant discharge limitations as a result of growing pollution throughout the world, the difficulty to construct a treatment plant due to space constraints in metropolitan areas, and the necessity to modify numerous facilities to remove nitrogen and phosphorus, high wastewater treatment efficiency is achieved by employing lower areas with novel approaches. Within the scope of this project, a Membrane Bioreactor System (MBR) and the Integrated Fixed-Film Activated Sludge hybrid system (IFAS+MBR) installed as two parallel lines. Real wastewater that collected from Istanbul Technical University's sewere system was used as an influent of the parallel operated systems. Chemical Oxygen Demand (COD), Total Suspended Solids (TSS), Total Nitrogen (TN) and Total Phosphoros (TP) values followed at system's inputs and outputs regularly. By increasing the hydraulic loading rates, it was tried to determine the maximum amount of wastewater that can be treated in both parallel systems. The coefficients optimized by using the GPS-X program, taking into account the operating parameters. In the light of the data obtained in the design, it can be used as design parameter during the revision of wastewater treatment plants to MBR or IF-MBR hybrid system. In this way, economical engineering designs can be presented in future applications.

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Novel Trends in Membrane Contactor Processes: Behavioral and Structural Studies of Therapeutic Activity of Micelles of Emerging Green Solvents

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Hydrophobic deep eutectic solvents (DESs), a recent class of green solvents offer 100% atom economy, cost-effectiveness, potential biodegradability and negligible toxicity [1]. For augmenting biomedical applications wherein polar media is encountered, novel aroma-based therapeutic hydrophobic DESs were synthesised and dispersed in aqueous media to formulate nanoemulsions by newly introduced membrane-assisted nanoemulsification technique. Microengineered reusable stainless-steel isoporous membranes (9 µm pores) were fabricated by low energy-intensive laser drilling technique [2]. Novel hydrophobic DES systems were successfully synthesized combining terpenes (menthol & thymol) and aroma-based compounds (vanillin & raspberry ketone). For membrane emulsification studies, a 2% (w/w) Tween 80 aqueous solution was the continuous phase. Among the various DESs-based dispersed phase concentrations tested, 4% (v/v) DES operated at optimized dispersion rate exhibited monomodal distribution with stable emulsion droplets of ~147 nm size and polydispersity index <0.2. Interestingly, even though 9 µm pore-sized membrane was used, 60x smaller emulsion droplets were formulated. Although, we previously attributed these effects to the self-assembly traits of DESs in emulsion systems [2], various characterization studies were explored for in-depth understanding of these dynamic systems. The behavioral studies constituting the static contact angle, interfacial tension and electrokinetic stability measurements depicted unique observations. Furthermore, the structural studies of these emulsion systems were investigated for the first-time by advanced NMR techniques like DOSY, NOESY and ROESY to substantiate the intermolecular and intramolecular interactions and orientations existing within the emulsions. To harness the therapeutic effects of the starting components of hydrophobic DESs, first-ever studies on antifungal activities of these DESs-in-water nanoemulsions were explored. Four fungi strains, namely, Aspergillus fumigatus, Candida albicans, Candida krusei and Trichophyton mentagrophytes were tested successfully against the individual compounds, synthesised DESs and optimised nanoemulsions to determine the minimum inhibitory and fungicidal concentrations. Interestingly, enhanced synergetic therapeutic effects of the individual components within the nanoemulsions were witnessed.

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Pilot-Scale Synthesis of Novel Membrane Chemistries: Case Studies and Lessons Learned

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The rapid pace of research and development ensures that novel membranes are created every day. These innovations often utilize advanced materials or techniques to provide improved membrane performance. The advanced practices, however, often become obstacles to pilot-scale production of novel membranes, as there is no existing knowledge on their use with roll-to-roll techniques at larger scale. This challenge is further exacerbated by the fact that many membrane developments take place in an academic setting, which lacks direct access to the pilot-scale technical experience residing in industry. As it stands, these challenges pose a significant barrier to the development of advanced, novel membrane materials at pilot scale, which limits their potential for implementation in industrial settings. The Membrane Technology Group at KU Leuven has the unique opportunity of having an in-house pilot-scale membrane manufacturing facility in an academic setting. With this equipment, we are actively working to scale up the synthesis of many of the novel membrane chemistries developed in our lab for aqueous- and solvent-based applications. Several chemicals or process steps cause specific challenges for the fabrication of these membranes, posing a challenge to facile membrane synthesis. In this presentation, we will discuss our approach to the scale up of multiple novel membrane chemistries, while sharing the specific challenges faced by each membrane and the innovative solutions which were developed to overcome them. Lessons learned and best practices for membrane development at pilot scale will also be shared, with the goal to reduce the gap between cutting-edge membrane research and pilot-scale development.

Potential Reuse of Old RO Membrane as NF Membrane by Surface Regeneration

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With the inadequate presence of fresh water resources, desalination has been the key solution to address the problem of drinking water shortage in various developing countries. Thin Film Composite Reverse Osmosis (TFC-RO) membranes used in desalination are highly selective and efficient, due to the presence of its active thin polyamide layer. RO membranes suffer performance depreciation with respect to time because of extreme operating conditions of high pressure and salt concentration in the feed, presence of foulants and harsh cleaning conditions. Here in our study, we report the alternative route for disposal of the End-of-Life (EoL) membranes by treating the top polyamide, to convert them into Nanofiltration membranes using Sodium Hypochlorite (NaOCl). Membrane performance before and after the treatment is analyzed using Pure Water Flux (PWF) and the individual monovalent and bivalent salt rejection with 2000 ppm feed concentration at 15 bar operating pressure. Membrane acquired negative charge upon chlorination and is evident from its performance and characterization studies such as XPS and Zeta Potential. When treated at 2000-6 ppm-h (concentration-time) in alkaline pH, membrane exhibited % salt rejection of 63.8 ± 6.46 of NaCl, 91.8± 1.9 of $NaSO_4$, 84.5±1.6 of $MgSO_4$ and 66±2.8 of $MgCl_2$ with a higher pure water flux of 147.73 ± 32.26 LMH at 15 bar. Membrane is also characterized by FTIR, SEM and contact angle to check with its physical and chemical transformation. The regenerated membranes have obtained high flux with less energy requirement and is confirmed from the pilot scale study. An environmental friendly recycling process is established for the regeneration of the old RO membranes into NF membranes.

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Application of selected chemometric methods to describe and predict the properties of grafted ceramic membranes

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In recent years, methods for functionalization and/or hydrophobization of the surface of ceramic membranes have become increasingly important. Originally, ceramic membranes exhibit hydrophilic character resulting from the presence of the hydroxide groups on the membrane surface [1]. The most frequently applied modification methods involve surface hydrophobization. For this purpose, the technique of grafting with the application of silane compounds and their derivatives is successfully used. The structure of these compounds (modifier, linker) significantly impacts the properties of the obtained modified membranes. Chemometric methods such as hierarchical cluster analysis (HCA) and principal component analysis (PCA) can help to select the appropriate compound for membrane modification. The theoretical calculation of molecular descriptors (resulting from the structure of the compound) for individual modifiers and application of HCA and/or PCA enables to detection of multidimensional similarity. Therefore it is possible to plan their synthesis (or purchase, if commercially available) to obtain membranes with the desired properties. PCA also identifies parameters (physicochemical properties described by molecular descriptors) that impact the observed properties most. These chemometric methods can be used to interpret the already obtained experimental results, e.g., HCA can limit the number of consecutive experiments aimed at estimating the stability of membranes in various organic solvents [2]. Multiple regression analysis enables the selection of significant variables, leading to the equation predicting the value of the water flux observed in vacuum membrane distillation [2]. The work will present the most important findings of the application of chemometric methods to the prediction of fluxes and separation in membrane vacuum distillation using modified ceramic membranes.

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Zeolite membranes for gas separation: possible reconciliation between experiments, modelling, and molecular simulation?

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Inorganic zeolite membranes have been extensively studied for gas separation as they are beneficial in terms of thermal and chemical stability, high permeability, and selectivity. Besides, zeolites have uniform pores of molecular size making them very promising molecular sieves [1]. These membranes can be used for natural gas and biogas purification as well as carbon dioxide capture which are of great industrial interest. However, mass transport mechanisms in the nanoporous membranes are complex and their quantification is challenging (surface diffusion, gas translational diffusion, molecular sieving). We report here a multi-scale study coupling molecular, microscopic and macroscopic approach aimed to determine zeolite membrane efficiency and transport properties for the separation of industrially important gases (like CO_2, CH_4). - Experimental measurements of membrane performance (flux, permeance, and selectivities) are made for single gases and their mixtures at different operating conditions (T, P) using commercially available zeolite membranes. - Modeling of gas transport through zeolite membrane is used to estimate involved mechanisms and to predict transport properties considering classical 1D equations from literature [2]. Such models imply adsorption/diffusion terms (saturation coverage, intracrystalline diffusion coefficients, and activation energies) which can be hardly obtained from experimental approach. For this purpose molecular simulations are carried in addition to experiments. - Molecular simulation techniques as Grand Canonical Monte Carlo and Molecular Dynamics are applied to predict required parameters for mass transfer modeling. Interactions between particles are computed with force field parameters which allow one to gain an insight into adsorption, diffusion, and molecular sieving phenomena at atomic level from “ab initio” approach. Results are to be compared with experimental data. This project aims to reconcile three presented approaches for predicting separation performances of membrane-based processes. It can bring new insights on efficiency evaluation of zeolite membranes which could be later used in product design.

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Intermolecular interaction estimation in polymer solutions

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To design membranes targeted at specific applications, trying experimentally to find the right components in the polymer solutions is time consuming, since thousands of solvents and polymers are commercially available. More efficient ways to decide on the starting point of composition of polymer solutions are desired. To fabricate polymeric membranes with the properties aimed at, selecting the right components is crucial and this influences on the membrane morphology and pore size. Here, we studied basic ways to estimate the thermodynamic intermolecular interaction, both theoretically and experimentally. The chosen estimation parameters are the Flory-Huggins parameter calculated using Hansen solubility parameters and the activity coefficient calculated using COnductor like Screening MOdel for Real Solvents (COSMO-RS). We first computed the Flory-Huggins parameter and the activity coefficient using software tools. Subsequently, we screened the appropriate combination of solutes and solvents based on the values of the Flory-Huggins parameter and the activity coefficient. If the Flory-Huggins parameter is below 0.5, the solute and solvent are miscible. In case of the activity coefficient if it is below 1, it denotes that between solute and solvent there is an attractive interaction, while if it is above 1, the interaction is repulsive according to Raoult's law. These estimations help to understand the polymer solution system better and select the right compounds. This makes the design of the polymer solutions more efficient and effective and can eventually be extended to improve the membrane design. We believed that this study provides inspiration for defining approach methodologies based on the thermodynamic interaction of compounds as a first step in the design of polymer solutions.

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Flux estimation of dynamic ultrafiltration for wastewater treatment using combinations of machine learning methods

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Modelling dynamic ultrafiltration for real petrochemical industry wastewater treatment is challenging due to the high variance/uncertainty in inlet water composition and the time-variant nature of the process [1]. Then, several machine learning (ML) strategies plus their combination are investigated to determine their capabilities for membrane flux prediction under different production scenarios. The experimental data of dynamic ultrafiltration experiments are obtained at a real oil and production facility using a step-up and down strategy at constant flux operation. The dynamic operating conditions of these experiments were generated by using in situ back-shocks and backwashing at different frequencies. Dynamic ultrafiltration enables to mitigate fouling issues and allows operation with higher fluxes than conventional crossflow operation. However, fouling is not fully avoided, so a flux estimation is required for determining the best operation scenarios. Due to the limited dynamic system understanding, machine learning models are proposed since these data-driven approaches allow estimating system outputs from a black box perspective [2]. The used inputs were time, back-shock signal, backwashing signal, flux set-point of flux, and temperature. Particularly, the flux setpoint was employed as this is a manipulable variable for the flux control. Through simulations, the input signals are used to predict the experimental flux and transmembrane pressure required to achieve constant flux operation at each production scenario. The machine learning methods used were neural networks, decision trees, random forest, regression Gaussian processes and classical methods such as space-state and ARMAX. The novelty of the approach lies in the proposed combination of several models to achieve a prediction with low uncertainty. The minimum number of training models was also estimated for each machine learning strategy. The validation of the methods shows that the highest is obtained using neural networks, then they could be further investigated for more applications to determine the best production scenarios.

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Performance optimisation of curly hollow membrane modules using CFD

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Converting waste greenhouse gases such as CO_2, CO, and CH_4 into valuable products is an exciting opportunity that is gaining attention as we move from the linear economy to a more circular economy. Gas fermentation using bacteria as bio-catalysts is increasing in popularity as the reaction does not require extreme operating conditions and expensive catalysts [1]. One major drawback of gas fermentation is that it is limited by gas to liquid mass transfer resulting in low productivity. This problem can be overcome by using membrane modules to increase gas to liquid mass transfer. The relationship between the membrane bioreactor system hydrodynamics and performance is examined in this study. The effect of curly hollow fibre membranes on the mass transfer compared to the straight membrane systems is investigated. Studies show that different configuration of fibres (structured-straight, curly-fiber, central-tubing, spacer-wrapped and spacer-knitted module) enhances flux from 53% to 92% depending on the module [2]. In this project, there are three levels of investigation. The first one examines a single curly fibre (0.76 mm diameter) in a small tube (8 to 12 mm diameter). The second study compares multiple curly fibres in a small tube (8 to 12 mm) and multiple curly fibres (61 fibres) in a large tube (50 mm diameter). The experimental set-up is simulated using OpenFOAM software and the model validation is underway. Preliminary results from the single fibre systems showed that there is a range where the peak of the residence time distribution (RTD) curve is decreasing at a significant rate.

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Polyphenols - polyethersulfone interactions: Impact of ions on adsorption on ultrafiltration polymer membranes

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Polyphenols have recently received increasing attention due to their antioxidant and anti-inflammatory properties leading to numerous therapeutic applications [1]. Ultrafiltration membrane processes have recently emerged as an ecological and economical alternative for the extraction of polyphenols from biomass juices. However, the fouling of the membranes, which leads to a significant decrease in performance, limits the use of this process [2]. Various studies have shown that the presence of ions in solution impacts the membrane fouling. However, the mechanism by which cations impact fouling is still poorly understood [3]. The determination of the interactions present in the vicinity of the membrane is therefore essential to understand the fouling mechanism and thus improve the operating conditions. A model system containing a porous polymeric membrane made of polyethersulfone (PES) and polyphenols represented by tannic acid (TA) molecules was studied by molecular dynamics (MD) simulations at the atomic scale. First, the comparison of experimental and theoretical infrared spectra validated the modeling of the membrane and TA molecules. Then, the complete systems were modeled, including the PES membrane and TA molecules, in the presence of different solvents (water; water/NaCl; water/$CaCl_2$). The analysis of the radial distribution functions (RDF) between different characteristic groups of the system shows that hydrogen bonding interactions are at the origin of the adsorption of TA on the PES membrane, responsible for the fouling. This adsorption is also governed by π-stacking interactions. These same types of interactions are involved in the aggregation of the TA molecules, in agreement with experimental studies demonstrating formation of TA aggregates. Cations are associated with an increase in TA adsorption, accompanied by a decrease in aggregation. The RDF analysis explains the differences observed in the presence of salts. Indeed, the same groups are involved in the TA/cation and TA/TA interactions, which are therefore in competition.

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Towards the Generation of Machine Learned Potentials for Coarse Grained Molecular Simulations of Polymer Membranes for Gas Separations

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Molecular simulations offer deep insight into the molecular mechanisms underlying macroscopic behavior, supporting theoretical development and the discovery of structure-property relationship for a rational membrane materials design. In recent years, Machine learning (ML) is having increasing impact in membrane technology for gas separation, addressing problems that range from process control optimization to high-throughput screening of membrane materials [1]. Recently, Neural Networks have shown great promise in the development of improved atomistic force fields, trained on quantum mechanical calculations. Conversely, the implementation of ML for the generation of coarse grained (CG) force fields on the basis of atomistic simulations is a less explored topic. However, great untapped potential lies in the integration of ML techniques into multiscale molecular simulation studies of macromolecular systems, such as polymers, in particular in the context of Coarse Grained (CG) simulations, which currently require complex ad-hoc methods. A main advantage of ML models in the context of molecular simulations is that they are not constrained to a predefined mathematical function, therefore they are endowed with higher flexibility and expressive character compared to traditional CG models, thus they are potentially capable of accurately capturing many-body interactions and enable larger and longer scale simulations, which are necessary to study macromolecular systems. In this work, we adopted Graph Convolutional Neural Networks architectures [2] to develop CG Machine Learned potentials, implementing a scheme that includes a force-matching procedure [3]. The obtained models were used to perform CG Molecular Dynamics simulations. The structural and thermodynamic properties of the CG systems were systematically compared with the underlying atomistic reference, examining the effect of the Neural Networks model size and hyperparameters tuning on the simulation results. This methodology has the potential to streamline the generation of CG force fields, enabling systematic multiscale studies of complex molecules, including polymer membranes for gas separation applications.

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A mechanistic bridge between pulmonary epithelial membrane architecture, inhaled drug dosage and localized disposal, and permeation following dissolution: a fully parameterized predictive approach

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The enormous surface area of the pulmonary epithelium membrane (PEM) provides an exceptional and non-invasive administration route for Nanoparticles (NP)-based powdered inhaler (and homologues). A fairly comprehensive mechanistic bridging among PEM structure, aerodynamic guided deposition pattern, and dissolution followed permeability through distinct local PEM – which is solely necessary for its guaranteed efficacy, compliance, and safety - is presented. First, the post-natal evolution of the PEM surface area was parametrically (age, gender, height, and arm-span) modeled ($R^2 = 0.88$, $P < 0.04$) in accordance with Weibel's Trumpet-based symmetric lung morphology using 3,500 non-smoking data (health directory, lung autopsy, CT, and MRI scan data). It is subsequently coupled with an axial aerodynamic-controlled disposition model to predict region-selective fate of inhaled NPs on PEM as a function of PSD and (breathing interpolated) inhaling pattern. The NP's subsequent post-dissolution permeability across the (airway and alveolar) PEM is evaluated from the inverse of the net resistance, encountered by the molecule, during the surrogate's (16HBE14o- and Calu-3 cell-lines respectively) in vivo permeation-assay. Predictability was further enhanced by a novel machine-learning-based linkage with the chemical space of the drug and the distributed compositional matrix of the cell-line membranes (largest reported sample size 150 of curated permeability data, $\text{RMSE}_{[\text{train, test}]}$ = [0.81, 0.88] and [0.87, 0.93], and $\text{MSE}_{[\text{train, test}]}$ = [0.27, 0.34] and [0.24, 0.30] respectively). By employing a parametric sweep, the role of formulation and administrative-configurations of this route are presented together with pertinent available data. A multi-objective constraint optimization is used to demonstrate appropriate settings for a regulated (region and dose selective) targeting within intricate PEM network. Finally, via integrating this distributed (controllable) drug-dose throughout the physical space of PEM (area, morphology, and permeability) – a virtual bio-availability projection approach is proposed (with comparison to literature-data) to aide formulators during different stages of drug (product) development.

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Graphical abstract and Abstract:
Drive-link: https://drive.google.com/drive/folders/18IpAbDcUHzO6owoKji41cMa_nooZP7gC?usp=sharing

Upscaling preparation of polymer-based membranes

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The Membrane Technology Group of KU Leuven has a roll-to-roll pilot-line for membrane preparation that has been used to upscale corrugated membranes for use in MBRs and algae harvesting, and UV-cured polysulfone membranes for use in solvent-based feeds and as support material for thin film composite membranes. These membranes will be presented and the coating line described.

The newest challenge is the up-scaling of mixed matrix membranes (MMMs) for gas separation. A recent development allowed preparation of zeolite-filled membranes with record-high combinations of selectivity and permeability for a variety of gas pairs, including CO2/N2, CO2/CH4 and N2/CH4, reaching the right-top corner of the corresponding Robeson plots. It involved tuning of the zeolite shape and composition, as well as careful selection of solvent and curing conditions. With such separation properties and with their excellent mechanical stability and flexibility, even at high loadings (> 55 wt%), the preparation of such supported MMMs as thin film nanocomposites becomes interesting.

Complementing this more applied work, the Membrane Technology Group also aims at better understanding membrane formation processes at a more fundamental level and has developed microscopic techniques using microfluidic chips to visualize membrane formation during interfacial polymerization and phase inversion processes. This characterization technique will be presented and some examples shown.

New concepts for submerged membrane modules in water treatment

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Submerged membrane modules either in flat or hollow fibre configuration have established themselves as a standard module for membrane bioreactors (MBRs) in municipal and industrial wastewater treatment. In recent years two new concepts using submerged membrane modules have been developed for (1) direct membrane filtration (DMF) for municipal wastewater treatment and for (2) storm and rain harvesting. The key difference between the established MBR concept and the new concepts is that the biological treatment step is excluded. In this presentation the latest development and experiences with these two new concepts will be presented. The DMF concept operates abiotic and is an interesting alternative for municipal wastewater treatment. In this concept municipal wastewater is physico-chemically pretreated in terms of coagulation, flocculation and microsieving prior to the membrane treatment. Since the DMF concept has a high carbon rejection biogas production can be significantly increased resulting in a potential energy neutral or positive wastewater treatment process. Furthermore, the process has a smaller footprint compared to conventional wastewater treatment technologies. The potential of this application will be demonstrated based on three pilot installations in Sweden and Norway. The water harvesting concept upgrades storm- and rainwater collected from e.g., stormwater ponds, house roof or parking lots to a quality suitable to reuse the water for none-drinking purposes such toilet flushing and garden watering. In this case, the water is pre-screened and then polished with submerged membrane modules to remove micropollutants and microplastics. Results from a pilot installation in Sweden connected to a stormwater pond will show the capacity of this concept. Overall, it will be shown that the submerged membrane module concept has an application potential beyond MBRs.

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Valorization of Olive Oil Washing Wastewater through a hybrid system: Membrane processes, Adsorption/Desorption and Biological treatment

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Due to the increase in social pressures and awareness of improving the environment, it is urgent to find solutions to environmental problems, such as the large amount of wastewater generated by various industrial sectors. Moreover, the recovery of effluents and valuable substances is becoming a promising approach in the context of the circular economy [1]. The present work tries to comply with these two approaches considering the treatment of olive mill wastewater. For this, a hybrid procedure that included membrane processes, adsorption/desorption and biological treatment was proposed for the recovery of antioxidant phenolic compounds (PhC), and the reclamation of wastewaters for future reuse. The two-phase continuous centrifugation process is the most widely used in Spain for olive oil production. The wastewater generated, called olive oil washing wastewater (OOWW), was the object of this study. First, a pretreatment (flotation, sedimentation and filtration (60 µm)) was considered, to then proceed with ultrafiltration (UF), where the UP005 (Microdyn-Nadir) membrane (2 bar and 2.5 m/s) was selected to remove organic matter without affecting PhC concentration. The UF permeate passed to nanofiltration (NF), where the NF270 membrane (Dow-Filmtech) (1 m/s and 10 bar) was the best option for the concentration of PhC in the retentate stream. Forward osmosis (FO) using (HFFO.6 and FTSH2O) was also tested for this purpose with fermentation brines from table olive production as draw solutions. PhC from FO and NF concentrates were recovered with 40 g/L of the MN200 adsorption resin (Purolite) and a 50% ethanol/water solution as eluent. The streams without PhC, together with the UF retentate, were submitted to a biological treatment where the feasibility of a sequencing batch reactor to reduce the organic load was analyzed. The main results obtained with the combination of processes will be presented, as well as the costs associated with the implementation.

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Removal of nanoplastics by microfiltration and ultrafiltration membranes

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Plastics, once in the environment, undergo abiotic and biotic weathering processes that cause their degradation and fragmentation into smaller particles: microplastics (MPs<5 mm) and nanoplastics (NPs< 1 µm) [1]. Wastewater treatment plants (WWTPs) have been identified as one of the main dominant sources of MPs in freshwater [2]. Despite of the high removal ability of the wastewater treatments technologies, research efforts have been limited to the relatively big size MPs, leaving NPs out of the studied size spectrum. The aim of this study is to evaluate the removal of NPs (Fluorescent Polystyrene (PS), 120 and 500 nm) by microfiltration (MF) (pore sizes: 0.1 - 0.4 µm) and ultrafiltration (UF) (MWCO:30 and 150 kDa) membranes. The fouling mechanism of these pollutants was studied, evaluating the effect of different type of membranes and water composition. This study was complemented by surface membrane characterization (Scanning Electron Microscopy and Confocal Laser Scanning Microscopy). This study shows that PS of 500 nm was efficiently removed by all the membranes studied, obtaining rejection coefficient values of 100 %. Regarding the 120 nm PS, UF membranes also showed 100 % rejection coefficient. However, rejection coefficients decreased when using MF membranes obtaining 90 % and 26 % for 0.1 µm and 0.4 µm membranes, respectively. In general terms, the presence of BSA acted in two different ways: i) stabilizer, that helps to have a better dispersion of NPs, increasing the permeability recovery factor up to 80% for MF membranes and ii) foulant, that decreases the permeability recovery factor from 100% to 30 % in 30 kDa UF membranes. This study shows that membrane technology could be an effective tertiary treatment in WWTPs for the removal of NPs. However, there is the need for understanding better the fouling mechanism in order to propose efficient cleaning protocols.

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Pore-filling microfiltration membrane with zwitterionic polymer brush provides ultrahigh flux with an excellent virus removal from water

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Membrane technology is one of the most attractive alternatives to water treatment with high virus removal. Here we developed and investigated zwitterionic (ZI) polysulfobetaine (polySPE) brush pore-filling polyethersulfone (PES) microfiltration (MF) membranes for high virus removal and low energy consumption. To this end, a 0.22 μm PES MF was pore-grafted with the polySPE brush with different SPE concentrations (0.1 M, 0.2 M, and 0.5 M) using a two-step atom transfer radical polymerization (ATRP). ATR-FTIR, XPS, CA, and SEM confirmed the successful modification. The virus removal and removal mechanisms were estimated using bacteriophages T4 (80 nm) and NT1 (70 nm), used as surrogates for pathogenic human norovirus. Remarkably, up to 4.7 and 3.3 log reduction values (LRVs) of T4 and NT1 were achieved, respectively, using the pore-filling membranes, while maintaining membrane permeability at few ~103 LMH·bar-1. The virus removal mechanisms by the MF membranes were investigated using a mechanistic mathematical model with the pore size distributions determined by a liquid-liquid porometer (LLP). It was found that the size exclusion is the main removal mechanism for pristine MF membranes, but that adsorption was dominant for the ZI-polySPE pore-filling membranes. μ-X-ray fluorescence mapping (μ-XRF) was used to study the polymer brush effects on location and amount of 100 nm Silica spheres after filtration under similar conditions. Here, to elucidate the effect of the polySPE brush morphology, a model surface with a mushroom-like polyZI grafted (nonbrush) via free radical polymerization was also studied. Quartz crystal microbalance with dissipation (QCM-D) and the μ-XRF results corroborated, showing higher adsorption of T4/NT1 and Silica spheres, respectively, to the brush surfaces than the PES and nonbrush surfaces/membranes. These results correspond to the bacteriophages removal results with the pore-filling brush membranes and support the assumption that the increased removal was due to electrostatic and hydrophobic interactions.

Cr(III) recovery and water reclamation from electroplating industry effluents

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Untreated effluents of electroplating industries are important sources of pollution in the environment, especially due to the large concentration of heavy metals such as chromium, posing health risks for humans, animals, and marine life. Therefore, the chromium-containing effluents necessitate effective treatment methods in conjunction with the recovery and reuse of chromium into industrial processes. In this study, an integrated method of exploiting the rinse solutions of chromium plastic electroplating using reverse osmosis (RO) is suggested. The electroplating rinse (containing ~300 mg/L of Cr(III) and ~700 mg/L boric acid), having a pH 3.5, was fed to Filmtec SW30-2540 spiral-wound RO membranes with the concentrate stream being constantly recirculated to the feed tank until the Cr(III) concentration reached the specifications of the electrolyte bath (~10,000 ppm Cr(III)). The RO system was operated at different transmembrane pressures of 30, 40, 50 bars to define the optimal conditions. Operation at a feed flow rate of 0.7m3/h and transmembrane pressure of 50 bar, exhibited a 15% recovery and showed chromium rejection values of up to 99.95%. Since RO membranes show relatively low boron rejection at acidic pH values [1], the rich in boric acid, first-pass permeate was secondarily treated by the same RO membrane after pH adjustment (using KOH) to value 11 (the maximum allowed for the RO operation), achieving a boric acid rejection of 97.57%. The industry requirements of boron concentration were achieved by recirculation of the concentrate stream, while the boric acid concentration in the permeate was reduced to 2 mg/L, which even reaches the EU drinking water standards for boron. This methodology allows the recovery and reuse of chromium and boron in the electroplating industry while adhering to the wastewater discharge specifications.

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TEMPO-oxidized High Surface Charge Cellulosic Membranes for Removal of Charged Compounds from Water

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The removal of small inorganic salts from aqueous matrices is usually approached with tight nanofiltration or reverse osmosis membranes. Albeit efficient in terms of removal, these dense membranes require high pressure difference to obtain satisfactory flux performance. The problem of high-pressure gradient is the significant energy expenditure, which ought to be minimized according to the principles of green chemistry. As a novel way of removing trace salts from water, highly charged ultrafiltration membranes can be used instead. This approach will not completely replace the typical NF or RO processes, but could be used especially for dilute solutions of multivalent salts. Main benefit of using charged UF membranes in salt removal is the dramatically lower energy requirement in comparison to the tighter membranes. For this work, we intensify the surface charge of a commercial regenerated cellulose ultrafiltration membrane. For the cellulosic top-layer, TEMPO-mediated oxidation can be used for rather non-destructive surface modification method [1]. As a consequence of the oxidation, significantly more negative surface charges are observed. The surface charge enhancement was characterized with zeta potential and dye staining procedures. Intensified surface charge correlated with the increased number of carboxylate groups on membrane surface. The charged ultrafiltration membrane performance in removal of dissolved anionic compounds is investigated. The salt rejections of especially multivalent anionic species are increased as a function of membrane's carboxylate surface density. As model compounds for filtrations, different anionic substances found in wastewater streams are examined. For instance, 200 ppm sulfate rejection could be increased from 7 % up to 60 % by a mild surface oxidation of RC70PP membrane. In addition to surface charge, the effects of feed concentration and permeate flux on separation were examined, and the filtration performance of charge-modified cellulose membranes was compared to similar cut-off membrane with more neutral surface charge.

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Combined application of coagulation/flocculation and membrane filtration technology for the treatment of soap factory wastewater

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Soap wastewater contains generally appreciable concentration of both organic and inorganic pollutants. It is usually containing appreciable amount of oil and fatty acid matters that increase the organic strength of wastewater dramatically, raising it above legislative discharge limits [1]. Thus, improper treatment of such effluents may pose health hazards through posing risks to the aquatic environment. Therefore, constant efforts have been making to alleviate the pollution of this type of effluents. In this context, the present study investigates the efficiency of coagulation/flocculation combined to membrane filtration in the treatment of highly polluted soap factory effluents. Coagulation/Floculation experiments were performed according to Jar-test standard procedure with the operating conditions optimized using response surface methodology via a Box–Behnken design. DEF was performed using different MF flat membranes with pore size of 5µm, 1,22µm, 0,45 µm and 0,22µm µm and UF membranes with a molecular weight cut off (MWCO) of 100 kDa, 30 kDa, 10 kDa and 5 kDa. The optimization results of CF obtained revealed that the maximum removal of turbidity (99%) and COD (27%) were obtained for $Al_2(SO_4)_3$ concentration of 2.17 g/L, flocculant (Chimfloc) dosage of 55.32 mg/L and initial wastewater pH of 5.3. Regarding membrane filtration process, turbidity and COD removal rates gradually increased with the decrease of membrane pore size ranging from 99% and 34% using 5µm-MF to 100% and 49% using 0.22µm-MF, respectively. For UF membranes, maximum turbidity removal (100%) was obtained regardless MWCO of the membrane while COD removals increased from 60% using 100 kDa-UF to 88% using 5 kDa-UF. Regarding the efficiency of treatment techniques used, CF-UF showed more efficient treatment performance compared to CF-MF. Overall, the obtained results can be useful for the development of industrial installation to treat and reuse soap factory wastewater using CF combined to cross flow ultrafiltration process.

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Role of the membrane's properties on bubble formation, mass transfer coefficients and efficiencies during the aeration process

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Aeration in wastewater is the operation of adding directly gaseous air to wastewater in order to promote oxygen transfer as a solute in the aqueous phase. The key problem affecting the process is the limitation of the mass transfer at the gas/liquid interface due to the low solubility of oxygen in water [1]. This cause low efficiencies and therefore high costs of this unit associated to the energy demand of compressors. To overcome this drawback is necessary improve the exchange between the phases acting on the mass transfer coefficient of the process KLa in which (a) is the specific available exchange interfacial area. To promote the interphase area, porous membrane could be an appealing alternative to traditional systems due to their high porosity which results into a high exchange interface area and a low volume of the equipment. In bubble aeration, the contact surface is the sum of the surfaces of the bubbles in the system and the surface area offered by the membrane. In this context, the bubble size plays a key role since it affects the contact area and residence time thereby affecting the whole process efficiency. The dimension of the bubbles formed is affectd by many operating conditions such as the flowrate, the dimension of the pores of the aerator and the adhesion of the bubble which is related to the surface properties of the solid [2,3]. The aim of this work is to investigate the feasibility of the membrane system as aerator device studying the mass transfer coefficients and the efficiencies at different operating conditions. Moreover, during the experiments, membranes with different characteristics (material, pore size, configuration, geometry) were used and images of the bubbles formed were captured. From these images, size, release time, number in the system and total exchange interfacial area were evaluated.

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Implementation of Quorum Quenching to limit bacterial communication in membrane bioreactors.

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Quorum Quenching (QQ) has recently emerged as an innovative solution for fouling control as a Quorum Sensing (QS) limiting tool in membrane bioreactors (MBRs). QS is defined as cell-to-cell communication mediated by N-acyl-L-homoserine lactones (AHLs) signaling molecules, allowing bacteria to form a biofilm on the filtration membrane [1]. QQ consists in disrupting the QS by acting on the AHLs. QQ uses porous polymer solid materials (QQ-media) composed of Polyvinyl alcohol and Alginate mixture to trap bacteria (in beads, hollow cylinders, or sheet shapes) with the ability to disrupt quorum sensing molecules through enzyme production. These enzymes, by catalyzing degradation reactions of AHLs, will delay biofilm formation [2]. Even though the effectiveness of using bacterial QQ to delay the rate of fouling has been proven in literature, its development has been done without a transversal and integrative scientific approach to the different phenomena involved, allowing the process optimization. This is the objective of this study, which will address issues related to the QQ-mat material and the transport of the AHLs signal molecules in the QQ-media. In this regard we report on developing a new QQ-media while investigating the effects of different polymer mixture compositions and preparation conditions on the hydrogel's mechanical properties in relation with the AHLs adsorption capacity. The physicochemical properties of these hydrogels were systematically characterized using scanning electron microscopy (SEM), Brunauer–Emmett–Teller, texture profile, and rheological analysis. Mass transfer studies were carried out using batch adsorption experiments of AHLs from the mixed liquor to the QQ media. The studies revealed that the elasticity, hardness, surface area, and porosity of the QQ media distinctly differ based on the initial polymer mixture composition and the preparation conditions, which in turn influenced the mass transfer of AHLs from the liquid phase to the QQ media.

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Preparation of Natural Polyelectrolyte Complex Membranes through Sustainable Aqueous Phase Separation

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Membrane technology plays an important role in many fields, including agriculture, industry, and medicine. Recently, an aqueous phase separation (APS) approach has been successfully developed to prepare synthetic polyelectrolyte complex membranes. This novel approach uses water as both solvent and non-solvent, which effectively eliminates the need for harmful organic solvents as required for the traditional nonsolvent induced phase separation (NIPS) method. In this work, the green APS approach was applied to natural polyelectrolytes to prepare even more eco-friendly and sustainable polyelectrolyte complex membranes. Two natural polyelectrolytes, polycationic chitosan (CS) and polyanionic sodium carboxymethyl cellulose (CMC) were used. By changing the pH and monomer ratio of the solution, a desired homogenous casting solution was prepared at low pH where CMC was uncharged. Integral and stable CS-CMC membranes were obtained in an acetate buffer bath of higher pH. In the bath, CMC became negatively charged, and thus formed complexes with the positively charged CS, leading to the formation of membranes. The structure and properties of the membranes could be tuned by changing the concentration of the coagulation bath. All the membranes showed an asymmetric structure with a dense top layer and porous inner structure. Moreover, glutaraldehyde could be used as a crosslinker to further improve the properties of the membranes. The membranes showed tunable pure water permeability and were able to be used in water treatment, for example, to purify an oil-in-water emulsion. This work demonstrates that APS is an alternative method to fabricate more sustainable natural polyelectrolyte complex membranes.

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Preparation of porous PET membranes using recycled polymer in a phase inversion process

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Membrane preparation via nonsolvent-induced phase separation (NIPS) is a well established process to create porous membrane materials from conventional polymers like PES or PVDF. Unfortunately, other common polymers like PE, PP, or PET are not suitable for the NIPS process due to the lack of solubility in commonly used solvents, which are applicable in large scale processes. In this work, a new approach to prepare microfiltration membranes from PET via NIPS is presented. PET is a promising material for membranes as it is highly inert and possesses good mechanical and chemical stability, especially considering its stability against organic solvents. Furthermore, PET can be obtained from plastic waste, which has good availability and a lower price compared to other membrane polymers. A previous study [1] used harmful and expensive solvents like trifluoroacetic acid, dichloromethane, or hexafluoroisopropanol to prepare PET membranes via NIPS. In this study, an alternative approach was developed using common solvents and unconventional process parameters, namely higher temperature of the membrane casting solution, to create PET membranes. Recycled PET from different sources (beverage bottles, mixed food-grade PET, PET recyclate) was tested and evaluated regarding its applicability for membrane preparation. The casting solution needs to be processed in hot state and the coagulation can be carried out as usual in the NIPS process. Conventional pore forming agents like e.g. poly(ethylene glycol) or poly(vinylpyrrolidone) were applied to support uniform pore formation. A nonwoven support was used to improve the mechanical stability of the PET membranes. Different PET membranes were prepared according to a design of experiments approach and a thorough characterization was carried out. The membranes were characterized in detail and compared to a commercially available PVDF membrane, demonstrating a competing membrane performance. The new NIPS procedure enables the preparation of sustainable, cheap, and robust PET membrane filters.

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Polymerizable ionic liquid-based membranes for transport in aqueous media

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Polymeric membranes can be modified to improve the efficiency of separation processes in a variety of fields, such as metal ion separation. One of these possible modifications is the incorporation of ionic liquids (ILs) in the membrane resulting in the development of numerous IL-based membranes such as polymer inclusion membranes (PIMs) [1]. Nevertheless, these separation processes could be limited due to the gradual release and leakage of the unbound IL from the membrane during its utilization. To overcome this limitation, different options can be applied, one possible approach is the use of polymerisable ILs in the membrane formulation [2]. In this study, we have investigated the use of different ILs, specifically phosphonium- and imidazolium-based ILs, for the preparation of dense membranes for transporting target species in an aqueous medium. The membranes were prepared using a formulation based on the copolymerization of the polymerizable ILs with suitable acrylic monomers, specifically 2-hydroxyethyl methacrylate and ethylene glycol dimethacrylate, which was developed by Galiano et al. for the CO2 separation from other gases [3]. The influence of the IL on the obtained membranes has been evaluated using different chemical and morphological techniques such as SEM and FTIR, as well as other parameters including contact angle and water uptake. Moreover, static soaked tests were carried out to check the stability of the membranes in both acidic and basic media. Using the above-mentioned characterization techniques it was demonstrated that the great majority of the ILs investigated were suitable to produce dense membranes with a compact surface containing the IL polymerized in their matrix. Furthermore, all the studied membranes resulted stable in both acidic and basic media. Finally, the obtained dense membranes were applied for the transport of different target species, either organic or inorganic, in aqueous media.

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The use of 1,2-diaminocyclohexane as the new key in solvent-resistant nanofiltration membranes.

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SRNF is becoming very popular over the past decades, as it is an alternative or additional technique to separate and concentrate mixtures on a molecular level. Thin film composite membranes are state-of-the-art membranes for NF comprising a thin toplayer on top of a porous support. The most common TFC membranes are polyamides, typically prepared by reaction of tri(acyl chloride), often trimesoyl chloride, and a diamine, often m-phenylenediamine. In this project, 1,2-diaminecyclohexane is used as diamine. It is very reactive, rigid and mimics PIMs. In addition, it has two asymmetric centers as close as possible to each other, giving it utmost probability to use stereochemistry as an additional tool to steer the membrane performance. First, it is combined with TMC. This membrane performs better than a standard MPD/TMC membrane and demonstrates that stereochemistry can be an attractive parameter to tune the membrane performance. Diastereomeric interactions of the amine monomer are identified as a promising tool to change the membrane MWCO curve, hence, its performance. However, many separation processes in organic chemistry involve extreme conditions, e.g. aggressive solvents and high or low pH. Membranes stable in those conditions gained less attention due to more challenging reactions. Since polyamide cannot withstand these extreme conditions, this project also aims to prepare a polyamine membrane. Analogous to trimesoyl chloride, first 1,3,5-tris(bromomethyl)benzene is used. Although promising results are obtained, no reproducible membranes could be achieved. By changing the amount of functional groups of the bromomethylbenzene from three to four, a cross-linked polymer is obtained more quickly. This results in a reproducible membrane with outstanding performances.

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Preparation and characterization of chitosan-based membranes for Na-air batteries

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The rapid growth of the demand of energy storage technologies and the challenges posed by the scarcity of Li and other rare metals has led to the search of alternatives. Due to the natural abundance and low cost of sodium sources, sodium-air batteries are becoming a promising alternative. The energy density of sodium-based batteries is lower than that of Li based batteries, but the similarities in the physical and chemical properties of the Na-containing compounds and membrane-electrode assembly cells allow the latter to benefit from the developments achieved in the last decade on the former [1]. One of the biggest challenges is the large diffusion barrier of membranes towards Na+ due to the large size of the Na ion compared to Li ion. A low diffusion rate is related to a high-volume expansion upon cycling. Therefore, the fast transportation of alkali cations is critical to the diffusion rate. The finding and optimization of efficient materials within the circular economy is one task to uphold. Chitosan biopolymer (low cost and from natural resources, with polyelectrolyte nature) has been proposed as an alternative material for electrode and membrane separator fabrication due to its high hydrophilicity, tunable cation capacity, film forming properties, metal ions adsorption and diffusion [1]. In our laboratory we have studied the in-situ intercalation of different cation-exchanged inorganic fillers with Na+ in their channels and layers. The aim of the present work is to establish the correlation between membrane composition and properties such as the water uptake, ion exchange capacity and Na+ diffusion transport to optimize the fabrication of sustainable Na-air batteries.

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A facile approach to modify cellulose nanocrystal “CNC “with PTMAEMA for the adsorption of perfluoroalkyl substances "PFAS."

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Cellulose-based material is a sustainable alternative to polymers derived from petroleum. Cellulose nanocrystal belongs to this family and is commonly known for its important physical and chemical properties and ability to form a film.[1] Modifying CNC via electrostatic adsorption of cationic polymers is a facile and promising technique to enlarge the application of this material. Herein, we developed a membrane-based on blending negatively charged CNC and positively charged polymer (Poly (trimethyl aminoethyl methacrylate) “PTMAEMA”) and by using Whatman™ paper as a support. First poly (dimethyl aminoethyl methacrylate) was synthesized by polymerizing dimethyl aminoethyl methacrylate monomer using Reversible Addition-Fragmentation Chain Transfer (RAFT). Then the tertiary amine of the polymer was quaternized by metathesis using iodomethane to obtain PTMAEMA. The cationic polymer was characterized by NMR and size-exclusion chromatography. The membrane was then developed by depositing CNC and PTMAEMA consecutively on Whatman™ support by spin-coating. The interaction between CNC and PTMAEMA was investigated by quartz crystal microbalance with dissipation monitoring (QCM-D) and by measuring the particle size and ζ-potential of the mixture. Then the developed membrane was applied for the adsorption of Perfluorooctanoic acid "PFOA," a persistent pollutant with a toxic effect on human health and the ecosystem.[2]

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Progress and challenges on dicarboxymethyl cellulose adsorptive membranes

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The research on adsorptive membranes arises from a need for cost-effective and efficient water treatment technologies. This type of membrane focuses on the removal of trace pollutants, which hinges on both chemical composition and morphological structure of the membrane. Therefore, the incorporation of adsorbent materials with process-specific functional groups contributes to a more selective and reversible process. Biopolymers, such as cellulose-based polymers, are highly accessible and may overcome drawbacks related to the process cost [1]. Dicarboxymethyl cellulose (DCMC) is produced by heterogeneous etherification of cellulose with a β-halocarbonyl compound. This cellulose derivative has a wide pH working window due to its low pKa, making it suitable for adsorption of several cationic species (dyes, proteins, micropollutants). DCMC, when produced with a degree of substitution higher than 0.45, is water-soluble. This presents a challenge for the fabrication of membranes [2]. Preparation of DCMC membranes is in a preliminary stage. Several phase separation methods have been tested for the fabrication of DCMC membranes, namely solvent evaporation induced phase separation (EIPS) and non-solvent induced phase separation (NIPS). DCMC membranes were prepared via EIPS using N, N-dimethylformamide (DMF) and showed promising results. Experiments with Pickering emulsions comprising a DCMC solution, corn oil and graphene oxide were blade-casted and immersed in ethanol. However, a slow phase separation with this non-solvent may be responsible for the lack of pores in the resulting films. A cryogel was prepared by freeze-drying a mixture of DCMC and dialdehyde-DCMC (D-DCMC, 10%wt). In short, the novelty of this polymer presents great challenges and opportunities for the development of new DCMC-based materials [3].

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Esters of nature-identical engineered polysaccharides as new materials for packaging applications

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Polysaccharides are abundant, sustainable and versatile biopolymers with a long tradition in industrial applications. While numerous polysaccharides of varying structures are naturally available, the cost effective isolation of such materials at scale and consistent purity levels required for industrial applications remains often challenging. For this reason, the enzymatic polymerization of sucrose to generate nature-identical polysaccharides, with specific linkage control and thus of defined and desirable properties, has recently been extensively studied. One example of engineered polysaccharides is α-1,3-glucan, linear and semicrystalline polymer that containes α-linked glycosidic bonds as in starch. Such material is not directly melt-processable and is water-insoluble, as typical for highly aggregated polysaccharide materials, with direct consequence of limiting its use in different applications. Thus a similar approach used to convert cellulose to cellulose esters can be used in order to produce glucan ester derivatives, potentially suitable for packaging applications. In this work, the transport properties of different long-chain acid esters of α-1,3-glucan, namely Glucan Palmitate (GP) and Glucan Laurate Acetate (GLA) were analysed. The polymers were used to produce self-standing dense transparent films by solvent casting technique. Transport properties were analysed through sorption tests, while water sorption was studied through gravimetric analysis. This work is aming to promote the design approach learnt from systhetic polymers in order to obtain biopolymers with engineered properties, useful for separation applications.

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Toward a Catalytic DNA-Membrane Reactor

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One way to approximate the ingenuity of biological membranes while maintaining the robustness of artificial membranes is the use of hybrid materials. We have studied modular systems comprising functional-DNA and mesoporous anodized aluminum oxide (AAO). Functional nucleic acids are highly versatile molecular building blocks: thanks to their specific hybridization they enable systematically creating nanoscale structures; they may specifically recognize a wide variety of molecules; and they may even serve as biocatalysts [1]. Recently, DNA was shown to be capable of catalyzing a 1,3-dipolar reaction which does otherwise not exist in living systems [2]. However, commonly the applications of functional nucleic acids are limited to aqueous environments, because molecular organic solvents widely annihilate their function or their thermal or conformational stability. In this study we made use of the intrinsic property of DNA-modified membranes of creating a reactive interface between compartments of different chemical composition, similarly to what biological membranes do in a far more ingenious manner under physiological conditions. For this purpose, AAO membranes were functionalized with DNA whose chirality can be transferred directly to a metal-catalyzed reaction, and in particular a copper(II)-catalyzed Diels– Alder reaction. In this way, we created a prototype of what might be denominated a DNA-membrane reactor. We studied the immobilization efficiency and functional stability of DNA under prolonged operation of the reactor and compared the results with homogenous DNA-catalysis. In particular, we focused on how non-aqueous solvents may enhance product recovery but maintain the function and stability of the membrane-based DNA. The results suggest that we can use versatile functional molecules such as nucleic acids beyond physiological conditions and in a DNA-membrane reactor which offers mechanical robustness and the specific enzymatic activity of DNA.

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Membrane modification methods for high performance enzymatic membrane reactors

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Enzymatic membrane reactors (EMR) enable integration of enzymatic catalysis and membrane separation and play a big role in the new generation of highly efficient downstream bioprocess in which enzyme reactions are involved. In-situ product separation in EMRs contributes to avoid side reactions that have a negative impact on the product quality. EMRs have been studied in various applications, being particularly interesting the ones related to production of biomolecules (e.g., oligosaccharides, organic acid and methanol). In EMRs membranes are used as supports for enzyme immobilization that allow for reuse of enzymes improving in this manner the overall productivity of the process. However, there are several challenges that limit their upscale and use in commercialization of bioproducts. One of them is the limited surface area of the membrane, that hampers high enzyme loading which leads to low immobilized activity. Large substrates (e.g., polysaccharides) can also have problems accessing the active sites of the immobilized enzymes which in turn decreases the activity. Moreover, enzyme denaturation during the immobilization also causes deactivation of the enzymes. In this work, we aim to find suitable solutions to these challenges by defining novel enzyme immobilization strategies to retain high immobilized enzyme activity and improve the catalytic performance of EMRs. Fibrous membranes (fabricated by electrospinning) were used as the supports for immobilizing enzymes, as their chemical and physical properties can be more easily tailored. Surface modification (i.e., polydopamine, tannic acid, glutaraldehyde and polyelectrolytes) was carried out to introduce functional groups and control the charge density on the membrane surface, thus the enzymes can attach to the membrane via covalent/non-covalent binding. The results of the enzyme activity tests suggested that the porous structure of the fibrous membrane increased the enzyme loading and reduced the steric hindrance of the substrate transfer. On the polyelectrolytes modified membrane surface, enzymes were immobilized by electrostatic attractions, which had a low impact on the native structure of the enzymes, therefore, the immobilized enzymes exhibited high activity and good stability on the membrane surface.

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Functionalised food packaging films: Greener routes to synthesise and exploit novel agro-based hydrophobic deep eutectic solvents

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Hydrophobic deep eutectic solvents (DESs) are an emerging class of green solvents, which are economic, tunable and environmentally friendly [1]. Three different approaches to synthesise agro-based hydrophobic functional DESs by sonication, microwave-assistance and rotavapor techniques have been explored in this study, in addition to the conventional heating method. For the first time, alcohol-based hydrophobic DESs consisting of thymol and alcohols (hexadecanol, vanillyl alcohol and β–sitosterol) of varying individual melting points have been synthesised, for a range of molar compositions. Along with detailed physicochemical, thermal and rheological characterization of the DESs synthesised, an EcoScale score of > 95% re-establishes agro-based DESs as green solvents for its pioneering usage in membranes for food packaging. Hydrophobic DES-in-water nanoemulsions, where the aqueous phase comprises pectin (biopolymer), glycerol (plasticiser) and Tween 80 (surfactant), were produced by Ultra-Turrax homogenisation (15 min, 10000 rpm) and ultrasound emulsification (750 W, 10 min, 1s pulse on/off). The nanoemulsions obtained were compared to those produced with a more sustainable technique: membrane emulsification [2], (continuous phase cross-flow velocity 0.32 m/s, dispersed phase flow-rate 0.05 ml/min). All the DES-based systems were able to formulate nanoemulsions with sizes <200 nm and polydispersity index <0.3. Subsequently, the optimised DES-in-water nanoemulsions were cast at a controlled temperature of 40 °C to form flat-sheet membranes. From the application perspective, the antioxidant properties of the synthesized DESs, their individual components and the composite membranes were tested by Ferric Reduction Antioxidant Power method. Synergetic effects were witnessed as the antioxidant properties were enhanced in all the DES-based systems tested. Additionally, the membranes were characterised in terms of optical and mechanical properties, surface hydrophobicity, and barrier properties against water vapour, gases (CO2, O2, N2) and ultraviolet-visible (UV-vis) radiation. The ongoing studies firmly suggest promising applications of these membranes as packaging materials for food products with low water content.

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Long-term stability of cellulose membranes used in the treatment of spent Deep Eutectic Solvent for solvent purification and lignin recovery

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Deep Eutectic Solvents (DESs) have drawn attention as ecofriendly solvents for efficient lignin recovery from biomass. DESs demonstrate many advantages among which are nontoxicity, biocompatibility, biodegradability and almost negligible vapor pressure. Advantageous qualities of DESs stem from the combination of Hydrogen Bond Donor (HBD) and Hydrogen Bond Acceptor (HBA). Hydrogen bonding between them leads to lattice energy depression and eutectic point formation. Choline chloride based DESs are usually used in biomass treatment. Despite all the advantages of DES, scaling of DES treatment processes to industrial level is still very expensive. One of the possible solutions to reduce these costs could be development of DES purification and recycling process. In our previous work we proved that filtration with cellulosic membranes can be an effective solution to DES (Choline Chloride: Lactic Acid 1:10 mole ratio) purification and lignin fractionation. We found out that the combination of the commercial membranes – RC70PP by Alfa Laval and 5kD Disc by Millipore – lead to removal of 95% water insoluble compounds. [1] In this research we focus on the long-term stability of the previously studied membranes (RC70PP and 5kD Disc) in spent DES solution at increased temperature (45 °C). The possible changes in filtration capacity and retention properties, which are induced to the membranes due to the exposure of them to the spent DES, will be discussed. We also compare the characteristics of the membranes before and after the exposure to the spent DES.

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Fractionation of biologically active compounds extracted from onion skin bio-waste

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Biologically active compounds (BACs), such as polyphenols, have become an intense focus of research interest because of their perceived health-beneficial effects. They occur in a variety of fruits, vegetables, nuts, seeds, flowers, bark, and bio-waste. They have been reported to exhibit anti-carcinogenic, anti- atherogenic, anti-ulcer, anti-thrombotic, anti-inflammatory, immunomodulating, anti- microbial, vasodilatory, and analgesic effects [1]. Ultrasound-assisted extraction (UAE) of BACs possesses many advantages over conventional solid–liquid extractions in terms of higher yield, shorter extraction time and preserved biological activity of the extracted compounds Being also green and economically viable, the UAE technique has a great potential for application in the plant-based chemistry. Traditional approaches, including simple steam distillation and vacuum distillation, are adopted to BACs fractions. Generally, these methods require an increased temperature and high energy consumption. The first is inappropriate for heat-sensitive products. These methods may also result in a loss of compounds of low molecular weight, which can be removed together with the solvent during evaporation [2]. The aims of the present investigation are: (i) fractionation of BACs extracted from skin onion bio-waste using 60% ethanol and UAE by means of nanofiltration, and (ii) determination of anti-proliferative properties of the fractions after nanofiltrations.

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An Eco-Friendly Integrated Membrane Process for the Concentration and Purification of Functional Bioactive Compounds from Citrus Hybrid Tacle® Extract

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Tacle is a tangerine-like citrus obtained from the crossbreeding of Clementine and Tarocco cultivars. The extract of this fruit is rich in polyphenol glycosides, mainly naringin and hesperidin, and other bioactive phytochemicals typical of Citrus fruits (such as carotenoids). These compounds are well recognized for their protective activity against oxidative agents involved in several degenerative disorders [1-3]. Developing sustainable and effective purification procedures of bioactive compounds from vegetable sources has always been a challenging task for scientists. In this work, an eco-friendly process based on a combination of pressure-driven membrane operations and osmotic distillation has been investigated, for the first time, to purify and concentrate functional biomolecules in tacle extracts. In the first step, a concentrated stream rich in carotenoids was obtained by using hollow-fiber ultrafiltration (UF) membranes according to a batch concentration-diafiltration mode. The selected membranes showed a retention towards β-carotene higher than 98% and lower than 5% for polyphenols and sugars. In the second step, nanofiltration (NF) membranes in spiral-wound configuration were tested in order to produce concentrated fractions enriched in flavonoids and depleted in sugars. Membranes with molecular weight cut-off (MWCO) of 300-500 Da showed a sugar removal efficiency from the clarified extract (permeate UF+diafiltrate) of about 65%; on the other hand, more than 85% of flavonoids were rejected. A further concentration of the NF retentate was achieved by a final step of osmotic distillation (OD). According to the investigated process, three valuable and innovative fractions were obtained: i) a UF retentate fraction rich in carotenoids useful for cosmeceutical formulations, food supplements or as natural colorant; ii) a NF retentate rich in flavonoids of interest for pharmaceutical applications due to its anticholesterolemic activity; iii) a purified fraction enriched in sugars and salts suitable for drinking formulations.

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Reuse of permeate stream from the dewatering of microalgae biomass using ceramic membrane filtration

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As the microalgae market grows, efforts should be devoted to reduce the water footprint of algal biomass cultivation and harvesting. The goal of this study is to advance the implementation of ceramic membrane filtration for effective microalgae dewatering with the additional goal of reusing the permeate as new cultivation medium. To this purpose, different algal strains were initially tested in bench-scale experiments by filtering suspensions with four ceramic membranes having nominal pore sizes in the UF/MF range. Similar near steady-state fluxes (70-120 LMH) were observed when the nominal pore size was above 15 kDa, suggesting the dominance of cake layer filtration independently from the initial flux. Complete algae cells rejections were observed, together with adequate nutrients passage and achievement of high water recovery rates (hence, biomass concentration factors). The combination of these results highlights the effectiveness of the investigated concentration/separation process and the potential for water reuse in subsequent microalgae growth cycles. When the permeate streams were used as media for algae growth in bench-scale bioreactors, Chlorella s, Scenedesmus o., and Galdieria p. showed promising results, approaching the growth rate observed in their ideal fresh media. On the contrary, no or little growth was observed for Spirulina p.. Dewatering in a pilot-scale ceramic membrane system and subsequent cultivation in a pilot-scale photobioreactor confirmed the potential for scaling up this water reuse strategy with both Galdieria p. and Scenedesmus o.. Additional efforts are being devoted to understand the change in composition of the reused water after different cycles of dewatering and reuse, with special focus on nutrients and algal organic matter, thus providing insight into the feasibility and best operational conditions of a continuous MF-photobioreactor system performing dewatering and reuse.

Recycling of End-of-Life membrane: Fouling resistance NF Multilayer polyelectrolyte membrane as a new platform in pulp and paper industries

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Membrane technologies underpin modern biorefinery's core activities and are fundamental drivers in exploiting the full potential of lignocellulosic biomass via creating and separating diverse high-value products across the wood-based biorefineries. However, due to the complexity of such biorefineries' streams, membrane fouling is still one of the main bottlenecks [1], diminishing the flourishing of its wider applicability in pulp and paper industries. The study unravels new insights on the manifestation of using End-of-Life membranes (EOL) modified with layer by layer (LBL) deposition technique in pulp and paper industries. Herein, to fully implement the European Directive guideline on sustainable waste management [2], the main effort is devoted to developing antifouling membranes with unprecedented performance in the treatment of pressurized hot water wood extract (PHWE). Using two strong polyelectrolytes (PDADMAC and PSS), different types of multilayer polyelectrolye membranes (MPMs) were fabricated, and their performance was compared with a commercial UF membrane (UH004P, 4kDa, Microdyn-Nadir) and an unmodified recycled EOL membrane. The current work demonstrated that possessing an ultrathin bilayer of polyelectrolyte can substantially enhance the antifouling characteristics of the recycled membranes, where only 3–7% of pure water flux decline was observed after PHWE filtration, compared to 40–60% with the UH004P membrane. Moreover, under the same operating conditions, MPMs reveal higher filtration capacity (Filtration up to 50% volume reduction (VR)) compared to UH004P (12.5% VR) and unmodified EOL-membrane (27.5%VR). In the best-case scenario at the same VR, MPMs revealed 92%, 91%, and 53% rejection of total dissolved organic carbon, Brix, and conductivity, which was higher by 1.3, 1.2, and 10.6 times compared to the UH004 membrane, respectively. This study contributes by establishing, for the first time, the basis for biorefinery industries underlying that LBL techniques can be adopted to develop an antifouling membrane with an eco-friendly treatment method in the reusability of EOL membranes.

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Biosensors development by tuning *in situ* gold nanoparticles formation in PDMS matrix

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AuNPs incorporation into the PDMS matrix allows the development of localized biosensors by converting chemical or biomolecular information into a physically readable signal [1]. Generally, AuNPs-PDMS system is prepared by immersing a sheet of polymerized PDMS in an aqueous solution of chloroauric acid ($HAuCl_4$), however, the insufficiency and non-uniform distribution of AuNPs in the PDMS matrix led to poor conductivity of PDMS [2,3]. In this work, the swelling properties of PDMS membranes in different solvents (ethanol and ethyl acetate) were exploited to improve the amount and the uniformity of AuNPs within the polymer matrix. The swollen polymer allows a higher permeation rate of the gold precursor into the film, thus improving the reduction reaction. In addition, the behavior of the swelling solvents on the films obtained using different ratios of the two polymer components (base polymer and curing agent) and the effect of the AuNPs and the swelling step on the transport properties of AuNPs-PDMS membranes were evaluated. Results evidenced that the reaction without the use of swelling solvents prior to incubation in the aqueous solution of the gold precursor was much slower and the color of the final sample is lighter (after 24 hours), showing a smaller amount of Au nanoparticles. Instead, using swollen PDMS already after only 2 hours of incubation, highly colored samples are obtained. In addition, a larger amount of curing agent than the polymer base also increased the reduction rate of the gold precursor. The swelling of the polymer in ethyl acetate allowed the formation of a more uniform distribution of gold nanoparticles in the AuNP-PDMS composite. This result may be due to the higher swelling ratio of PDMS in ethyl acetate compared to ethanol (1.04 versus 1.18). The plasmonic signal of the prepared films was confirmed by uv-vis measurements with an absorption peak at 536 nm.

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The role of electromembranes in the recovery of energy

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Since the first applications of charged membranes focused on industrial water desalination through electrodialysis and electrolysis processes, nowadays, charged membranes have expanded their application in different fields such as new energy conversion and storage systems (redox flow batteries, reverse electrodialysis, membrane capacitive deionization, microbial fuel cells and ion exchange bioreactors) as well as efficient desalination and wastewater treatment processes.

Reverse electrodialysis, RED, is a membrane process that builds upon the electrodialysis, ED, process. Similar, to ED, RED uses an array of cation and anion exchange membranes to transport ions. The flow of ions generates an electric current that is captured by electrodes. The compartments between the membranes are alternately filled with a high salinity water and a low salinity water. The salinity gradient difference is the driving force in transporting ions that results in an electric potential, which is then converted to electricity. RED technology has known interesting works at lab and pilot scale aimed at transforming into electricity the saline gradient energy generated between different water sources, e.g., sea water-lake water, brines-brackish water etc, that have contributed to increase the Technology Readiness Level. Besides, the progress made on the mathematical description of the phenomena that take place during the energy recovery facilitates the decision-making process on the selection of the optimum location site and conditions for implementation of the technology.

Here, two promising case studies will be presented where integration of the recovery of the saline gradient energy using electromembranes in industrial processes, i.e., seawater desalination plants and wastewater treatment plants, contributes to increase the energy sustainability and reduce the carbon footprint of these industrial processes.

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Effect of particle size on the desalination performance and flowing properties of activated carbon flow electrodes in capacitive deionization

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With the increasing global water shortage danger, intensive research activities are directed toward advanced wastewater remediation and brackish water desalination technologies. Flow-electrode capacitive deionization (FCDI) is an advanced electro-chemical ion removal technology. It combines ion-exchange membranes and flow electrodes. This technology has been actively explored over the last decade, driven by the advantage of energy-efficiency supported by an energy recovery process, sustainability, and continuity in the production of freshwater [1]. FCDI performance can be improved mainly by improving the flow-electrode conductivity via enhanced charge transfer. The key to this lies in the improvement of the carbon materials in use. Therefore, the performance of carbon flow electrodes in FCDI system is related to several parameters such as particle size, viscosity, carbon loading, stability, specific surface area, hydrophilicity, etc. Here, we adopted a new approach that consists of controlling the particle size distribution of commercial activated carbon (AC) used in FCDI flow electrodes by dry milling method. Particle size of commercial AC was reduced after ball milling; this was confirmed by scanning electron microscopy (SEM) and particle analyzer. Then, the effect of particle size distribution on the rheological behavior, physical stability and desalination performances of AC flow electrodes was investigated. The rheological properties and flowability are critical parameters in flow electrodes based capacitive technologies.1 It is essential to ensure that there is no clogging while carbon slurry flows along narrow channels in the FCDI cell [2]. It was found that carbon suspensions of fine activated carbon (FAC) particles have better conductivity enhancing salt removal, but this at the expense of high viscosity, thus requiring a high amount of energy for pumping [3]. For this reason, the effect of particle size distribution on the viscosity and desalination performance of the flow electrodes was further investigated. Different AC slurries were prepared from two populations of different particle size: low size range (0.65-0.92 µm) and large size range (1.5-2.3 µm). Monomodal and bimodal mixtures with different fine particles to large particles ratio were chosen for the experiment. The results of this study showed that the fine to large particles ratio largely influenced the desalination capacity and the rheological properties of AC water slurries used as electrodes in FCDI.

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A new process for resource recovery from surface water RO brines for permeate remineralization

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Low-Pressure Reverse Osmosis (LPRO) membranes could be valuable alternatives to conventional surface water potabilization processes [1] thanks to a combined ability to: remove micropollutants, soften, disinfect and remove organic substances. However, these processes produce an aggressive permeate and a concentrated brine with discharge constraints [2]. Assisted Reverse Electrodialysis (A-RED, [3]) was tested for LPRO permeate remineralization by recovery of target minerals from the corresponding LPRO brine while maintaining product water integrity with respect to the organic compounds present in the concentrate. Bench-scale experiments were carried out with SUEZ-WTS membranes (CR67T-AR204T), operating under equal permeate and brine flows (2 cm/s velocities). The process was first tested using synthetic salt solutions, with varying applied voltage, permeate and brine inlet conductivity and brine composition to study the selective transport of individual species. Tests were then performed with Seine River LPRO brine spiked with 17 micropollutants to investigate membrane retention capacity. Results showed that ion transport numbers and permselectivity were weakly influenced by inlet solution conductivity and applied current/voltage, while permselectivity was mainly influenced by brine composition. High passage of salts was observed, allowing for a significant increase of permeate mineral content from ≈20 mg/L $CaCO_3$ up to ≈490 mg/L $CaCO_3$ and from ≈120 µS/cm up to 1284 µS/cm for hardness and conductivity, respectively. Spiked brine runs highlighted low micropollutant passage with over 98% rejection for 15 out of 17 compounds while natural organic matter breakthrough averaged 2% (0.2 mg C/L). Following these positive results a pilot unit was set-up with two 70 cell-pair stacks in series, fed with LPRO permeate and brine at nominal flow rates of 3.6 m^3/h. Preliminary results have confirmed process feasibility while long-term performance stability evaluation is ongoing. Overall, results of this study highlighted the viability of the proposed process for LPRO permeate remineralization to target potable levels.

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Application of Bipolar Membrane Electrodialysis in Skim Milk Acidification

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Membrane and electromembrane processes have been used for the processing of food products and ingredients. Bipolar membrane electrodialysis (EDBM) has been shown to cause acidification of skim milk [1] which can be required in dairy processing. A bipolar membrane (BM) consists of a cation exchange layer and an anion exchange layer which are joined together in series. Its prominent feature, electric field enhanced (EFE) water splitting stems from this particular arrangement. The EFE water splitting starts at the bipolar junction when a sufficient DC current is directed through the bipolar junction under reverse polarization [2]. With a particular arrangement of the membranes in the stack, the described phenomena can be used in EDBM to increase the H_3O+ or OH- concentrations in the compartments next to BM. Recently, EDBM has been used for isoelectric precipitation of skim milk proteins, during casein or caseinates production [3] but limited work exists regarding the acidification process to lower pH than the isoelectric point. This work aims to present the possibilities of EDBM for acidification and partial removal of calcium phosphates ($Cax(HyPO_4)z$) from skim milk. Physicochemical properties of the feed and product were analyzed, namely, mineral profile, pH, conductivity, current, and membrane properties. The pH dynamic of skim milk decreased from 6.9 to 5.5, as well as titrable acidity increased from 25% to 40% for different samples. Furthermore, the scaling and fouling of bipolar and cation exchange membranes by calcium-containing salts and proteins were analyzed.

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From hollow fibre to flat sheet – How to develop membranes with similar properties and different shapes

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A brief comparison of hollow fibre modules and spiral wound flat sheet modules shows that both technologies have low investment costs and high maintenance costs due to high fouling tendency and poor cleaning possibility. So why do hollow fibres not have a higher market penetration considering their 10-fold higher packing density? Amongst other things, hollow fibre applications need additional feed conditioning to prevent blocking and they exhibit comparatively poor heat recovery. Both types of module design are applied in membrane distillation (MD). MD in turn is usually coupled with other processes as it makes do with waste heat. In our case, a reverse electrodialysis (RED) pilot plant employs a solar powered MD step that takes effluent from a mining operation to produce clean water and a concentrated brine that feeds the RED process. In order to meet the harsh requirements of the aforementioned process, the MD unit preferably employs flat sheet membranes in multi-envelope spiral-wound modules over hollow fibre modules. Therefore, the properties of proven lab-scale PVDF hollow fibres are being transferred onto PVDF flat sheet membranes, in order that the membranes have similar properties that result in comparable membrane process parameters (e.g. retention, permeation rate) and only differ in shape. All relevant process parameters of the dry-jet wet-spinning process are identified and translated into those of a vapour-assisted non-solvent induced phase separation process. A DoE approach ensures an efficient technology transfer from the lab scale hollow fibre production to a large scale flat sheet membrane production.

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Application of bipolar membrane electrodialysis for sodium hydroxide production from saltwork brines

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Electrodialysis with bipolar membranes (EDBM) has drawn attention motivated by their application in valorizing brines for the conversion of salts into acids and bases. Due to the water splitting occurring at the junction of the bipolar membranes (BPMs), and when treating a NaCl brine, H^+ and OH^- exit the junction of the BPMs forming acid (HCl) and base (NaOH) at the opposite side of the channels. However, the concentration of the products (i.e., NaOH) may limit the direct applicability of this technology, since a very diluted stream would demand too much pumping and impractical storage [1]. The world annual consumption of NaOH in 2015 was reported to be 82 million of tons [2]. In consequence, due to its economic and industrial importance, efforts must be focused on searching for a more productive conversion of salt (NaCl) into NaOH using EDBM. In this regard, this work presents a study of the generation of NaOH from saltwork brines free of hardness (Ca^{2+} and Mg^{2+}), thus avoiding scaling on the membranes. Experiments were carried out using an EDBM cell in batch configuration working at constant current (300 A/m^2) and testing commercially available membranes (Eurodia). The evolution of NaOH concentration was monitored, while parameters such as specific energy consumption and current efficiency were utilized to evaluate the EDBM performance. With the aim to achieve the most concentrated NaOH, different volume ratios in the base (VNaOH) and salt compartment (VNaCl) were examinated. Moreover, the ion-exchange capacity (IEC) of the membranes was determined by using the back titration method. Results suggested a significant change in the concentration of NaOH by means of varying the volume ratio of Vbase:Vsalt. Additionaly, the IEC determination lead to a discussion of the intrinsic degradation of the membranes and how the transport properties of the membranes were modified by this effect.

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Anion exchange membranes based on block copolymers for use in alkaline electrolysers

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Anion exchange membranes based on polymer backbone of block copolymer PSEBS (polystyrene-block-poly(ethylene-ran-butylene)-block-polystyrene) were synthesized, chloromethylated by „indirect method", cast on Teflon plate and subsequently reacted with amine 1,4-diazabicyclo[2.2.2]octane (DABCO). Obtained membranes show high hydroxide conductivity up to 7.5 S m^{-1} at 70 °C. Membranes show high stability in concentrated KOH. Same polymer was used as a precursor for binder of catalyst, where it showed higher current densities that PFTE. Prepared membranes were tested in alkaline membrane water electrolyser (AMWE) with current densities over 1 A cm^{-2} at 2V and 50 °C in diluted (0.1M) KOH.

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GREEN BRINE: Agri-food brine valorisation through integration of sustainable membrane-based technologies

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Table olive processing wastewaters and brines in particular are very complex and voluminous liquid matrices due to their high content in organic matter and salinity, which makes them a serious environmental concern with a very difficult and expensive management. Nowadays, their treatment requires specially designed systems because conventional ones present in wastewater treatment plants (WWTPs) are not prepared to deal with such a complex waste matrix, but they require large impermeable surfaces as evaporation ponds, thus causing gaseous odorous emissions, polluting soils and aquifers and prolifering the occurrence of insects. This worrying outlook leads to seek sustainable and environmentally-friendly treatments to those mentioned before, thus resulting in minimising emissions and discharges and, as a consequence, reducing the environmental impact of agri-food activities. The main objective of the GREEN BRINE project is to develop an alternative and more sustainable approach to valorisiting table olive processing wastewaters and, in particular, brines. So, the integral valorisation of agri-food brines through the integration of sustainable systems such as low-cost ceramic membrane technology (based on clayey materials) and membrane-based bioelectrochemical systems could be a successful case study of technological application based on the concept of circular economy and is being investigated with the purpose of obtaining bioactive compounds, water purification and in situ generated products of industrial interest, such as energy carriers, acids and bases.

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Development of a Polymeric Diaphragm for Alkaline Water Electrolysis

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Alkaline water electrolysis (AWE) is one of the most studied and efficient methods for hydrogen production. One of the key components of this technology is certainly the separator. The separators used in the past were made of asbestos nets, which are hazardous to health. The current ones are not dangerous to human beings but have ohmic resistances that can be improved to increase the efficiency of electrochemical cell. The development of an advanced separator with high wettability, low ohmic resistance and long life-time is essential for the performance and safety of such apparatus. A new polymeric separator containing an inorganic filler is presented in this study. The separator is made of a high temperature and chemical resistant techno-polymer produced by a non-solvent induced phase separation (NIPS). This technique is flexible and allows the tuning of the type of coagulation bath and the time of immersion to optimize the performance of the separators. In addition, the most appropriate support for the separator was investigated and selected. The characterization of the product separators was done in terms of: surface morphology (SEM), porosity, thickness, KOH uptake, ohmic resistance and ionic conductivity. The results showed that the ohmic resistance and the ionic conductivity of the synthesized separators were significantly improved compared to those of competitors. The next step after this study will be to industrialize the process developed on a laboratory scale in order to obtain a product that will be more cost-effective and environmentally friendly.

Covalent Organic Framework-Based Mixed Matrix Membranes for CO_2 Separation

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Covalent Organic Frameworks (COFs) are porous crystalline materials conformed by purely light elements linked by strong covalent bonds, which provide them a high framework stability, low density, permanent porosity, and high thermal stability. These materials have emerged as a molecular platform for designing organic materials with promising characteristics for a wide range of applications, such as gas storage and gas separation, where the high surface area and tunable nature of COFs can play an important role [1-2]. In this regard, its utilization as filler in the so-called Mixed Matrix Membranes (MMMs) has the potential to combine the versatility and easy processability of conventional polymers with the high CO_2 affinity of COF RIO-13. In this work, elastomeric Pebax®1657 was selected as polymeric matrix to prepare MMMs with different RIO-13 loadings (5, 10 and 20wt.%). The membranes were prepared following a modified "green" phase inversion method using water and ethanol as solvents, thus eliminating the use of harsh solvents commonly used to prepare MMMs. The prepared MMMs showed a dense and defect-free morphology with good compatibility and dispersion of the filler in the polymeric matrix. Moreover, the incorporation of RIO-13 provided MMMs with a higher thermal stability, compared to the neat Pebax®1657 membrane. Higher CO_2 permeabilities were achieved with increasing content of COF RIO-13. It was also possible to analyze the influence of temperature and pressure in the MMMs' separation performance, under mixed gas conditions. Furthermore, it was observed that, compared to a Pebax®1657-based MMM containing ZIF-8, it was possible to achieve a higher CO_2 permeability at the same loading (10 wt.%). This finding unveils the possibility of preparing CO_2-selective MMMs using completely organic materials as fillers, such as RIO-13, decreasing the manufacturing cost of the membranes and improving the compatibility between the materials.

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Semi-Interpenetrating Poly(ionic liquid) Networks: Towards the Development of Mixed Matrix Iongel Membranes for CO_2 Separation

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Semi-interpenetrating polymer networks (semi-IPNs) are a unique class of polymeric materials that combine polymer networks and linear (or branched) polymers, which are partially interlaced on a molecular scale. This approach allows for the development of polymeric materials with diverse and tailored properties. In the context of gas separation, semi-IPNs can be developed to tailor the thermal, mechanical and gas transport properties of polymeric membranes. In this work, a polymer network, poly(ethylene glycol) diacrylate (PEGDA), was combined, for the first time, with a pyrrolidinium-based poly(ionic liquid), bearing the fluorinated [TFSI]- anion in its structure, to create a semi-IPN. A high content of ionic liquid (80 wt% of 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide [C2mim][TFSI]) was blended with the semi-IPN to develop iongel membranes to be tested for CO_2 separation. The high CO_2 affinity provided by the [TFSI]- anion present in both the IL and semi-IPN allows for the development of iongel membranes with improved separation performances. Moreover, in this study, materials with different nature were studied as potential fillers to develop mixed matrix iongel membranes based on semi-IPNs. Inorganic nanoclays (MMT), inorganic/organic MOFs (Cu(BTC) and MIL-53) and azo-porous organic polymers (azo-POP-1 and azo-POP-11) were selected and their CO_2/N_2 and CO_2/CH_4 separation performance was assessed. Since Cu(BTC) and MIL-53 offered the best compromise between permeability and ideal selectivity among all studied iongels, their separation performance under experimental conditions that mimic flue gas (CO_2/N_2) or biogas upgrading (CO_2/CH_4) streams were also evaluated. The temperature and presence of water vapor were found to be important parameters influencing the performance of mixed matrix iongel membranes, by significantly improving permeability, at the expense of CO_2/N_2 selectivity. On the other hand, under the mild experimental conditions of the biogas mixture, the prepared mixed matrix iongel membranes showed a separation performance similar to the one obtained under single-gas ideal conditions.

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Microstructured PVDF-based microporous layer for polymer electrolyte fuel cells by phase inversion technique and Cyrene as a green solvent

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In low temperature polymer electrolyte fuel cells (PEMFC), gas diffusion layers (GDLs) play an important role to distribute the reactants over the catalytic layers and to provide the electrical conductivity to transfer the electrons to the flow field current collectors. A GDL is usually composed of a microporous layer (MPL) over a supporting carbon substrate. Moreover, the MPL due to its hydrophobic character enables an easier liquid water expulsion. Usually, expensive polytetrafluoroethylene (PTFE) is used as hydrophobic agent which is unhomogeneously distributed over the MPL with an uncontrolled porous structure. In our previous studies [1, 2] we have proposed polyvinylidene difluoride (PVDF) as hydropobic binder with a more accurate control of the microporous structure of the MPL by adopting the nonsolvent induced phase inversion technique (NIPS) which is well-known for the preparation of porous polymeric membranes. Organic solvents, such as N,N-dimethyl formamide (DMF), or N-methyl pyrrolidone (NMP) have been extensively studied for membrane synthesis and MPL preparation. Unfortunately, these are all toxic and fossil fuel derived organic solvents. A recent interest on the use of green solvents for membrane preparation is arising [3]. In this work, dihydrolevoglucosenone (commercially known as Cyrene) was employed as a green solvent to dissolve PVDF. Then the polymer solution was mixed with Carbon black (CB) as electroconductive filler and the microstructured MPL was precipitated into water as non solvent. The influence of polymer/filler ratio and coagulation bath conditions on the characteristics of the MPLs was thoroughly analyzed. The experimental data were discussed with respect to the ternary phase diagram, contact angle, roughness, morphology by optical and electron microscopy, porosity, pore size distribution by liquid-liquid displacement porometry, in-plane and through-plane electrical conductivity and through-plane air permeability. The effectiveness of the MPLs was proved in a PEMFC fed with hydrogen and air at room temperature.

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Bactericide Effect of Silver Nanoparticles and Silver Ion-Exchanged Zeolites on Composite Cellulose Acetate Nanofiltration Membranes

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Membrane fouling represents the major limiting factor in the large-scale implementation of membrane processes for water treatment. Biofouling contributes to more than 45% of all membrane fouling and occurs due to deposition, accumulation, growth, and metabolism of microorganisms on the membrane surface. Currently exists intense research for biofouling control with membranes functionalized with nanomaterials. Silver nanoparticles (AgNP) are amongst these nanomaterials, but it is still not clear the mechanism of action of AgNP. To help elucidate, two nanofiltration membrane systems, one providing readily available silver ions and another containing AgNP, were tested against Escherichia coli (EC). Cellulose acetate membranes with polyvinylpyrrolidone (PVP) coated AgNP, silver ion-exchanged β-zeolite and β-zeolite were casted by the phase inversion technique and subjected to annealing. The synthesized membranes were characterized in terms of hydraulic permeability (Lp) and rejection to neutral solutes and mono- and bivalent salts, and the antibacterial property of the membranes was evaluated against EC by the plate count method. The incorporation of AgNP produces a threefold increase in the membrane Lp when compared to the silver-free membranes and the incorporation of silver ion loaded zeolite resulted in a 56.3% increase in Lp. The results demonstrated that the synthesized membranes had high rejection coefficients to neutral solutes and to bivalent salts which are associated to nanofiltration membranes with enhanced Lp. The data from the bactericidal experiment showed that the silver ion-exchanged β-zeolite membrane was effective in inactivating EC after just 210 min of contact time. No bacterial activity was detected following 24 h of contact time with the membrane containing polyvinylpyrrolidone coated silver nanoparticles. Different patterns of bactericide activity are associated to the silver speciation in metallic or ionic form. The high flux nanofiltration membranes with bactericidal properties represent a strong asset in water treatment biofouling control.

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Up-grade mixed matrix hollow fiber membrane fabrication system: accurate production control and data management

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Large-scale separation applications demand highly productive membranes. Therefore, commercial separation membranes are processed into a highly elaborated structure: an extremely thin dense top layer (100 – 200 nm) supported by a porous layer. Preferentially, this structure is processed in the form of a hollow fiber (HF) because fibers can be densely packed into separation modules (over 10000 m^2/m^3 membrane area per module volume) and increase furthermore the productivity. The introduction of well-defined nanostructured porous fillers with pores close to the kinetic diameter of the gas molecule into polymer matrix potentially combine the advantages in separation performances of filler particles with the processability of polymers [1]. The concept of dual layer HF membranes applied for MMMs aims to create a mixed matrix selective top layer and at the same time minimize the quantity of filler by adding this component only in the outer polymer solution while the inner polymer solution is filler free. Fabrication of asymmetric HF membranes is done by dry followed by wet phase inversion method and the top layer and the support are formed simultaneously. There are multiple parameters that influence this process. Small fluctuations in each parameter can have a strong effect over final fiber gas permeation properties. Therefore, process parameter precise control is essential when experimental campaigns are conducted for the elaboration of a new spinning recipe. Also, in line monitoring and registration of all spinning parameters with the objective to automatically correlate fabrication parameter with membrane separation properties in a sequent step via a customized software will allow easy and in deep data analysis and interpretation of the results. Within INNOMEM project, Tecnalia´s spinning line has been up-graded in terms of production capacity and improved control over process parameters. The up-grade will be demonstrated with a a dual layer mixed matrix hollo fiber membrane.

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Ceramic membranes activation via Piranha reagent– a facile way for significant enhancement in membrane performance

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Various silane-based modifiers were selected to functionalize titania ceramic membranes, i.e. three fluorinated molecules: 1H,1H,2H,2H-perfluorooctyltrichlorosilane - FC_6Cl_3, heptadecafluoro-1,1,2,2-tetrahydrodecyl)methyldichlorosilane - FC_8Cl_2Me, heptadecafluoro-1,1,2,2-tetrahydrodecyl)dimethyl-chlorosilane - FC_8ClMe_3, and two types of non-fluorinated (n-octyltrichlorosilane - C_6Cl_3 and trichloro(octadecyl)silane - $C_{16}Cl_3$). It was evaluated how these modifiers can influence the features of the material and then transport and separation properties. The additional aim of the work was to activate the ceramic material using piranha reagent which resulted in the generation of additional reactive hydroxyl (OH) groups and enhanced the efficiency of the modification process. It was important to compare pristine and activated materials and evaluate the impact of the activation on the grafting process. Finally, the modified membranes were tested in vacuum membrane distillation (VMD) for VOCs (metyl-tert-butyl ether and ethyl acetate) removal from water. In the VMD process, all membranes were selective towards VOCs. The comprehensive material studies, including evaluation of morphological, wetting, surface charge, and structural properties, were performed and referred to the membrane performance in membrane distillation. The activation process significantly improved membrane functionalization owing to the high level of OH groups which was confirmed by the water contact angle reduction from 60.2° for pristine material to 6.4° for the 15-minutes activated ceramic. The hydrophilization of the starting materials was also verified with the zeta potential measurements. The improvement in the grafting efficiency for material which underwent the activation was in the range of 6%-20%. The most significant change was found for the treatment with FC8Cl2Me molecules. The reason for such enhancement was related to the mechanism of molecule attachment.

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Visible Light Responsive Nanofiltration Membranes Development to Mitigate Biofouling in Water and Wastewater Treatment

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Biofouling is one of the major challenges in membrane processes. Membranes get exposed to a wide range of contaminants, e.g. natural organic matter (NOM), bacteria, proteins and polysaccharides resulting in irreversible biofouling. Light-responsive surface modifications are gaining significant attention to mitigate fouling in membrane processes for the ability to tune precisely the physicochemical and morphological properties that allow remote and contactless interactions without changing the original chemical environment [1,2]. Hence, in this project, a facile copolymer of light responsive unit (modified-Azobenzene) and zwitterionic unit was synthesised and coated on commercial nanofiltration NF270 membranes. The coated membranes impart antibiofouling properties during filtration due to the presence of zwitterionic moeities. In addition, after the filtration cycles, when exposed under alternate visible irradiation, azobenzene undergoes reversible photoisomerization between the trans and cis conformation leading to dynamic change of surface physico-chemical and morphological properties [3]. Therefore, irreversible fouling is cleaned by activating the azobenzene unit under complete visible (or near Infrared NIR) photoirradiation, resulting in clean membranes with high permeability and retention recovery. The photochemical conversion efficiency was systematically determined by NMR stoichiometry analysis, UV/Vis spectroscopy and DSC thermogram of the copolymer. Moreover, we intend to understand the irreversible fouling removal efficiency of the modified membrane after filtration of water containing NOMs and bacterial (P. Putida) suspension. The light induced membrane process could be highly desirable for creating circular economy that could trigger interests for further development of renewable energy based separations, especially solar energy, without the consumption of fossil fuels.

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An Eco-Friendly Integrated Membrane Process for the Concentration and Purification of Functional Bioactive Compounds from Citrus Hybrid Tacle® Extract

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Tacle is a tangerine-like citrus obtained from the crossbreeding of Clementine and Tarocco cultivars. The extract of this fruit is rich in polyphenol glycosides, mainly naringin and hesperidin, and other bioactive phytochemicals typical of Citrus fruits (such as carotenoids). These compounds are well recognized for their protective activity against oxidative agents involved in several degenerative disorders [1-3]. Developing sustainable and effective purification procedures of bioactive compounds from vegetable sources has always been a challenging task for scientists. In this work, an eco-friendly process based on a combination of pressure-driven membrane operations and osmotic distillation has been investigated, for the first time, to purify and concentrate functional biomolecules in tacle extracts. In the first step, a concentrated stream rich in carotenoids was obtained by using hollow-fiber ultrafiltration (UF) membranes according to a batch concentration-diafiltration mode. The selected membranes showed a retention towards β-carotene higher than 98% and lower than 5% for polyphenols and sugars. In the second step, nanofiltration (NF) membranes in spiral-wound configuration were tested in order to produce concentrated fractions enriched in flavonoids and depleted in sugars. Membranes with molecular weight cut-off (MWCO) of 300-500 Da showed a sugar removal efficiency from the clarified extract (permeate UF+diafiltrate) of about 65%; on the other hand, more than 85% of flavonoids were rejected. A further concentration of the NF retentate was achieved by a final step of osmotic distillation (OD). According to the investigated process, three valuable and innovative fractions were obtained: i) a UF retentate fraction rich in carotenoids useful for cosmeceutical formulations, food supplements or as natural colorant; ii) a NF retentate rich in flavonoids of interest for pharmaceutical applications due to its anticholesterolemic activity; iii) a purified fraction enriched in sugars and salts suitable for drinking formulations.

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Hybrid FO-RO/MD Desalination Processes: System-Scale Analysis at the Water-Energy Nexus

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Hybrid desalination systems coupling Forward Osmosis (FO) with Reverse Osmosis (RO) or Membrane Distillation (MD) are investigated evaluating the water-energy nexus existing between the filtration performances in FO and the RO/MD energy consumption to re-establishing the concentration of the draw solution. In detail, we investigate the performance in case of low- and hyper- salinity feed solutions, discussing under which scenario each hybrid technology performs more effectively. An in-depth investigation of two innovative FO configurations is reported, discussing the potential applicability of a multi-stage approach via co-current membrane module design, as an alternative to single-stage counter-current operation. Then, energy and exergy analyses are performed to investigate the RO and MD, separately, taking into account un-conventional operating parameters envisaging future membrane and module developments. Finally, the coupling between FO and RO/MD systems is analyzed, discussing how the relative energy consumption of the two hybrid systems varies depending on the operating parameters of the FO. While FO-RO coupling provides the most efficient solution in terms of power consumption, it is restricted by the salinity level of the draw solution. On the other hand, the hybrid FO-MD solution is more versatile, as performance is less affected by the concentration of the feed solution, and more competitive exergy-wise due to the ability to harness low-grade heat.

Semi-Closed Reverse Osmosis (SCRO): A Concise, Flexible, and Energy-Efficient Desalination Process

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As a promising solution to intensified water scarcity, desalination via membrane-based reverse osmosis (RO) attracts unprecedented interest in recent decades. However, the prevailing process, single-stage RO (SSRO), still consumes much higher than thermodynamic minimum energy as its operation requires over-pressurization (OP) of the feed stream. Although alternative RO processes, such as closed-circuit RO (CCRO), batch RO (BRO) and multiple-stage RO (MSRO), can ameliorate OP, the mixing of recirculated concentrate and feed in CCRO and BRO generates entropy and increases energy consumption, while MSRO requires additional boosting systems that incur additional capital expenditure. Herein, we propose a new RO process with concise design and flexible operation, namely semi-closed reverse osmosis (SCRO). SCRO runs multiple cycles with varied applied pressures to mitigate OP and stores immediate feed and recirculated concentrate at each cycle in separate tanks to circumvent mixing and additional staging. Analytic results reveal that the SCRO integrating energy recovery devices (ERDs) consumes less energy than state-of-the-art RO processes for desalination of various water sources within typical water recovery regions. The superiority of energy efficiency of SCRO is more significant at higher recoveries. This study suggests that the proposed SCRO is a promising alternative to the existing RO processes in low-energy desalination.

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Positron Annihilation Lifetime Spectroscopy of Membranes – Probing the Free Volume with Antimatter

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A pulsed beam of antimatter particles, i.e. positrons, allows the quantitative characterization of free volume sizes and distributions inside thin polymer layers. With this technique, called positron annihilation lifetime spectroscopy (PALS), it is possible to investigate nanoscopic free volume elements (FVEs) in the range of ~ 0.2–3 nm non-destructively. Also FVE structures, enclosed inside the material without any connection to the surface are possible to determine. Depth resolution is reached by varying the kinetic energy of the particle beam. Depending on the material density, FVEs can be characterized in different depths (5 nm – 5 µm) beneath the surface. In insulating materials such as polymers, positrons may form a bound state together with present electrons – Positronium (Ps). From the Positronium lifetime, one can calculate an effective FVE radius by a semi-empirical, quantum-mechanical model (Tao-Eldrup-model). For this purpose, the pulsed low-energy positron system (PLEPS) [1] was developed. It is operated at the reactor-based, high intense positron source NEPOMUC. At present, it is possible to obtain with PLEPS positron and Positronium lifetime spectra of membrane samples including 4×10^6 counts in less than 10 minutes for a single implantation energy. A full depth-profile of 10-15 different beam energies requires about 2-4 hours. The system also allows in situ manipulation of the sample temperature between 80 K and 600 K during lifetime measurements. This enables to investigate free volume elements as a function of the temperature, and thus, to determine material phase transitions, e.g. the glass transition temperature. In this talk we describe the method and the present setup of PLEPS, its performance, and show a selection of exemplary applications (e.g. [2], [3]) to membrane problems.

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Current Status and Challenges in the Artificial Lung Membrane Technology

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The recent outbreak of COVID-19 pandemic in 2019 reasserted the necessity of artificial lung (AL) membrane technology to treat patients with acute lung failures. In addition, the aging world population inevitably leads to higher demand for better AL devices. Although membrane-based AL technology has improved significantly in the past few decades, the life quality of organ failure patients is still poor and the technology must be improved further. Most of the current AL literatures focus on the treatment and the clinical use of AL, while the research in the membrane development aspect of AL is relatively scarce. One of the speculated reasons is the wide interdisciplinary spectrum of the AL technology ranging from biotechnology to polymer chemistry and process engineering. There is a clear need for better membranes in terms of biocompatibility, permselectivity, module design, and process configuration. In this presentation, current status and challenges of AL research will be summarized. Also, an overview of our lab's research results will be presented.

Synthesis of Silica Derivatives and their Incorporation in Cellulose Acetate-based Membranes for Blood Purification

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Chronic kidney disease (CKD) has a prevalence from 11 to 13% in the world's population and the main treatment which partially restores kidney function is hemodialysis (HD). Despite current HD membranes being effective in the removal of small water-soluble compounds (molecular weight (MW) <500Da) and certain middle molecules (500<MW<60000Da), protein bound uremic toxins (PBUTs) remain essentially untouched. Accumulation of PBUTs in CKD patients has been associated to adverse cardiovascular complications by inducing inflammatory responses or by causing different vascular dysfunctions [1,2]. The development of synthetic membranes with larger pore sizes and molecular weight cut-off (MWCO) of ~40kDa resulted in higher removal of certain middle molecules [2], however, these are inefficient in the removal of PBUTs. Due to their high affinity towards plasma proteins, mainly albumin, when bound, PBUTs have MWs larger than 60 kDa. Thus, different approaches, such as the use of binding competitors of albumin have been attempted with promising results [2]. The main objective of this work is to study the possibility of incorporation of albumin binding competitors into monophasic hybrid cellulose acetate-based membranes towards enhanced removal of PBUTs. For this, as a proof of concept, silica derivatives using dyes that mimic the moiety of some binding competitors for albumin were synthesized and cellulose acetate (CA)/silica (SiO_2) membranes were prepared by coupling phase inversion and sol-gel technologies [3]. The use of the dye derivatives allowed the confirmation of chemical bonding of the newly developed SiO_2 derivatives to the CA matrix based on the final color of the CA/SiO_2 membrane obtained. Pure water permeation tests performed at 0.5bar for 3h revealed no detection of dye in the permeate and membranes maintained the same color for over 60 days. Therefore, it is possible to use this strategy to functionalize silica derivatives with binding competitors of albumin to produce CA/SiO_2-based membranes for enhanced PBUTs removal.

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Improved Gas Transport of Polyurethane-Based Membranes for Blood Oxygenation

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Extracorporeal Membrane Oxygenation (ECMO) is essential in critical care for the management of severe respiratory and cardiac failure. Considered the most important part of the ECMO circuit, the membrane blood oxygenator (BO) is responsible for maintaining O_2 and CO_2 levels in the blood. Yet, the poor hemocompatibility of current BOs, associated with the need of large surface areas, leads to activation of the coagulation system and the adhesion of platelets to the membrane surface and enhancing thrombin generation, contributing to the higher rates of thrombosis seen in patients supported by ECMO. Despite the considerable progress during the last decades in component technology and at the circuit level, efficient BOs for prolonged ECMO do not exist. In order to improve the O_2 permeability our research group has been focused on the development of new blend polyurethane-based (PUR) membranes, using polyether and polyester-based segmented PURs, which exhibit enhanced hemocompatibility, in association with a good flex-life and mechanical strength [1]. In this work, two groups of dense symmetric membranes were prepared by the solvent evaporation technique: pure polyurethane (PU) membranes and polyurethane blend membranes using different total polymer/solvent and polyurethane/second reagent weight ratios [2]. The mechanical properties of the membranes were studied through tensile tests. Single gas, O_2 and CO_2 permeation studies were carried out by the constant volume method at 37°C in an in-house built experimental set-up [3]. The permeability coefficients obtained from the permeation curves ranged from 239 to 347 Barrer for CO_2 and 26 to 30 Barrer for O_2. The ranges obtained for the diffusion coefficients by the time-lag method were between 1.4 and 3.0×10^{-6} cm^2/s for CO_2 and between 2.0 and 2.5×10^{-6} cm^2/s for O_2. The solubility coefficients varied between 116 and 186×10^{-4} cm^3/cm^3.cmHg for CO_2, and between 11 and 13×10^{-4} cm^3/cm^3.cmHg for O_2.

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Biomimetic Membrane Platform for Anti-Cancer Therapeutic Strategies

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Cancer is a leading cause of death worldwide and a major issue in society today. It represents an area of intensive research that needs more accurate models of tumours for preclinical evaluation of new therapeutics. Recent advancements in the field of tissue engineering by means of membrane technology, led to the creation of membrane-based tools that provide reliable in vitro tissue/organ models [1-2]. In this study, a biomimetic membrane platform that recapitulates cancer environment was developed as innovative tool for studying, for the first time, the anti-cancer effect of Plasma-Treated Water Solutions (PTWS). The efficacy of cold plasmas (CP) and PTWS in killing tumour cells is an emerging field of research [3] but needs suitable biomimetic environments for the in vitro validation. The membrane platform consists of gas permeable fluorocarbon (FC) membranes, biofunctionalized with Poly-L-lysine (PLL) to mimic the extracellular matrix and to provide specific cues for modulating tissues growth. The selectivity, together with structural, physico-chemical and mechanical properties of PLL-FC membranes, which also ensure the adequate supply of oxygen, allow the realization of a suitable biomimetic interface for the growth of cancer cells. This system was employed as a reliable in vitro model of tumours, namely neuroblastoma, breast and colorectal cancers characterized by worst prognosis. Specific tumour cells were cultured within the biomimetic membrane platform and treated with different amounts of reactive oxygen and nitrogen species (RONS) delivered by PTWS, which were previously enriched with L-Tyrosine and exposed to different CP. PTWS displayed high cytotoxicity, anti-proliferative, pro-oxidant and pro-apoptotic effects through molecular mechanisms involving the intracellular ROS production and the activation of JNK and caspase-3, depending on PTWS's ROS/RNS ratios. The present study provides new membrane tools and approaches for the in vitro screening of anti-cancer treatments.

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Application of 2D Material Membrane for Health Care Technology

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Advances in membrane technology have extended over and beyond filtration to applications in the field of medicine and agriculture. A targeted and systemic release of active agents is a highly desirable requirement of a controlled delivery systems [1]. Here in we report the controlled release of antimicrobial agents from 2D material-based membranes. We demonstrate a regulated release of these agents in an in-vitro wound scenario to maintain anti-microbial activity for more than 7 days, preventing infection due to notorious bacteria like staphylococcus aureus (S.aureus) [2]. The antimicrobial agents currently used in practice may prove lethal to the healthy cells if not delivered in controlled doses. Hence it is required that only the essential and active form of the drug is delivered for the desired purpose. For orthopedic wound care applications, we treat the 2D material-based laminates coated on standard Ti rods with antimicrobial molecules. The molecules eluted in a very controlled fashion were observed to maintain a clear bactericidal state during initial stages of wound healing. The presence of serum is considered to be a bottle-neck in microbial tests as they hinder the efficacy of the drug making it unavailable to attack the bacteria. Very remarkably, we also observe the sustained release of these anti-microbial agents despite re-challenging with fresh serum in the surrounding environment. The detected bacterial counts from the surface of the Ti rods were also minimal and observed non-culturable. This further suggests that this technology can also be applicable in filtration applications where the membranes frequently suffer from biofouling [3]. These membranes can be successfully tuned to capture and release anti-microbial agents to desired requirements, enabling them for different applications. Furthermore, by incorporating bio-compatible polymers, these membrane coatings become robust and abrasion resistant.

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Membranes Loaded with 18-β-Glycyrrhetic Acid for Skin Regeneration, Repair and Protection

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The development of biodegradable nano-structured membranes with specific functionalities and structural features able to provide adaptable biomimetic microenvironments for the control of the cell fate, represents one of the major challenges in skin tissue engineering [1-2]. Moreover, the membrane loading with a bioactive molecule constantly released in the time represents a further strategy to trigger or modulate biological processes needed for skin regeneration and repair. In this work, microporous membranes of chitosan loaded with a natural compound with therapeutic and healing properties were developed. 18-β-Glycyrrhetic Acid (GA) is a natural derivative of licorice (Glycyrrhiza) extract, with a wide range of pharmacological activities, including anti-oxidant, anti-carcinogenic, anti-inflammatory, wound healing, and protective of aging- and UV-induced skin damages. The biomolecule was incorporated in the microporous structure of the chitosan membrane as a water-soluble inclusion complex with β-cyclodextrin (β-CD). Composite CHT membranes, with different ratio of βCD-GA inclusion complex, were realized by phase inversion process [3]. Membranes were characterized to evaluate structural, physico-chemical and biodegradation properties, and the GA incorporation and release were assessed in the time. Successively, the developed membranes were used for the formation of epidermal constructs by using human keratinocytes. The morphological behavior, proliferation, metabolic functions, and the expression of specific cytokeratins involved in the keratinocytes differentiation, were expressed at different levels by cells cultured on native chitosan membrane, and on chitosan/βCD-GA composite membranes. Moreover, the protective effect of the GA was assessed after UV and oxidative stress damages induced in the developed epidermal membrane constructs, highlighting the pivotal protective role played by GA incorporated and released in the chitosan/βCD-GA membrane system. These achievements represent an attractive tissue engineering approach for the creation of membrane skin models that incorporate bioactive molecules useful in the tissue regeneration processes, and that could be used as a patch for topical drug delivery, wound healing and skin repair.

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High-pressure, high-recovery reverse osmosis for recycling of metal plating rinse water

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Metal processing industries discard substantial quantities of toxic wastewater containing valuable metals. Membrane reverse osmosis (RO) has been considered for the recycling of metal ions, but the requirements for high concentration factor (×10 to 20) and high pressure (up to 120 bar) are challenging for conventional RO systems. This would typically require triple-stage reverse osmosis with large footprint and high energy input. As an alternative, batch and semi-batch RO have been attracting interest for RO to achieve high concentration factor in a compact single-stage system with low energy consumption. Here we describe a high-pressure hybrid semi-batch/batch RO system designed to achieve concentration factor of up to 20 and operation at up to 120 bar. The system has been constructed based on a validated mathematical model and predicted to achieve Specific Energy Consumption <2.5 kWh/m^3 when concentrating trivalent chromium, Cr(III), metal plating rinse water by a factor of 20 corresponding to a water recovery of about 95%. Experiments with Filmtec™ SW30-2540 membranes (at ≤80 bar) provided preliminary data before upscaling to the high-pressure system, indicating that concentration factors for the electrolyte rinsing bath wastewater of up to ×10 are possible. We will report on results obtained from the high-pressure system in concentrating saline solutions including Cr(III) metal plating wastewater. We will also report on the post-treatment process needed to remove boron from the permeate which requires pH adjustment and a second RO stage at low pressure.

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Advanced wastewater treatment using a novel hybrid anaerobic membrane bioreactor with immobilized biomass foam

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Anaerobic membrane bioreactor (AnMBR) technology combines anaerobic wastewater treatment with membrane separation and produces biogas for potential energy generation while also producing high quality effluent. However, membrane fouling prevents this technology's widespread application. The goal of this research is to develop a novel immobilized biomass foam bioreactor as an anaerobic treatment, with membrane filtration (AnIFMBR) for sustainable domestic wastewater treatment. This innovative immobilization technique can stabilize biodegradation and reduce membrane fouling. A special bench-scale reactor containing submereged membrane modules (AnIFMBR) was designed, constructed and operated to evaluate the efficiency treating synthetic domestic wastewater at an organic loading rate of 1.3 g/L·day. The AnIFMBR was inoculated with anaerobic biomass immobilized within hydrophilic polyurethane [1, 2]. It was found that the operation of the AnIFMBR system (at 10 LMH) using polyethersulfone membranes without biogas sparging caused rapid fouling formation. However, when biogas sparging was applied, a stable process was achieved during 70 days of operation, with removal efficiency of 58-78% for the first anaerobic chamber, and total removal of about 90% of the COD (for the entire AnIFMBR system). By comparison, a reduction of the COD removal down to 20-35% led to a rapid fouling formation after about 20 days of AnIFMBR operation. The expected outcome of this research can lead to the development of a novel engineering and cost-effective paradigm for wastewater treatment to produce high quality effluent and biogas. However, there is still a need for further optimization and testing of the system, specifically by comparing AnIFMBR system stability with two separated systems of immobilized biomass foam and AnMBR mode.

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Gravity-Driven Membrane Bioreactors for Decentralized Wastewater Treatment: Understanding the impact of fouling control strategies on treatment performance

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In rural areas and developing countries, Decentralized Wastewater Treatment (DWWT) systems are considered a reliable solution due to their limited cost for installation, operation, and maintenance [1]. Media filters, lagoons, and constructed wetlands have been proposed so far. However, in some scenarios, the effluent quality produced cannot meet the wastewater standards for discharge or reuse [2]. Conventional membrane bioreactors (MBRs) are a reliable alternative due to the high quality of the permeate. Nevertheless, MBRs require several equipment's and skilled operation, which limit their application for decentralized water treatment [3]. Recently, Gravity-Driven Membrane (GDM) filtration process has been investigated as DWWT. This system does not require any additional energy for the filtration since it is driven only by hydrostatic pressure, leading to the achievement of a stable permeate flux without any chemical or physical cleaning. In addition, as low energy and chemical-free process, GDM displays lower costs compared to conventional membrane processes. The bottleneck of GD-MBR is represented by the permeate flux, which is significantly lower than the typical MBR processes [3]. To this extent, it is crucial to enhance GD-MBR permeate flux by applying physical cleanings. In this study, the effects of periodic membrane fouling control strategies in Gravity-Driven Membrane Bioreactor (GD-MBR) treating primary wastewater. The impact of each control strategy (i.e intermittent filtration, air scouring, intermittent filtration coupled with air scouring) on the reactor performance, biomass morphology, and fouling composition were evaluated. The key findings of our study reveal that GD-MBR is a versatile treatment process capable of achieving different treatment performances, where the choice of the appropriate operation relies on the eventual discharge or reuse of the treated effluent. In this study, the reactor employed with intermittent filtration and air scouring achieved the best performances, showing the better compromise between water quality (i.e., TN and COD removal) and production.

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Treatment of wastewater effluents using nanofiltration and low pressure UV treatment to produce high quality water that can be reused for irrigation for food production

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Agriculture irrigation accounts for approximately 70% of all water withdrawals. Climate change and the increase in human population will lead to a higher food and water demand. Hence, it is crucial to explore new water sources for agriculture irrigation, such as the reuse of wastewater effluents. Prior to irrigation with wastewater effluents, it is important to evaluate the potential uptake of contaminants by food crops. In this study, different water sources (wastewater effluents, wastewater effluent after nanofiltration treatment and tap water) were used for the irrigation of raspberries. Several antibiotics (ciprofloxacin, levofloxacin, ampicillin, ertapenem and meropenem) as well as total coliforms and E. coli resistant to different antibiotics (ciprofloxacin, levofloxacin, meropenem, streptomycin and ampicillin) were quantified in the different irrigation waters over a 5 Month irrigation period. Of all the target antibiotics, only ciprofloxacin, levofloxacin, and meropenem were detected at different concentrations in the different irrigation waters. The discharged wastewater effluent had higher levels of ciprofloxacin and levofloxacin, ranging from 174 to 1335ng/L. The combination of UV photolysis with membrane filtration was tested to further treat the permeate and retentate samples from the nanofiltration unit. When ciprofloxacin was present in the permeate samples, after exposure to UV photolysis using a low-pressure mercury lamp the antibiotic was not detected. In the highly concentrated retentate samples, 57% of ciprofloxacin and 31% of levofloxacin were degraded by UV photolysis using an extremely low UV fluence. If a higher exposure time was used, higher degradation levels would be achieved. These low UV fluences were able to inactivate successfully the antibiotic resistant bacteria in the retentate samples (with percent inactivation's higher than 4-log). Analytical methods were validated for the detection of the target contaminants in the solid matrices and the potential uptake of the target contaminants by the fruits will be evaluated.

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Treatment of pickling and passivation solutions by diffusion dialysis and membrane distillation at a pilot scale

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In the electroplating process, pickling and passivation baths are rapidly contaminated by metals such as zinc and iron, which decreases their efficiency and leave them to discard. The regeneration of these solutions is complicated by the wide concentration ranges of acids and metals, by the presence of inhibitors and surfactants and by the existence of various metals in solution [1, 2]. A pilot plant composed of a diffusion dialysis (DD) module and a membrane distillation (MD) module has been installed in Electroníquel S.A. company and tested to treat pickling and passivation waste solutions. The performance of the DD on the separation of the acid and the metal compounds and of the MD on the concentration of chromium and production of high purity permeate were evaluated. The effect of different flow rates at retentate and diffusate sides on the performance of DD was studied keeping constant the concentration of the compounds. The MD module was tested at different thermostatic flow rates, feed flow rates and temperatures of hot and cold sides, studying the effect of these variations on the permeate production and on the specific thermal energy consumption. For a concentration of H_2SO_4 of 10 %, 30 g/L of iron and retentate/diffusate flow rates 6/12 L/h, the acid recovery was kept between 74 % and 65 %. Iron rejection was greater than 80 % in all the cases. Water cleaning was enough to recuperate initial recoveries, manifesting the resistance of the membrane to these solutions. Also the efficiency of the process was tested for HCl solutions. HCl recoveries was in the range of 90-80 %. For a feed flow rate of 20 L/h, a hot temperature of 70 ºC and a concentration of chromium of 2.6 g/L, the concentration of chromium was 40 %. Impurities of zinc and iron did not affect the efficiency of the process. MD proved suitable for the treatment of passivation solutions regenerating solutions with anticorrosive properties, with no deterioration of the membrane.

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Natural organic matter membrane fractionation of surface water – A new approach

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Introduction Natural organic matter (NOM), contained in drinking water resources, acts as a nutrient for bacterial regrowth in the distribution system. Chlorine is a common disinfectant to inactivate pathogens, but it also reacts with NOM to form disinfection by-products, which are correlated with health issues. To identify the precursors that are responsible for the formation of these by-products from the complex NOM mixture, the latter is split into smaller fractions with similar chemical or physical properties. Dead-end ultrafiltration membrane fractionation is the most commonly used technique to split NOM by size but is unable to produce sharply separated fractions. Therefore, this research will seek for a new membrane fractionation method that separates NOM into biopolymers (BP), humic substances (HS) and low, molecular weight compounds (LMW). Materials and Methods A nano- (MWCO 150-300 Da) and ultrafiltration membrane (MWCO 1000 Da) were selected based on their performance to separate the three fractions and are used for the fractionation process. Cross-flow nanofiltration is performed to collect the LMW fraction in the permeate. The retentate is send to a cross-flow ultrafiltration to split the BP from the HS. For both nanofiltration and ultrafiltration, a diafiltration is executed after a certain filtration time. This is the addition of demiwater to the feed. It is used to reach a near 100% separation of the fractions and to reduce the risk of (severe) fouling due to concentration of the feed. Results and Discussion After the fractionation, a 90% pure LMW and 99% pure HS fraction were obtained. Although diafiltration removed 90% of the HS compounds, the process should be executed longer to increase the purity of the BP (26% for now). A model was built and verified to predict the diafiltration time needed to increase the BP purity and will be used in the future.

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A smart and responsive crystalline porous organic cage membrane with switchable pore apertures for graded molecular sieving

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Membranes with high selectivity offer an attractive route to molecular separations, where technologies such as distillation and chromatography are energy-intensive. However, it remains challenging to fine-tune the structure and porosity in membranes, particularly to separate molecules of similar size. Here, we report a process for producing composite membranes that comprise crystalline porous organic cage films fabricated by interfacial synthesis on a polyacrylonitrile support. These membranes exhibit ultrafast solvent permeance and high rejection of organic dyes with molecular weights over 600 g·mol-1. The crystalline cage film is dynamic and its pore aperture can be switched in methanol to generate larger pores that provide increased methanol permeance and higher molecular weight cut-offs (1400 g·mol-1). By varying the water/methanol ratio, the film can be switched between two phases that have different selectivities, such that a single, ‘smart’ crystalline membrane can perform graded molecular sieving. We exemplify this by separating three organic dyes in a single-stage, single-membrane process [1].

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Alkyl levulinates: a new class of green solvents for PVDF membrane preparation

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Environmental protection and climate change are current issues of the modern society. The increasing awareness towards environment protection and safeguard is driving the search of new, more sustainable solvents and raw materials. The 5th principle of Green Chemistry encourages the use of safer solvents and auxiliaries [1,2]. Currently polymeric membranes are produced using organic solvents such as N,N-Dimethylformamide (DMF), N,N-Dimethylacetamide (DMA) and N-methyl-2-pyrrolidone (NMP) which are considered toxic and harmful for the environment. The search for new green and more benign solvents is, therefore, very attractive in the membrane preparation field. In this work, for the first time, alkyl levulinates were used for the preparation of PVDF copolymer (Solef®PVDF6012) and homopolymer (Solef®PVDF-HFP 21510) membranes. Alkyl levulinates originate from biomass-derived products such as levulinic acid, upon reaction with alcohols, or directly from cellulose [3]. Different alkyl levulinates were used by evaluating the effect of their alkyl chain length on dope solution thermodynamic factors and on the phase inversion process. Membranes were prepared by coupling vapor induced phase separation with non-solvent induced phase separation (VIPS-NIPS) techniques using polyvinylpyrrolidone and polyethylene glycol as pore-forming agents. The structure of the membranes was tailored by acting on the exposure time to humidity during VIPS process (from 0 to 5 min) and by using the water as a coagulation bath. The thermodynamic aspects (solubility parameters, polarity and polymer-solvent distance) and kinetic parameters (viscosity) were also studied. It was possible to produce membranes with controlled morphology characterized by finger-like and/or sponge-like structures. The membranes exhibited a pore size in the ultrafiltration (UF) and microfiltration (MF) range with a water permeability that makes them ideal for applications in water filtration processes.

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Influence of crosslinking reaction time and crosslinker amount on separation performance and stability of polyvinyl alcohol/pectin membranes

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The use of pectin (PEC) as a natural crosslinking agent for polyvinyl alcohol (PVA) [1] has allowed to develop a biopolymeric membrane highly stable in water (boiling, acidic and basic water), and with unprecedented mechanical resistance (Young's modulus = 3.11 GPa; tensile strength = 90 MPa) comparable to those of petrochemical polymers commonly used in manufacture of the state-of-the-art of durable membranes [2]. Furthermore, the membrane showed long-term unchanged water vapor barrier properties (1.34·10-2 g·m/(h·m^2·bar) falling within the range suitable for food packaging. The aim of this work was the understanding of the influence of both crosslinking reaction time and amount of crosslinker on the membrane stability and separation performance. A study was conducted evaluating the stability in water of PVA/PEC 1:1 membranes prepared at different crosslinking time (from 0.5 to 5 h). The minimum crosslinking time necessary to develop a stable membrane (weight loss in water less than 1%) was identified. The mechanical properties (Young's modulus, E-mod and tensile strength, Rm) improved by increasing the crosslinking time and the amount of PEC. The lecture will also discuss the structural properties of the membranes (including, Nominal Molecular Weight Cut-Off, pore size, porosity, membrane thickness, surface functional groups, wettability, zeta potential) and their separation performance in terms of pure water permeance, Bovine Serum Albumin rejection, flux recovery rate, reusability in multiple cycles.

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Cellulose acetate hollow fiber membranes for forward osmosis applications

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The need for membranes based on bio-based polymers is increasing, especially with regard to their fate after use. Cellulose-based membranes could be good candidates, as they have already been extensively studied e.g. in the field of drinking water purification.[1] Especially in applications such as forward osmosis, cellulose acetate membranes offer advantages in terms of robustness and fouling compared to TFC membranes.[2] However, environmentally friendly production using green solvents is also important for the development of sustainable membranes.[3] In this work, cellulose acetate hollow fiber membranes were manufactured by a combined vapor-induced and non-solvent-induced phase separation (VIPS-NIPS) using the green solvent Agnique® AMD 3 L (N,N-dimethyl lactamide; AMD) and acetone as co-solvent during wet spinning. The influence of solvent composition, polymer content in dope solution, extrusion pressure during membrane fabrication and thermal post-treatment of the membranes were investigated. The morphology, mechanical characteristics, molecular weight cut-offs (MWCO) as well as water permeances and salt rejections of the prepared hollow fiber membranes were evaluated. In addition, cellulose acetate hollow fiber membranes with a rejection of > 98 % of $MgSO_4$ were tested in a set-up for forward osmosis with a draw solution of 1 M NaCl and ultra-pure water as feed. With a mean water flux of 3.2 L m^{-2} h^{-1} and an inverse salt flux of 2.6 g m^{-2} h^{-1} the hollow fiber membranes are potential candidates for further osmotic membrane processes.

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Defeating Defects: New Innovations in Free-Surface Electrospinning

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Elmarco is the global leader in industrial electrospinning equipment and solutions. Many of our customers are interested in membrane applications such as liquid filtration, performance apparel and battery separators. It is well understood that these applications can benefit from the use of electrospun membranes vs. conventional membranes due to the ability to achieve higher flux (or flow rates) at the same rejection efficiencies. These higher flux and higher performance membranes will reduce energy consumption and process costs. One of the biggest advantages of the electrospinning process is that there are many variables that can be used to tune the nanofiber layer properties. However, despite controlling these variables, a major hurdle in reliable, industrial membrane production using electrospinning, is defect generation. For the aforementioned applications, any significant defect and/or pinholes in the membrane, whether electrospun or conventional, has always created a challenge due to the stringent material performance requirements. Elmarco has taken our significant knowledge and experience with our existing free-surface electrospinning technology and created a new generation of equipment technology which maintains the industrial, high-throughput, and scalable aspects of our existing equipment along with the industry leading ability to virtually eliminate significant membrane defects from these membranes. In this paper, we will demonstrate through filtration performance, hydrostatic head, and porometry, the impact of defects and the ability for our newest innovation to produce nanofiber membranes that are devoid (free) of significant defects. Moreover, we have developed a membrane visual scanning technology that can continuously quantify and track defects throughout the membrane production process. This material quality information provides confidence to our customers that the material can be properly processed downstream and will ultimately result in the final product with the necessary performance specifications and most importantly, without critical defects.

Constructing carbon molecular sieving membrane with high permselectivity for gas/vapor separation: from disk to free-standing type

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carbon molecular sieve membrane (CMSM) had become the spotlight in the scientific field for its specific features such as favorable stability in severe environments, silt-like microstructure, and bimodal pore size distribution [7, 8]. These unique merits render CMSM to achieve a challenging yet efficient gas separation process by the selective difference in the transportation rate for gas molecules at sub- nanosized levels regardless of the presence of common poisoning matters like acid components. Nevertheless, the aging phenomenon as well as the difficulty in reproducibility of promising CMSM constrained its long-term operation and availability [6].

The current work aims to feature optimal carbonization conditions for carbon hollow fiber membrane toward hydrogen purification. The prerequisite for fulfilling this purpose is the best permselectivity of the resultant membrane. Three different carbonization atmospheres (vacuum, helium-flowing, and argon-flowing) had been opted and compared their effect on the change in microstructure and gas permeation property of as-synthesized carbon membranes using pure gas permeation test coupled with an array of characterizations including Raman, XPS and. In addition, the issue of vacuum degree was addressed to maintain the best reproducibility of carbon membrane. The results revealed that CHFM-HV had the best separation capability for H_2/CH_4 of 516.90 and thus came to prominence toward hydrogen purification due to an attractive synergism of ordered microstructure and larger pore volume. It is worthwhile to note that the recovery of permselectivity for CHFM-HV reached up to 90% after 4 months, showcasing the aging-resistance ability. Therefore, this viable approach to altering the microstructure of the carbon membrane and thus enhancing separation performance and long-term stability was suitable for extended application of a diversity of polymer precursors and prolonged the lifespan of the as-prepared carbon membrane.

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CO_2 sorption and diffusion in fluorinated polymers for carbon capture and transport application

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Fluorinated polymers play a significant role in the Carbon Capture and Storage (CCS) value chain, which is considered as the most viable solution to limit the CO_2 emission in the atmosphere, with the potential to be applied at short-medium times [1]. Indeed, among the various classes of polymers, fluorinated materials look promising for their potential application for CO_2 capture as membrane materials and in CO_2 transport as liner materials of pipelines, vessels etc., due to their excellent thermal and chemical resistance combined with a pronounced favorable interaction with CO_2 [2,3]. In the current work, various reference fluorinated polymers (PVDF, PTFE, PVF, ETFE, PFA and FEP) have been characterized by CO_2 sorption and transport in a wide temperature and pressure range, aiming to investigate the gas solubility, permeability and diffusivity under various conditions. The aim is to assess the effect of the fluorine content on the CO_2 solubility and diffusivity and to investigate how the resulting membrane morphology alters gas transport properties. This may allow to predict how the gas affects the performance and the stability of the materials even in supercritical or liquid/dense state, due to a physical change in their structure. Furthermore, the results obtained are analysed by a thermodynamic equation of state (EoS) to describe the solubility behaviour, while the standard transport model (STM) provides a reliable representation of gas transport. The comparison of sorption and transport data by means of the dedicated model allows the reliable prediction of the effects of CO_2 on such fluorinated polymers at all desired temperature and pressure ranges, relevant for CO_2 transport (e.g. dense phases, up to supercritical conditions) or capture applications by membranes. This work is a step forward in understanding and modelling of the complex interaction between supercritical CO_2 and polymers in view of their future use in industrial applications.

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Simultaneous GHG removal from wet CO_2 streams using novel composite membranes

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Typical streams requiring CO_2 removal such as natural gas and power plant flue gas contain high content of water necessitating dehydration as a pre- or post-treatment unit. In the current century, CO_2 removal is important in order to obtain clean and high combustion fuels calling for more efficient and environmentally benign processes. Membrane-based separation which already offer numerous advantages compared to conventional amine technology also has potential to further footprint reduction with smart membrane design. In this study, CO_2-philic mixed matrix membranes (MMMs) are fabricated using a water harvesting metal organic framework, MOF-801, with an average particle size of 120 nm to evaluate simultaneous H_2O and CO_2 separation from CO_2/N_2 and CO_2/CH_4 gas streams. A new green synthesis procedure for MOF-801 was devised yielding fillers with CO_2 adsorption capacity almost twice those of CH_4 and N_2 also verified with molecular simulation approaches with H_2O cluster formation due to H-bonding at Zr metal sites. These high performing MOF-801 nanoparticles are well-dispersed in a matrix of Pebax to fabricate MMMs confirmed with SEM analysis. MMM having 20 wt.% of MOF-801 revealed 109 and 81 % enhancement in H_2O and CO_2 permeabilities for the humid CO_2/CH_4 stream compared to the neat Pebax membrane at 6 bar with 11 % increase in CO_2/CH_4 separation factor. Similarly, 145 % CO_2 permeability improvement in humid CO_2/N_2 stream for MMM having 20 wt.% of MOF-801 is observed compared to neat Pebax membrane with a considerable increase as 151 % in CO_2/N_2 separation factor. In both humid and dry conditions, MMM having 20 wt.% of MOF-801 revealed superior CO_2/N_2 separation performances by exceeding the Upperbound. This study shows that membrane with MOF-801 is efficient for simultaneous CO_2 and H_2O removal leading to economic and environmental benefits by simplifying and downsizing process equipment in many CO_2 separation applications.

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Mixed gas diffusion and permeation of ternary and quaternary gas mixtures in glassy membranes for CO2/CO separation in CO2 utilization routes

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CO2 reforming by non-thermal plasma (NTP) conversion is an emerging technique for CO2 recycling. CO2 dissociation occurs by reaction CO2-->CO+1/2 O2 and requires only 5.5 eV [1,2]. When driven by renewable energy, this innovative conversion process would be an important step towards a sustainable energy scenario. The mixed-gas separation performance of Matrimid®, polyetherimide (PEI) and polylactic acid (PLA) membranes, were characterized in the presence of ternary and quaternary gas mixtures containing O2, N2, CO and CO2 and mimicking the products of reforming CO2 conversion reactions. The membrane-based separation of this mixture is poorly characterized and original data were obtained in a novel mass spectrometric apparatus that permits to monitor the instantaneous permeate composition, thus allowing to evaluate both mixed gas diffusion and permeability coefficients of all gases. [3] In all examined polymer films the CO2/CO selectivity has a solubility-selective character that favours the permeation of CO2. [3] The ideal selectivity of all membrane samples decreases with temperature, reaching values of 10 at 335 K in Matrimid®. The CO2/CO selective performances of all examined membrane do not show remarkable variations exposing the membrane samples to CO2- rich gas mixtures as feed gas. The Robeson's upper bound correlation for the CO2/CO gas couple is here proposed for the first time. With the growth of CO2 utilisation efforts, we expect the CO2/CO separation to be a relevant one, and the membrane-based separation one of the main strategies to achieve it.[3]

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Bioinspired ionic liquid-graphene-based smart membranes with electrical tunable channels for gas separation

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Fast response and adjustable selectivity have always been the main goals pursued by the manufacturers of smart membranes. Herein, we demonstrate smart membrane materials composed of ionic liquid (IL), PVDF, and graphene. The infusion of IL aims to increase the β crystal phase of PVDF, which is also assisted by the presence of graphene in the system. β-PVDF increased from 48.9% to 79.8% when the ionic liquid content [BMIM][BF4] increases from 2 to 10 wt.%, which makes the membrane more electro responsive. The optimum membrane of PG-BF4 10 wt.%, upon supply of 3 V DC electricity, exhibited CO_2/N_2 selectivity value of about 129.03 and CO_2 permeability of 193.55 Barrer, exceeding the 2008 Robeson upper bound. Positron Annihilation Lifetime Spectroscopy (PALS) data and Density Function Theory (DFT) simulations helped to realize the mechanism of selective transportation of CO_2 gas through the membrane both theoretically and experimentally. PALS results indicated that the free volume of PVDF increased; indicating that CO_2 molecules can easily pass through the system. DFT simulation revealed that the CO_2 molecules show more attraction and lower adsorption energy under electric stimuli, confirming that CO_2 molecules adsorb and diffuse quickly through the system than O_2 or N_2. Overall, tuning the free volume and molecular interactions within the membrane channels provide a rational molecular sieving approach to improve membrane selectivity [1-2].

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Asymmetric Polybenzimidazole Membranes with High Performance and Durability for Anion-Exchange Membrane Water Electrolysis

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A high performance and durability asymmetric membrane (Asy-PBI) with dense and porous structure based on polybenzimidazole (PBI) was developed for anion-exchange membrane water electrolysis (AEMWE). In order to understand the role of the membrane structure in AEMWE performance, a dense membrane (Dense-PBI) mainly used for AEMWE was fabricated and compared to Asy-PBI. To fabricate the Asy-PBI the synthesized PBI was mixed with an appropriate amount of a pore-forming agent (dibutyl phthalate, DBP), which could be removed by immersing it in methanol solution. The formed Asy-PBI was separated from the glass and soaked in methanol overnight at room temperature to leach DBP and remove residual solvent to complete the final membrane. The relationship between the membrane structure and performance was discussed based on the sorption and diffusion properties of the membranes. It was found that the material transfer efficiency of water and KOH of the porous structure play a key role in membrane performance and stability. The Asy-PBI indicated KOH solution uptake of 207 wt% and hydroxide conductivity of 14.5 mS cm^{-1}, each higher than the corresponding values for the Dense-PBI (KOH solution uptake: 30 wt%, hydroxide conductivity: 9.5 mS cm^{-1}). As a result, the Asy-PBI exhibited a current density 4.3 times that of the Dense-PBI (i.e., 2.95 A cm^{-2} and 0.56 A cm^{-2}, respectively) at a cell voltage of 2.0 V in electrolysis tests performed with circulated 10 wt% KOH solution at 80 °C. Furthermore, the AEMWE showed excellent durability, generating 1 A cm^{-2} for over 100 h at 80 °C and the highest performance (3.31 A cm^{-2}) during the electrolysis test conducted over a pressurized process at 120 °C. This study demonstrates that the structure of asymmetric membranes can effectively improve AEMWE performance.

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The influence of anion-exchange membrane nanostructure on ion transport: Adjusting membrane performance through fabrication conditions

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Anion-exchange membranes are recently extensively investigated as key components in water electrolyzers and as a tool to cost-efficiently recycle acids from industrial processes.[1], [2] Still, for both applications the achieved transport rates and selectivities of state-of-the-art membranes limit the widespread use. Optimizing membrane performance was so far mostly done by searching for new polymers, but the membrane morphology also heavily contributes to the transport properties.[3] Moreover, research into the correlation between fabrication conditions and nanoscale structure only recently came into focus, and underlying mechanisms are still partially unknown. Herein, we present the fabrication of several anion-exchange membranes from the same polymer with variations in the self-assembled morphology of hydrophilic regions resulting in drastic changes in ionic conductivity and acid diffusion rates. Using an anion-exchange polymer, we systematically investigated how nanochannels form during membrane formation and could successfully alter their number density as well as their size and charge density. Using different solvents and additives for fabrication and specific post-treatment methods, we were able to tune the separation behavior. We also showed that this is directly connected to the overall macroscopic structure and properties of membranes, i.e. different density or water uptake. Our research demonstrates how important it is to control the nanoscale morphology; the different fabrication conditions led to membranes that differ up to 5-fold in their ionic conductivity and up to 3-fold in their acid diffusion rates while being made from the same polymer at same thickness. Additionally, acid selectivity was also influenced by the nanostructure, resulting in different diffusion rate ratios for HCl, HBr and acetic acid. Overall, we established protocols and knowledge to control the nanophase segregation during anion-exchange membrane fabrication and elucidated how the morphology influences ion transport rates and can be used to adjust membrane separation properties.

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Novel antifouling surface grafted mebranes for wastewater tretmnet

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The declining water flux over time due to fouling represents the main onstream issue of the existent membranes in wastewater treatment. Thus, preventing and mitigating membrane fouling is considered the key challenge to improving performance and extending the use of membranes in water treatment and recovery applications. Novel membranes with improved properties will lead to reduced energy consumption, reduced cleaning and consumption of chemicals, as well as the less frequent replacement of membranes. In the urrent investigation we developed methods for antfiouling and anti-scaling functionalizing of commercial ultrafiltration (UF) and microfiltration (MF) membranes. The manufacturing technology aims to obtain membranes with improved water flux, stability, antifouling properties exceeding the performance of commercial membranes. The membranes are aimed to treat the intake water in fish farms and wastewater in general. We aim for easily scalable methods for large production, such as direct surface modification inside existent commercial membrane modules. The fabrication process consists of UV grafting of polymer chains from acrylate type of monomers (acrylic acid, methacrylic acid, hydroxyethyl methacrylate) onto the surface of microfiltration membranes (PVDF and PES). The succesfull membrane hydrophilization was characterized by FTIR, wettability (contact angle measurements) and water permeability. The membranes shows a decrease in the contact angle from 85 to 70 for PVDF and from 61 to 44 for PES indicating hydrophilization of the membrane which was confirmed by FTIR as well. The clean water flux was measured in cross-flow configuration at room temeperature, various water flow rates and two applied pressures. The water flux was reduced due to the surface grafting but the membrane bulk prorosity was little affected by grafting. We investigated various fabrication strategies to prevent and reduce the clogging of the membranes pores during grafting. The porosity of the membrane prior a post grafting was investigated using mercury intrusion porosimetry.

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Anti-scaling thin-film composite membranes decorated with pseudo-bottle-brush polyzwitterions for high-recovery desalination of brackish water

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Desalination is the most common technology for producing potable water from brackish and seawater today. However, mineral scaling limits the recovery ratio during desalination. This is extremely important in inland desalination, where the concentrate cannot be discharged back to the sea. In addition, silica scaling presents an additional challenge in the case of brackish water. We present a two-step activation process to achieve polyzwitterion pseudo bottle brushes on reverse osmosis (RO) membranes to mitigate mineral scaling propensity almost completely. First, a thin layer of poly2-hydroxyethyl methacrylate (polyhydroxyethyl methacrylate) was grafted by free radical polymerization. Then, atom transfer radical polymerization (ATRP) initiators were incorporated into the poly(HEMA). Finally, poly(sulfobetaine methacrylate) brushes were grafted by surface-initiated activator generated by electron transfer-ATRP at three different thicknesses. The successful grafting and the differences in brush yield at different reaction times were verified by XPS and ATR-FTIR. Atomic force microscopy and contact angle measurements revealed that the surface roughness, surface stiffness, and hydrophilicity greatly decreased with prolonged reaction time. A higher grafting yield resulted in lower permeability but a higher salt selectivity. Desalination experiments with synthetic gypsum and silica scaling solutions mimicking 80% recovery of brackish water demonstrated that the scaling of the modified membranes was significantly reduced compared to the pristine RO membrane and that the anti-scaling tendency correlated with brush thickness. The highest-yield modified membrane was further studied for filtration of semisynthetic brackish water, mimicking 90% recovery. While severe scaling appeared on the pristine RO membrane, the modified membrane showed an excellent anti-scaling performance. HR-SEM images confirmed the extremely high scaling of the pristine membrane and very low scaling on the nano-structured membrane. Overall, this work provides a convenient approach for preparing bottle brush-like grafts on RO membrane surfaces and expands on the implications of brush-decorated TFC surfaces for mineral scaling.

Nanostructured pervaporation membranes exclusively made of amphiphilic diblock copolymers: polystyrene-block-poly(vinyl alcohol)

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Self-assembled copolymers have emerged as non-traditional materials with outstanding properties resulting from the combination of chemically different polymeric segments into a single polymer chain.[1] Nowadays, the advances in amphiphilic block copolymers offer the possibility to manipulate and control the nanostructure of materials and thus to exquisitely tune their final properties. The use of block copolymers in membrane science has became a revolutionary achievement as the number of preparation steps and the manufacturing cost of end products can be significantly reduced. By adequately choosing the solvent and the block copolymer composition, well-defined nanostructured membranes are obtained. Consequently, membranes with unequivocal separation performances can be produced.[2] In this work, we will present the amphiphilic polystyrene-block-poly(vinyl alcohol) diblock copolymer (PS-b-PVA) as an attractive material for designing pervaporation membranes. The combination of the benchmark hydrophilic polymer (PVA) [3] with a stabilizing hydrophobic domain within the membrane matrix has numerous benefits, such as the enhancement of separation performance and robustness (lifetime). Although this type of copolymers is commercially available, they are expensive and not suitable for the preparation of membranes in terms of molecular weight and PVA content. Thus, we have synthesized tailor-made PS-b-PVA block copolymers exclusively for pervaporation membranes. We will show a series of PS-b-PVA diblock copolymers that were successfully used to fabricate dense films, which were evaluated as membrane materials. We will also demonstrate that the flux or selectivity (for ethanol dehydration) of thin film composite membranes can be improved by simple manipulation of the self-assembly process and by the block copolymer composition. In addition, the new PVA-based membranes can be manufactured in the absence of chemical cross-linkers which is advantageous from an industrial point of view.

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Development of Ti2AlN MAX phase/cellulose acetate nanocomposite membrane for removal of dye, protein and lead ions

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In this research, the effect of Ti2AlN MAX phase on the performance of cellulose acetate (CA) polymeric membrane was investigated. In the first step, Ti_2AlN MAX phase was synthesized via the reactive sintering method, and characterized using diverse techniques. Successful synthesis of MAX phase with high purity in the hexagonal crystalline structure was confirmed with XRD pattern. Layered morphology was observed for synthesized MAX phase in images of scanning electron microscopy (SEM) and transmittance electron microscopy. The prepared MAX phase was used as a hydrophilic inorganic additive to improve the performance of CA membrane. The phase inversion method was used to fabricate MAX phase/CA nanocomposite membranes. The morphology of the fabricated membranes was assessed using SEM and atomic force microscopy. An improvement in hydrophilicity of the CA membranes was observed by incorporating the MAX phase into the matrix of membranes. The nanocomposite membrane containing optimum content of MAX phase (0.75 wt%) showed a threefold increase in permeability during filtration of pure water and dye solutions. In addition, the optimum nanocomposite membrane exhibited improved flux recovery ratio of 92.7% with high removal efficiency of 70.7% for reactive black 5, 93.5% for reactive red 120, and >98% for bovine serum albumin. Finally, the rejection of different salts was investigated and the optimum nanocomposite showed high rejection for lead ions (97%) with moderate rejection for Na2SO4 (~55%) and NaCl (~30%). The results of this research demonstrated the high potential of MAX phase-based materials for improving the polymeric membranes.

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Synergistic integration of photocatalytic and thermocatalytic nanoparticles with graphene oxide in nanofiltration membranes for water depollution

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Graphene oxide (GO) has emerged as a promising material for membrane fabrication. Indeed, GO membranes present high-water permeability, and their selectivity can be tuned by crosslinking GO sheets with molecular spacers of different sizes [1]. Concerns about the reproducibility and the durability of GO membranes remain. Nevertheless, GO can be prepared from inexpensive graphite (e.g., via the Hummers' method) and the 2D structure makes it easy to coat on top of porous substrates to form membranes with selectivities in the nanofiltration range. Moreover, synergistic integration of GO with specific inorganic semiconductors (e.g., TiO2) has been proven to enhance their (photo)catalytic activity in the abatement of water pollutants [1]. Therefore, a few (photo)catalytic GO membrane concepts have been proposed in recent years with the aim to combine the filtering properties of GO layers with the advanced oxidation of water pollutants by (photo)catalytic nanoparticles. At this conference, we wish to present newly developed nanofiltration membranes in which functional nanoparticles are embedded in a GO matrix, making it possible to produce clean water at the permeate side while degrading the organic pollutants in the retentate. A natural humic acid-like substance was used for the covalent crosslinking of GO sheets to obtain membranes that were stable under crossflow conditions. We tested the synergistic interaction of either photocatalytic or thermocatalytic materials (e.g., TiO_2 and Ce-doped SrFeO(3-x), respectively) with GO in the degradation of model contaminants of emerging concern, such as dyes, phenol, and bisphenol A. We were able to fabricate membrane layers with a nanoparticle loading up to 98.6% with the ability to retain and degrade the organic contaminants dissolved in water, simultaneously [2]. Our results enable us to propose simple strategies for the effective design and fabrication of these functional nanocomposite GO-based membranes.

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Oily wastewater treatment using Titanium Carbide composite membrane

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Treatment of oily wastewater such as the produced water (PW) generated by the oil and gas industries remains a pressing challenge. To mitigate this problem, a new ceramic membrane was fabricated for efficient separation of oil-in-water emulsion. The key component in this membrane is the titanium carbide (TiC) selective layer which was affixed onto an alumina ceramic support through interfacial polymerization (IP). The merits of the fabricated TiC@Alumina membrane were thoroughly characterized by different techniques and tested for separation of oil/water (O/W) emulsion. A flux of 600 LMH was achieved at a pressure of 8 bar. The membrane achieved a separation efficiency of >99 % for all of the tested O/W emulsions (125 ppm, 250 ppm and 500 ppm). The long-term stability test revealed that composite membrane maintained excellent separation efficiency (>99 %) for a period of 360 minutes.

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MOF-based mixed matrix membranes for olefin/paraffin separation

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The separation of olefins from their respective paraffins (e.g. ethane/ethylene, propane/propylyne) is among the most energy-intensive industrial separation processes because of the massive scale and because of the high energy requirement for the traditionally used cryogenic distillation. Alternatives have been proposed, such as membrane separation with facilitated transport membranes, but the latter tend to have a low stability and lose their efficiency over time [1]. In the present paper we will discuss the olefin/paraffin separation using mixed matrix membranes based on various metal organic framework (MOF) filllers from the MIL family and the ZIF family, dispersed in a suitable polymeric matrix, such as 6FDA-based polyimides. The membranes are prepared by the solution casting and solvent evaporation method. Single gas permeation measurements are carried out by the time-lag method in a fixed volume setup, yielding the pure propane and propylene permeability and diffusivity, and the ideal selectivities. Light gases (He, H_2, N_2, O_2, CH_4 and CO_2) are tested as well to obtain deeper insight into the gas transport mechanism. The propane/propylene mixed gas permeation was studied with an innovative permeation setup, based on the continuous online analysis of the permeate composition by a quadrupole residual gas analyser, with the unique possibility to determine the diffusion coefficients of the individual gases in the mixture from their permeation transient [2,3]. Argon is used both as the sweeping gas and as an internal standard for the determination of the permeate composition. The method is appropriately calibrated to account for the propylene mass fragment formed upon ionization of propane, in addition to the original permeating propylene. The effect of the MOF type and concentration, the gas composition and the feed pressure on the separation performance of the investigated membranes will be discussed.

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Operation and Scale-Up Conditions Optimization of Ultra-Permeable Block Copolymer-Coated Hollow Fibers for Air Dehydration

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Membrane dehumidification is being considered as an integral part of building air conditioning to provide comfortable ambience, prevent mold growth and elongate the building life. Classically, air is dehydrated with a vapor compression cycle, which uses a significant amount of energy in a two-step super cooling of the air to its dew point and reheated back to ambient temperature. Herein we developed coated hollow-fiber membranes with ultrahigh water transport, implementing a more energy-efficient dehumidification device. The advantage of membrane dehumidification is the lower price, power consumption, and footprint. We favor composite membranes with a porous support and a skin layer that provides the necessary separation and selectivity. In this work, we developed a hollow fibers dehumidification device, starting from the material to the design and manufacturing of the membrane module. We coated porous hollow-fiber membrane with thin selective hydrophilic moieties e.g. sulfonated pentablock copolymer, encapsulated ionic liquid etc. The membrane had 10000 GPU water vapor permeance with 3000 water vapor over nitrogen selectivity vacuum pressure. For the liquid desiccant system, dehumidification at 28°C, mimicking an internally cooled system, was more effective than the isothermal dehumidifier (0.6 kg m^{-2} h^{-1}, 0.73 latent effectiveness, 0.99 Coefficient of Performance COP), guiding towards future system designs for a broad scope of dehydration. For a 500 CFM/ton air flowrate, 10 m^2 total membrane area is sufficient.

Acknowledgements

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Integrated membrane distillation and solvent extraction for water, sulfuric acid, and copper recovery from gold mining wastewater

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New process development and optimization of existing ones are seen as a tool for better use of natural resources. The strategies depart from the linear focus of raw material extraction, transformation, use, and waste disposal, but aim at less dependence on raw material prioritizing recycling and reuse processes. Aligned with our increased demand for mineral resources is the decrease in ore grades in terms of both quality and concentration. The scenario led to the valorization of mining wastewater, seen as a potential source for mineral goods. With that in mind, we integrated the processes of membrane distillation (MD) and dispersive solvent extraction for gold mining wastewater valorization. High-quality water for reuse purposes was firstly recovered by membrane distillation. Its concentrate was then subjected to sulfuric acid and copper recovery by solvent extraction. For being an endothermic process, the sulfuric acid recovery would be favored by the greater temperature in which MD concentrate is produced. Copper, in turn, was selectively recovered (99.3% after a single contact stage) from the raffinate from acid extraction (βCu/Fe = 4,274, βCu/Al = 176, and βCu/Mg = 1,262) by Lix 84-I. The economic assessment was favorable for water (US\$/m^3 0.23) and copper (US\$/kgCu 4.25) recovery, with prices lower than currently practiced for both commodities. Besides the favorable technical results, there are still opportunities for improvement in sulfuric acid reclamation, in which costs for its obtainment were greater than commercially practiced. Nevertheless, the integrated membrane system could be inserted in a perspective that departs from the linear focus of raw material utilization, prioritizing recycling and reuse practices.

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Optimization of design and operation for FO-MD-MCr hybrid system in desalination

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This project aims to develop a low-energy consumption desalination technology using a solar thermal membrane distillation hybrid system (STMDH). High energy cost, severe fouling, and the potential impact of brine are critical challenges in current desalination technologies. To address such challenges, we have established the following goal for the proposed work: "To develop core technologies for solar thermal membrane distillation hybrid system (STMDH)." According to our previous works, membrane distillation (MD), forward osmosis (FO), and membrane crystallization (MCr) are desirable technologies to increase the recovery of brine from a desalination system. However, a key issue is its optimization under various conditions. Another issue is the optimum integration of MD with renewable energy sources. In the proposed research, we have designed and optimized the MD hybrid system combined with solar energy and evaluated its performance under various conditions. From these works, the following objectives are 1) to achieve deriving core technology for FO-MD combination and MCr process, 2) to develop optimal design technology for FO-MD-MCr hybrid processes, and 3) to prove operational technology for Solar-MD based hybrid processes. The achievement of the proposed objectives will allow the application of this technology not only for seawater desalination but also for wastewater reclamation, industrial water production, and resource recovery from various water sources.

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Multifunctional Membranes for the Extracellular Vesicles Capture: New insights into Regenerative Medicine

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Membranes have a great potential in the separation, concentration, and purification of subcellular components such as extracellular vesicles, which play an essential role in intercellular communication acting as signalling vehicles in both physiological and pathological processes (e.g., cancer, neurodegeneration, cardiovascular, inflammatory disease). They represent an important source of circulating biomarkers and it is expected to become the next-generation theranostic tools in precision healthcare and regenerative medicine [1-2]. However, several challenges have to be overcome to fully realise EV potential including those related to the separation and isolation of specific EV populations from other bio-nanoparticles and contaminants found in biological fluids. Here, we propose a strategy aimed at introducing functional ligands over the membrane surface to improve the separation process through combined size-exclusion and affinity-based mechanisms, avoiding the binding of contaminants and other non-target molecules. Polysulfone membranes were modified by a nanometric coating of differently functionalized copolymers with the dual purpose of limiting non-specific interactions while promoting the chemoselective conjugation of a membrane-sensing peptide ligand (BPt) for nanovesicles capture [3]. Copoly azide polymer coating positively affects the physico-chemical properties of the membrane, improving filtration performance and antifouling capacity. Coated membranes were chemoselectively functionalized with the membrane sensing peptide BPt by click chemistry reaction with a density ranged from 26 to 28 μg/cm². The capacity of the BPt-functionalized membranes to capture nanovesicles was assessed in a model experiment by using liposomes in a tangential flow filtration system. The membranes displayed a high capturing efficiency towards nanovesicles which, notably, can be promptly released upon mild treatment with a divalent cation solution. These results may open a new avenue in therapeutics and diagnostics based on extracellular vesicles. The advances in membrane-based EV isolation and purification would greatly contribute to the EV biology understanding and facilitate the translation of EVs in clinical settings.

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Fabrication of Composite Hollow Fiber Membrane Made of Fluorinated Polyimide for Blood Oxygenation

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The blood oxygenators which provide extracorporeal membrane oxygenation and intravenous oxygenation, are used to saturate oxygen levels and eliminate carbon dioxide from the bloodstream. This medical device is made of polypropylene and polymethylpentene polymeric materials. Due to their unique features as strong biocompatibility and chemical resistance, organofluorine compounds are commonly utilized in cardiovascular and plastic surgery, orthopedics, dentistry, and other professions. Fluoropolymers are supposed to have low surface energy, which prevents unwanted protein adsorption, immune response chain events, and blood coagulation. The goal of this research is to synthesize 4,4′-(Hexafluoroisopropylidene) diphthalic anhydride - 4,4'-Oxydianiline (6FDA-ODA) polymer and fabricate a composite hollow fiber membrane out of it by using polysulfone (PSF) as a support. The asymmetric PSF hollow membrane, which is successfully manufactured by dry-wet spining method, is a microporous support. Using a poly-(amic acid) salt precursor solution synthesized by 6FDA and ODA monomers, dip-coating was applied. The composite hollow fiber membrane with fluorinated polyimide was fabricated by thermal imidization process in vacuum oven. Finally, the findings of this study showed that 6FDA-ODA polyimide has a thickness of around 1.5 µm, which is beneficial to blood oxygenation.

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PLGA Membranes with Different Topographical Cues: Effect of Micropatterns on Cell Behaviour

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The introduction of specific and regular topographic patterns on biomaterial surface helps to study the cell behavior in response to defined geometrical cues. Lithographic molding technique is widely used for conferring, to a biomaterial surface, microscale cues able to enhance the performance of a biomedical device. In this study, different positive master molds made by UV mask lithography, and the related negative molds made by PDMS replicas, were used to prepare PLGA micropatterned membranes through phase inversion technique. In brief, PLGA (7.5% w/v) was dissolved in acetone and cast uniformly on the different PDMS molds, dried and then washed with distilled water. Beside the PLGA membranes in flat configuration, several patterned PLGA membranes were prepared in order to present at the cellular interface different topographical features, namely linear channels, circular pillars, rectangular pillars and pits. The effects of geometrical cues were investigated on different cell sources: neuronal cells, myoblasts and stem cells, evidencing peculiar cell behavior in response to the specific topographical stimulus sensed over the membrane surface. Confocal microscopy imaging of cells seeded on linear grooved membranes showed that this cue promoted an elongated cell morphology. Rectangular and circular pillars act instead as a discontinuous cue at cell-membrane interface, inducing a cell growth in multiple direction. The array of pits over the surface, highlighted also a precise spatio-temporal organization of the cells; they grew between the interconnected membrane space within pits avoiding the microscale holes. All the results demonstrated that the geometrical elements over the membrane surface can precisely tune and control in a different way the cell behavior in terms of adhesion, migration and differentiation. The realization biomimetic membranes able to modulate the cell response, helps to point out the mechanism of cell–microenvironment interactions providing new tools for the development of important technologies for advanced tissue engineering.

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Solute transport modeling tools for the application of nanofiltration membranes in minerals recovery processes from seawater desalination brines

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Seawater desalination plants (SWDP) face economic and environmental problems related to the management of the brine generated in the process. However, due to the high concentration of valuable elements in the brine it can be possible to recover them by the integration of several separation and purification technologies. Within this framework, the H2020 Sea4Value project aims to recover Mg(II), B(III), Li(I) and Rb(I), among others, from SWDP brines, making desalination plants the third source of valuable raw materials. Nevertheless, in order to improve the separation and recovery of these raw materials, nanofiltration (NF) is proposed as a pre-treatment technology for the separation of monovalent from multivalent elements [1]. Thus, this work focused on the treatment of SWDP brines using commercial available NF membranes. Experiments were carried out in a flat-sheet configuration (0.014 m^2) to study the effect of transmembrane pressure (6 – 30 bar) on rejections using synthetic brines. In addition, the experimental data was fitted to the Solution-Electro-Diffusion-Film (SEDF) model in order to characterize the transport of species across the NF membranes by means of membrane permeances to species. Experimental results showed that NF membranes could selectively separate monovalent from multivalent ions. The SEDF model was able to fit properly the experimental data and membrane permeances to species were determined. In addition, the mathematical model allowed to estimate the potential scaling at the membrane surface. Finally, membrane permeances were used to predict the performance of the NF membranes as a function of permeate recovery.

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Photothermal Air Gap Membrane Distillation for the Removal of Heavy Metal Ions from Wastewater

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As the world's population grows, pure water becomes increasingly scarce, making wastewater treatment necessary. However, the processes currently used to treat industrial wastewater are generally energy-intensive, highlighting the need to develop environmentally friendly wastewater treatment technologies. To address this issue, a photothermal air gap membrane distillation system was developed and tested for the first time to remove heavy metal ions from wastewater. Activated carbon particles at different concentrations were used in the prepared polyvinylidene fluoride membranes as components for solar energy recovery. Zinc, one of the most common heavy metals in industrial wastewater in the United Arab Emirates, was used as a model contaminant in this study. The results showed that the use of AC particles to modify PVDF membranes can significantly improve solar absorption (97%) and porosity (60.6%), resulting in a permeate flux of 0.32 L/m^{2}h and a rejection of 97% of Zn ions. Moreover, optimized PVDF-AC membrane was tested using synthetic heavy metal solution and showed high rejection rates for most of the tested heavy metals (Zn^{2+}, Fe^{3+}, Cu^{2+}, Cr^{3+}, and Ni^{2+}), hence can be potentially used in industrial heavy metal removal process. The developed photothermal water treatment system can provide a highly efficient and scalable water treatment process. Keywords: Air-Gap Membrane Distillation; Solar; Photothermal; Phase Inversion; Heavy Metal; Activated Carbon; Poly(vinylidene fluoride); Membranes.

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Molecular Separations in Organic Solvents with Polymer Membranes

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Membranes have had a huge impact in molecular separations in aqueous systems, especially desalination. It is generally accepted that 40-70% of capital and operating costs in chemical and pharmaceutical industries are dedicated to separations; and a substantial fraction of this cost is related to processing of organic liquids. Membrane technology has the potential to provide game changing alternatives to conventional concentration and purification technologies such as adsorption, chromatography, liquid extraction, evaporation and distillation, through Organic Solvent Nanofiltration (OSN). The membranes must offer resilience in organic environments, display attractive selectivities, and have good permeance. Ideally they should also be resistant to physical aging and fouling under use.

This presentation will focus on research into advanced membranes for OSN and their applications from class-based separations in crude oil refining to purification of pharmaceuticals [1]. Ultra-thin polymer films have been formed by interfacial polymerisation and then used to fabricate composite membranes. These can be activated by a strong solvent, and have excellent permeance and high rejection. The fascinating crystal morphology of these membranes can manipulated to vary their selectivity, while defined microporosity can be introduced into ultra-thin polymer films through alignment of macrocyclic precursors. Moreover creation of monomers that auto-assemble in solution to nanostructures that are preserved interfacial polymerisation can lead to outstanding performance. Finally integrally skinned asymmetric membranes with surfaces engineered through grafting can lead to membranes with anti-fouling properties [2] which can be deployed in the synthesis of new modality pharmaceuticals including peptides, oligonucleotides, and ADC linkers [3].

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Stability of biobased polyhydroxyalkanoate (PHA) membranes in cleaning solutions

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In order to make membrane fabrication more sustainable, a number of studies have been dedicated to the use of bio-based polymers as an alternative to fossil-based ones. Contributing to this effort, it has recently been demonstrated the possibility to use polyhydroxyalkanoates (PHA) as a suitable bio-based polymer family for membrane fabrication. PHA are biodegradable bacterial polyesters produced by fermentation. In particular, poly(hydroxybutyrate-co-hydroxyvalerate) (PHBHV) has been used as raw material for the fabrication of microfiltration, ultrafiltration and pervaporation by the phase inversion technique [1-2]. However, the stability of these membranes on time during use remains questionable, especially when subjected to cleaning/disinfection solutions. In this context, this work aims at investigating the stability of PHBHV membranes in contact with these aggressive solutions. Membranes were prepared by phase inversion using commercial PHBHV having different ratio of hydroxyvalerate monomers (range of 1-25% (mol)) in the polymer structure. Dense to microporous membranes were fabricated by adjusting the molecular weight and concentration of polyethylene glycol used as a pore forming additive. The membranes obtained were further exposed by soaking in different cleaning solutions including sodium hypochlorite, sodium hydroxide and sulfuric acid at different concentration, pH and exposure durations. The membranes were characterized before and after exposure in terms of surface charge (streaming current), surface chemistry (ATR-FTIR) and microstructure (SEM). Filtration tests were performed to determine their permeability to pure water and rejection ability.

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The permeation number - A dimensionless group for membrane operation design

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The permeation follows a diffusion mechanism in dense membrane layers or a convective one in porous membranes. The flow passing through the membrane separating layer, whatever the mass transport mechanism inside it is, has to be compared with the feed flow investing the membrane. A parameter for comparing the flux (outside the membrane layer) insisting on feed membrane interphases and that permeating (inside the membrane layer), enabling a "key" information for predicting the permeation performance of a membrane with known permeation properties (permeance) would be very useful for the appropriate design of a membrane permeator. The permeation through a membrane depends on several factors, some of them related to feed and permeate pressure ratio and feed/retentate and permeate concentrations, others correlated to the permeance and surface area of the membrane, feed flow and feed pressure. We grouped the latter all together in a dimensionless number, here identified as permeation number, which compares the feed flow investing the membrane surface and the feed fraction capable of permeating under a specific feed pressure. The ratio between these two elements indicates how adequate is the ability of the membrane to be permeated in comparison with the feed flow rate. It ranges 0-infinity depending on the parameters contributing to its evaluation. A value equals one, means that the whole feed stream permeates the membrane; a value <1 means that the feed stream is higher than that can permeate the membrane and only a fraction permeates the membrane. A linear relationship exists for values <=1. For any value >1 the permeation is complete. Pressure ratio, feed and permeate concentrations change this ideal condition of feed single component increasing the membrane request. The permeation number values are useful to design the permeation desired also considering recovery and purity as target parameters.

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A new method to reveal the mechanism of CO inhibition on H_2 flux through ultra-thin Pd/Ag membrane

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While it is generally established that CO reduces the H_2 permeation due to competitive adsorption, it has been reported that CO may also cause irreversible degradation [1, 2]. The temperature and CO concentration play a role and mass transfer limitations make the problem more complicated to study. In the present work we investigated the inhibition effect of CO on hydrogen permeation by combining permeation experiments and X-ray photoelectron spectroscopy (XPS) instrumentation equipped with a reaction cell. The permeation measurements were performed with ~4 µm thick Pd/Ag23wt% membranes in a microchannel configuration where the influences of concentration polarization and H_2 depletion are minimized (80% H_2 in 400 Nml/min at 1 bar relative pressure). The CO effect was strongly dependent on both temperature (300–400ºC) and CO partial pressure (0–5 mol%). The flux cannot be fully regenerated by replacing CO with N_2. At 400ºC, a sharp initial drop is followed by a gradual decrease of flux, while only the sharp initial drop was observed at 300 and 350ºC. The largest flux reduction found is 83.5% at 300ºC with 5% CO. The XPS reaction cell allows for exposure to realistic conditions and subsequent analysis without exposing the sample to air. XPS was carried out on fresh membrane, as well as membrane that had undergone heat treatment in air (HTA), H_2 activation and H_2/CO exposure (H_2:CO = 16); all under the same conditions as corresponding permeation tests. HTA caused a surface enrichment of Pd from 50% to 72%, while subsequent H_2 activation lowered the Pd/Ag ratio from 6 to 1.7. After H_2/CO exposure, the carbon content is increased from 5 to 12 % (atomic), which infers that strongly adsorbing carbon species were formed during the exposure.

Acknowledgements

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Innovative membrane systems for CO_2 capture and storage at sea (MemCCSea ACT-ERANET project)

Asimakopoulou Akrivi[1]*, Koutsonikolas Dimitrios[1], Damartzis Theodoros[2], Pantoleontos Grigorios[1], Skevis Georgios[1], Georgopoulou Chara[3], Dimopoulos George[3], Bougiouris Konstantinos[4], Richter Hannes[5], Lubenau Udo[6], Economopoulos Solon[7], Perinu Cristina[7], Hopkinson David[8], Panagakos Grigorios[8,9]

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Carbon Capture Storage and Utilization (CCS/CCU) is increasingly considered as a competitive option for the decarbonization of energy intensive industries (cement, steel etc.) and long-distance transportation, including aviation and shipping. In the maritime industry, CCS/CCU is envisaged as one of the technological solutions within the decarbonization portfolio that could contribute to achieving IMO's goal to cut at least half annual GHG emissions from international shipping by 2050, compared with their level in 2008. The technical feasibility of CCS onboard ships has been proven through studies and pilots of small capture rates, and there is potential for larger reduction rates through new materials, design and process integration. CCS technology can readily be integrated with the conventional marine engine combustion powerplant and adapted for operation with alternative fuels. The MemCCSea project is among the very few projects world-wide that aim to develop carbon capture technologies specifically designed and optimized for the maritime sector and define strategies for its integration in the ship powerplant (marinization), even by retrofitting in existing ships enabling quick CCS deployment. Innovative solvents, membrane materials and processes for Ceramic Gas-Liquid Membrane Contactors and Polymeric Mixed Matrix Membrane Permeators have been developed and evaluated in lab- and pilot-scale experimental facilities and through extensive modelling and simulation at component and system levels. The current presentation describes the carbon capture system design, performance characteristics of membrane and solvent materials, and ship integration considerations for a maritime application of CCS.

Acknowledgements

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Ultraselective and ultrapermeable tunable nanostructured stainless-steel coated membranes for oil/water emulsion separation

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This paper demonstrates for the first time the sputter-coating of stainless-steel on mixed cellulose ester (MCE) membranes for use in the separation of oil/water emulsions. A stainless-steel coating with nano-pyramid structures (diameter of ~20-50 nm and pore size of 156.1 nm) on an MCE membrane presented a superhydrophilic and antifouling surface with underwater superoleophobicity property. The coated membrane achieved separation efficiency of >99% when applied to light oil-water emulsions with a range of viscosities and densities, and the highest ever permeation flux of 1595 L m^{-2} h^{-1} when applied to toluene-in-water emulsions. The membrane also presented outstanding recyclability, as evidenced by oil rejection of >99% through four cycles. The coated membrane was shown to work well under harsh conditions, including salty water, extreme pH values (1 to 14), and high temperatures (60 °C). The membrane also presented high selectivity and permeation flux of not less than 972 L m^{-2} h^{-1}. The process of fabricating stainless-steel coatings using our facile sputtering method at room temperature is highly scalable.

PVDF-HFP polypyrrole-decorated nanocomposite fibrous membranes for the electrochemical detection of lactic acid

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When considering different diagnostic tools for healthcare monitoring, biosensors have emerged as a compelling route in the transitioning from invasive to non or minimally invasive practices. A biosensor typically consists of a biorecognition element (conventionally, enzymes or antibodies), a transducer component (semiconducting materials), and an electronic system [1]. The biorecognition element; the constituent of a biosensor responsible for the analyte recognition, delineates the selectivity and, in parts, the sensitivity of a biosensing platform [2]. Molecularly imprinted polymers (MIPs) have emerged as valuable alternatives to natural receptors as recognition materials, owing to their synthetically acquired predetermined selectivity towards a given analyte, their physical and chemical stability under harsh operating conditions, and biomimicry [3]. Although the evolvement of MIP technologies has seen great success in the past decades; their inflexibility hampers integration into biosensing devices. Electrospinning is a well-established method capable of producing diverse forms of fibrous assemblies with ease in manipulating the composition and properties of the formed nanofibers (NFs) [4]. Herein, we describe an adaptable method for the in situ synthesis of polypyrrole MIPs onto poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) NFs employing lactic acid as the template molecule. The PPy–decorated NFs, alter the hydrophobic nature of the PVDF into an absorbent, superhydrophilic composite. The work proceeds by assessing the aptitude of this nanocomposite to selectively detect the presence of lactic acid via spectroscopic and electrochemical methods. This work outlines a novel approach capable of uniting electrospinning and molecular imprinting to develop advanced nanocomposite conductive membranes with enhanced spatiotemporal configuration while overcoming difficulties associated with incorporating MIPs within analytical devices.

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Carbon molecular sieve membranes for water separation in CO_2 hydrogenation reactions: effect of the carbonization temperature

Poto Serena*[1], Aguirre Alejo[2], LLosa Tanco Margot A.[3], Pacheco Tanaka David A[3], Gallucci Fausto[1], Neira d'Angelo M. Fernanda[1]

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The separation of water from (H2-rich) gaseous mixture is a topic of increasing interest in the context of CO_2 valorization, where in-situ removal of water improves both reaction performance and catalyst stability. Carbon membranes are a potentially attractive candidate for this application because of their affinity to water [1]. As a matter of fact, their hydrophilicity and pore structure can be tuned by properly adjusting the synthesis procedure. Herein, we assess the effect of the carbonization temperature (450-750 ºC) on the performance of supported CMSM in terms of vapor/gas separation, in correlation with changes in their surface functionality and porous structure. FTIR spectra showed that the nature of the functional groups changes with the evolution of the carbonization step, leding to a gradual loss in hydrophilicity (i.e., OH stretching disappears at Tcarb ≥ 550 ºC). The extent of water adsorption displays an optimum at Tcarb of 550 ºC, which proves that, besides hydrophilicity, surface area also plays a key role in the adsorption of water. Indeed, the membrane surface area initially increases with Tcarb, due to the development of the pore structure. We found that the pore size distribution strongly influences the water permeance. At all Tcarb, adsorption-diffusion (AD) is the dominant transport mechanisms. However, as soon as ultra-micropores appear (Tcarb: 600-700 ºC) [2], molecular sieving (MS) contributes to an increase in the water permeance, despites a loss in hydrophilicity. At Tcarb ≥ 700 ºC, MS pores disappear, causing a drop in the water permeance. Finally, the permeance of different gases (N_2, H_2, CO, CO_2) is mostly affected by the pore size distribution, with MS being the dominant mechanism over the AD, except for CO_2. However, the extent and mechanism of gas permeation drastically change as a function of the water content in the feed, affecting the vapor/gas perm-selectivity.

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The special Edition of

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Posters Presentations

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EMS 1982

ALMA MATER STUDIORUM UNIVERSITÀ DI BOLOGNA

Poster Session I, Monday, 21 November 2022

Chairs: Enrico Drioli & Roger Ben Aim

Session: Membrane materials

PI_1. Preparation and characterization of semi-alicyclic homo-, co- and blend polyimides membranes using alcyclic dianhydrides and their hydrogen separation properties
Jeong-Hoon, Kim Chae-Hee Seo, Hoon-Moh Seong, Si-Woo Lim, Bo-ryoung-Park, Bong-Hun Chang, Jeong-Hoon Kim (Korean Research Institute of Chemical Technology, Korea)

PI_2. Towards sustainable manufacturing of electrospun PVDF mixed matrix membranes for water treatment and purification
Antonios Keirouz[1], Garyfalia Zoumpouli[1], Dinesh Mahalingam[1], Bernardo Castro Dominguez[1], *Davide Mattia[1] and Hannah Leese[1], Francesco Galiano[2], Francesca Russo[2], Maria Francesca Vigile[2], Enrica Fontananova[2], Alberto Figoli[2] ([1]University of Bath, United Kingdom; [2]CNR-ITM, Italy)*

PI_3. Electrospun poly(vinylidene fluoride-hexafluoropropylene) (PVDF-HFP)-CNT-GO nanocomposite membranes for Membrane Distillation Applications
Lassaad Gzara[1], Abdalrhman Taha Alsaggaf[1, 2], Mohamed Abdel Salam[2], Hussam Organji[1], Alberto Figoli[3] ([1]Center of Excellence in Desalination Technology and [2]Chemistry Department, King AbdulAziz University, Saudi Arabia; [3]CNR-ITM, Italy)

PI_4. Fabrication of Poly(vinylidene fluoride-hexafluoropropylene) (PVDF-HFP) nanofiber membranes by electrospinning process for Membrane Distillation Applications
Lassaad Gzara[1], Amjad Hazza[1,2], Nazeeha Alkayal[2], Hussam Organji[1], Alberto Figoli[3] ([1]Center of Excellence in Desalination Technology; [2]Chemistry Department, King AbdulAziz University, Saudi Arabia[3]; CNR-ITM, Italy)

PI_5. Highly permeable polyamides of intrinsic microporosity (PAIMs) with tunable molecular weight cut-offs for organic solvent nanofiltration (OSN)
Liu Dezhi, Malpass-evans Richard, Liu Tianyin, Mckeown B Neil, Song Qilei, Livingston Andrew (Imperial College London and Queen Mary University of London, United Kingdom)

Session: 2D materials

PI_6. Control of graphene oxide membrane performance in water filtration by compaction during fabrication
Miguel A. Gilarranz, Fernàndez-Màrquez M., Baeza J. A., Pla R., Calvo L., Alonso-Morales N., Gilarranz M. A. (Department of Chemical Engineering, Universidad Autónoma de Madrid, Spain)

PI_7. Engineering Superior Multi-Functionalized Holey Graphene toward Ultra-permeable Membrane for Emulsified Oil/water Separation

Hegab Hanaa M.[1,2], Elmekawy Ahmed [1,2], Aubry Cyrilc, Al Marzooqia Faisal [1,2], Aljundid Isam H.[4,5], Wadi Vijay S. [1,2], Yousef Ahmed F.[1,6], Banat Fawzi [1,2], Hasan Shadi W.[1,2] ([1]CMAT, UAE; [2] Khalifa University of Science and Technology, UAE; [3]Laboratories Operations, Khalifa University of Science and Technology, UAE; [4] King Fahd University of Petroleum and Minerals, Saudi Arabia; [5]King Fahd University of Petroleum and Minerals, Saudi Arabia.

PI_8. Effect of Graphene Oxide on Liquid Water-Based Waterproofing Bituminous Membranes
Giuseppe Di Luca[1], Marcello Filomia[2], Alessio Fuoco[1], Giovanni Chiappetta[1], Alberto Figoli[1] ([1]*CNR-ITM, Italy, [2]Research and Developed Department, HA ITALIA S.p.A, Italy)*

Session: Polymer, biopolymer membranes

PI_9. Changes of mechanical and spectroscopical characteristics of glassy polymeric membranes due to physical ageing
Durďáková Tereza-Markéta[1], Král Martin[1], Hrdlička Zdeněk[2], Budd Peter M.[3], Harrison Wayne[3], Friess Karel[1], Vopička Ondřej[1] ([1]UCT, Department of Physical Chemistry, Czech Republic, [2]UCT Department of Polymers, Czech Republic, [3]The University of Manchester, UK)

PI_10. Properties of polyhydroxyalkanoate membranes formed using non-halogenated solvents
Wang Liang-Shin, Borneman Zandrie, Nijmeijer Kitty, Werker Alan (Eindhoven University of Technology, The Netherlands; European Centre of Excellence for Sustainable Water Technology, The Netherlands)

PI_11. Understanding the non-solvent induced phase separation (NIPS) parameter effect during the fabrication of microporous DOCDA membranes
Kim Min-Ha, Lee Pyung-Soo (Chung-Ang University, Republic of Korea)

PI_12. Dope temperature influence on structure and performance parameters in an industrial PES UF NIPS hollow fiber process
Ley Adrian, Rohleder Darius, Grǜnig Lara Elena, Volkmar Thom (Sartorius Stedim Biotech GmbH, Germany)

PI_13. Study of spinning parameters of polyvinylidene fluoride (PVDF) hollow fiber membranes preparation for membrane distillation
Tournis Ioannis, Theodorakopoulos George V., Karousos Dionysios S., Boutsika Lamprini G., Kouvelos Evangelos P., Sapalidis Andreas A. (Institute of Nanoscience and Nanotechnology, National Center for Scientific Research, Greece; National and Kapodistrian University of Athens, Greece; National Technical University of Athens, Greece)

PI_14. Development of hydrophilic Polyvinylidene fluoride-based membrane by blending with Alkali Lignin
Regina Serena, Poerio Teresa, Mazzei Rosalinda, Giorno Lidietta, (CNR-ITM, Italy)

PI_15. Water selective membranes for use in electrocatalytic CO2 reduction to syngas
Boukili Aishah, Karvan Oguz, and Visser Tymen (EMI Twente, University of Twente, the Netherlands)

PI_16. Molecular layer deposition for the fabrication of desalination membranes with tunable metrics
Welch Brian, McIntee Olivia, Myers Tyler, Greenberg Alan, Bright Victor, George Steven (University of Colorado, USA; Technion − Israel Institute of Technology, Israel)

Session: Mixed matrix membranes

PI_17. Fabrication of high-performance CO2-philic mixed matrix membrane via interfacial blueprinting of the MOFs and polymer matrix for CO_2 capture applications
Floreková Jana, Ashtiani Saeed, Friess Karel (University of Chemistry and Technology, Czech Republic)

PI_18. Interfacial engineering of electrospun polyvinyl alcohol/carbon nanotube mats into dense films for sustainable alcohol dehydration
Su Kyung Kang[1,+], Kyujin Hwang[2,+], Ju Won Park[1], Min Ha Kim[1], Pyung Soo Lee[1,3,],Soo-Hwan Jeong[2,4] ([1]Department of Chemical Engineering and Material Science, Chung-Ang University, Republic of Korea, [2]Department of Chemical Engineering, Kyungpook National University, Republic of Korea, [3]Department of Intelligent Energy and Industry, Chung-Ang University, Republic of Korea, [4]Department of Hydrogen & Renewable Energy, Kyungpook National University, Republic of Korea)

PI_19. Selenium modified graphitic carbon nitride membrane for antibacterial properties
Sibiya D.[1], Dlamini B.C.[2], Malinga S.P.[1] ([1]University of Johannesburg, South Africa; [2]Department of Biotechnology and Food technology, University of Johannesburg, South Africa)

PI_20. Modification of cellulose acetate ultrafiltration membrane with zeolitic imidazole framework-8
Vahid Vatanpour, Ayse Yuksekdag, Meltem Ağtaş, Ismail Koyuncu, Gulmire Ilyasoglu (National Research Center on Membrane Technologies, Turkey and Kharazmi University, Iran)

PI_21. Mixed matrix membranes with MOFs for removal heavy metals from water
Monteleone Marcello[1], Jansen Johannes C.[1], Longo Mariagiulia[1], Esposito Elisa[1], Fuoco Alessio[1], Mastropietro Teresa Fina[2], Armentano Donatella[2], Rita Maria Percoco[2], Emilio Pardo[3] ([1]CNR-ITM, Italy, [2]University of Calabria, Italy, [3]Instituto de Ciencia Molecular (ICMol), Universidad de Valencia, Spain)

PI_22. Study of nanocarbon-based mixed matrix flat sheet membranes preparation for CO_2/CH_4 separation
Vasileiou Athanasios N.[1], Theodorakopoulos George V.[1,2], Karousos Dionysios S.[1], Sapalidis Andreas A.[1], Favvas Evangelos P.[1] ([1]Institute of Nanoscience and Nanotechnology, National Center for Scientific Research, Greece; [2]School of Chemical Engineering, National Technical University of Athens, Greece)

PI_23. Addition of different powder activated carbon (PAC) in non-solvent induced phase-inversion membranes: effect and characterisation.
Raúl Mompó-Curell[1], Simbarashe Biti[2], Alicia Iborra-Clar[1,3], María Isabel Iborra-Clar[1,3], Esperanza M. Garcia-Castello[1,4]; Claudia Fernández-Martín[2,5]([1]ISIRYM, Universitat Politècnica

de València, Spain; [2]University of Aberdeen, United Kingdom; [3]Department of Chemical and Nuclear Engineering, Universitat Politècnica de València,, Spain; [4]Institute of Food Engineering for Development (IIAD), Universitat Politècnica de València, Spain; [5]Centre for Energy Transition, University of Aberdeen, United Kingdom)

PI_24. Impact of layered perovskite oxide on pervaporation performance of polymer membranes
Pulyalina Alexandra, Rostovtseva Valeriia, Faykov Ilya, Polotskaya Galina (Saint Petersburg State University, Russia)

PI_25. Organic solvent separation using a hybrid of size-controlled porous organic polymer and polyethersulfone membrane *Mafika A.[1], Smith V.[2], Arderne, C.[1], Vatsha B.[1] ([1]University of Johannesburg, South Africa; [2]Rhodes University, South Africa)*

PI_26. Synthesis and Gas Permeation Properties of the 2D ZIF-8 and PEBAX Mixed Matrix Membranes *Sung Kuk Jeong, Semin Lim, Jinsoo Kim (Department of Chemical Engineering, Kyung Hee University, Korea)*

Session: Inorganic membranes

PI_27. Development and Characterization of High Porosity Recrystallized Silicon Carbide Membrane Substrate
Wang Yilin, Yuan Linfeng (Landson Emission Technologies A/S, Denmark)

PI_28. Scaling-up of zeolite ZSM-5 membranes preparation for natural gas treatment
Hannes Richter[1], Adrian Simon[1], Jan-Thomas Kühnert[1], Ingolf Voigt[1], Udo Lubenau[2], Raymond Mothes[2], Andreas Ossmann[2], Sebastian Wohlrab[3] ([1]Fraunhofer IKTS, Germany, [2]DBI-GUT GmbH, Germany, [3]LIKAT, Germany)

PI_29. A New Through-the Membrane-Flow Atomic Layer Deposition Reactor Design Used for the Development of Ceramic Nanofiltration Membranes
Nijboer Michiel, Chen Mingliang, Batenburg Kevin, Aarnink Tom, Jan Asif, Heijman Bas, Roozeboom Fred, Kovalgin Alexey, Nijmeijer Arian, Luiten-Olieman Mieke (University of Twente, The Netherlands; Delft University of Technology, The Netherlands)

PI_30. Semi-flexible ultrafiltration inorganic membrane based on GO and ultra-long hydroxyapatite for wastewater purification
Gomaa Mohamed G.[1] Russo Francesca[2], Frappa Mirko[2], Macedonio Francesca[2], Figoli Alberto2, Galiano Francesco[2], Abdel-Ghafar Hamdy M.[1] ([1]Central Metallurgical Research and Development Institute (CMRDI), Egypt; [2]CNR-ITM, Italy)

PI_31. Influence of silicon carbide content and temperature on mechanical and electrochemical characteristics of ceramic membranes based on kaolin and saponite
Dontsova Tetiana, Yanushevska Olena, Molchan Ylia (National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute", Ukraine)

Session: Membranes in biotechnology and agrifood processes

PI_32. Fabrication of polyelectrolyte complex hollow fiber membranes through salt dilution induced phase inversion
Zhao Ang, Emonds Stephan, Restrepo Maria A., Roth Hannah and Wessling Matthias (AVT.CVT - Chemische Verfahrenstechnik (RWTH Aachen University), Germany)

PI_33. Bio-inspired Membranes with Directional Water Transport Characteristics
Grillo Luca, Weder Christoph (Adolphe Merkle Institute, Switzerland)

PI_34. Magnetic hybrid separation materials with controlled properties – covalently modified PVDF with lanthanides oxide particles
Joanna Kujawa[1], Wojciech Kujawski[1], Samer Al. Gharabli[2] ([1]Nicolaus Copernicus University, Poland; [2]Pharmaceutical and Chemical Engineering Department, German Jordanian University, Jordan)

PI_35. NMR and MS characterization of apple juice and partial removal of sugar by dia-nanofiltration process
Gaglianò Martina[1], Conidi Carmela[2], De Luca Giuseppina[1], Cassano Alfredo[2] ([1]University of Calabria, Italy; [2]Institute on Membrane Technology (CNR-ITM), Italy)

PI_36. Microfiltration of milk: Linking membrane fouling with the colloidal properties of milk
Mardal Freja[1,2], R. Parjikolaei Behnaz[2], Corredig Milena[1] ([1]Aarhus University, Denmark; [2]Arla Foods Ingredients Group P/S, Denmark)

PI_37. Sequential ultrafiltration for the fractionation of lignin and galactoglucomannan from acidic Norway Spruce steam explosion liquors
Al-Rudainy Basel, Wallberg Ola (Lund University, Sweden)

PI_38. LIBERATE: Nanofiltration for the recovery of lignin and depolymerization products
Sonia Matencio[1], Marcel Boerrigter[1], Kyra van Dijk[2], Joris de Grooth[2] ([1]LEITAT Technological Center, Spain; [2]NX Filtration, The Netherlands)

PI_39. The performance comparison and troubleshooting of PVDF transfer membranes using different chemiluminescent substrates
Mehrabi Najmeh, Marta Bonara (GVS filter technology S.p.A, Italy)

PI_40. Nanofiltration and ultrafiltration of sugar beet molasses used for the production of hydroxymethylfurfural
Sjölin Mikael, Sayed Mahmoud, Wallberg Ola, Lipnizki Frank (Lund University, Sweden)

PI_41. Nanofiltration in purification of hemicellulosic sugars in biorefinery side streams
Valkama Hanna, Rathnayake Buddhika, Ohenoja Markku, Keiski Riitta L. (University of Oulu, Finland)

PI_42. Novel paths towards sustainable membrane manufacturing in industrial scale
Greie Lea[1,2,3], Grünig Lara Elena[1], Rohleder Darius[1], Ley Adrian[1], Roth Hannah[2,3], Tepper Maik[2,3], Restrepo Maria[2,3], Zhao Ang[2,3], Wessling Matthias[2,3] ([1]Sartorius Stedim Biotech

GmbH, Germany; [2]RWTH Aachen University, Germany. [3]DWI, Institut für Interaktive Materialien, Germany)

PI_43. Membrane processes for fractionation of bioactive compounds extracted from tomato leaves and used in vivo to induce plant defense
Bazzarelli Fabio[1], Mazzei Rosalinda[1], Papaioannou Emmanouil[2], Giannakopoulos Vasileios[3], Roberts Michael R.[3], Giorno Lidietta[1] ([1]Institute on Membrane Technology (CNR-ITM), Italy; [2]Engineering Department, Lancaster University, UK; [3]Lancaster Environment Centre, Lancaster University, UK)

Session: Nanostructured and multifunctional membranes

PI_44. Rawmina: Integrated innovative pilot system for Critical Raw Materials recovery from mines wastes in a circular economy context
Morillo Diego, Matencio Sonia, Gispert Joaquim (LEITAT Technological Center, Spain)

PI_45. Exfoliation of MoS2 nanosheets using stimuli responsive poly (N-isopropylacrylamide-co-allylamine) for multipurpose membranes
Keshebo Degu Lere, Darge Haile Fentahun, Hu Chien-Chieh, Juin-Yih Lai (National Taiwan University of Science and Technology, Taiwan)

PI_46. Innomem project: open innovation test bed for nano-enabled membranes
Jon Zuńiga[1], Adele Brunetti[2] ([1]Tecnalia, Basque Research and Technology Alliance (BRTA), Spain [2] CNR-ITM, Italy)

PI_47. Molecularly imprinted membranes in advanced separations
Donato Laura, Enrico Drioli (CNR-ITM, Italy)

Session: Facilitated transport membranes

PI_48. Removal of Pharmaceutical Contaminants from Aqueous Water: Diclofenac Case Study
Farah M., Fabregat A., Fortuny A. (University of Rovira I virgili-Tarragona Spain)

PI_49. CO_2 removal from a low partial pressure environment
Mendes Felipe[1], Borges Cristiano[2] ([1]Brazillian Navy Research Institute, Brazil; [2] Federal University of Rio de Janeiro, Brazil)

PI_50. Three-phase nanocomposite membranes containing guanidine-based mobile carriers for CO_2/H_2 separation
Xu Wenqi, Lindbråthen Arne, Deng Liyuan (Department of Chemical Engineering, Norwegian University of Science and Technology (NTNU), Norway)

PI_50a *Extraction and recovery of rare earth elements by a polymer inclusion membrane*
Malki Mohamed[1,2], Sahmoune Amar[2], Fontàs Clàudia[1] (Universitat de Girona, Spain; Université Mouloud Mammeri de Tizi Ouzou, Algeria)

Session: Membrane surface modification/membrane functionalization

PI_51. Low-fouling coated PES and PVDF membranes for seafood wastewater treatment
Vigile M.F.[1], Galiano F.[1], Russo F.[1], Tran Le L.[2], Figoli A.[1] ([1]CNR-ITM, Italy; [2]Vietnamese-German University, Viet Nam)

PI_52. Modification of PVDF/PSF hollow fiber membranes
Dong Zou, Hyun Woo Kim, Seong Min Jeon, Joon Yong Bae, Young Moo Lee (Department of Energy Engineering, Hanyang University, Seoul, Korea)

PI_53. In-situ hydrophilization of hollow fiber membranes using electron beam grafting
Konrad Leopold, Daniel Breite, Andrea Prager, Lutz Drößler, Steffen Weiß, Dirk Enk, Agnes Schulze (Leibniz Institute of Surface Engineering (IOM), Germany; [1]Institute of Chemical Technology, Leipzig University, Germany)

PI_54. Effect of amine modification on CO2 separation performance of commercial silica membranes
Grekou Triantafyllia, Koutsonikolas Dimitrios, Karagiannakis George, Kikkinides Eustathios (Aristotle University of Thessaloniki (AUTH), Greece, Chemical Process & Energy Resources Institute (CPERI), Centre for Research & Technology Hellas (CERTH), Greece)

PI_55. Novel antifouling surface grafted membranes for wastewater treatment
Sandru E. M.[1], Guzman Gutierrez M.T. [1], Sandru M.[1], Thelin W. R.[2], Azrague K. [2]; Seglem K.N.[2] ([1] Sintef Industry, Norway [2]; Sintef Community, Norway)

PI_56. Systematic development of a low fouling modification for micro and ultrafilter polyethersulfone membranes
Weissbach Sebastian, Rohleder Darius, Ley Adrian, Thom Volkmar, Ulbricht Mathias (Sartorius Stedim Biotech GmbH, Germany; Lehrstuhl für Technische Chemie II, Universität Duisburg-Essen, Universitätsstrasse, German)

PI_57. Waltzing around the stereochemistry of membrane crosslinkers for precise molecular sieving in organic solvents
Abdulhamid Mahmoud, Hardian Rifan, Szekely Gyorgy (King Fahd University of Petroleum and Minerals)

PI_58. Potential of PVDF-chitosan hybrids in water purification contaminated by pharmaceuticals – paracetamol
Joanna Kujawa[1], Wojciech Kujawski[1], Samer Al. Gharabli[2] ([1]Nicolaus Copernicus University in Toruń, Poland; [2]German Jordanian University, Jordan)

PI_59. Fabrication of functionalized hollow fiber membranes by e-beam irradiation
Kyra van Dijk, Herman Teunis, Heleen Sombekke, Tymen Visser (EMI Twente, University of Twente, The Netherlands)

PI_60. Fine-tuning of carbon nanostructures/alginate nanofiltration performance: Towards electrically-conductive and self-cleaning properties
Aburabie Jamaliah; Nassrullah Haya; Hashaikeh Raed (New York University Abu Dhabi, UAE)

Session: Mass transport in membranes

PI_61. Modelling protein fractionation with Ultrafiltration
Suryawirawan Eric, Janssen Anja, van der Padt Albert (Wageningen University, The Netherlands and Institute for Sustainable Process Technology, The Netherlands)

PI_62. Advanced methods for the analysis of mixed gas diffusion in polymeric membranes
Monteleone Marcello[1], Fuoco Alessio[1], Esposito Elisa[1], Rose Ian[2], Chen Jie[2], Comesaña-Gándara Bibiana[2], Bezzu C. Grazia[3], Carta Mariolino[4], McKeown Neil B[2], Shalygin Maxim G.[5], Teplyakov Vladimir V. [5], Jansen Johannes C[1] ([1]CNR-ITM, Italy [2] EaStCHEM, University of Edinburgh, UK[3]; Cardiff University, UK [4]; Swansea University, Swansea, UK [5]; A.V. Topchiev Institute of Petrochemical Synthesis, Russia)

PI_63. Easy tool for prediction of separation performance
Mazza Ivan, Brunetti Adele, Barbieri Giuseppe (CNR-ITM, Italy)

PI_64. Osmotic Membrane Distillation Crystallization of $NaHCO_3$
Mar Garcia Alvarez, Vida Sang Sefidi, Marine Beguin, Alexander Collet, Raul Bahamonde Soria and Patricia Luis (UCLouvain, Belgium)

Session: Modelling and simulation in membrane science & engineering

PI_65. Effect of water content on the properties of the polymer electrolyte membrane
Park Chi Hoon, Kang Hoseong (Gyeongsang National University (GNU), Republic of Korea)

PI_66. Molecular dynamics simulation of alkaline ionomer under excessive water condition for water electrolysis
Kang Hoseong, Park Chi Hoon (Gyeongsang National University (GNU), Republic of Korea)

PI_67. Advanced control of Reverse Osmosis with a data-driven fouling model
Gaublomme Dorien[1,2], Vanoppen Marjolein[1,3], De Gusseme Bart[1,4], Verliefde Arne[1,3], Nopens Ingmar[1,2], Torfs Elena[1,2] ([1]Centre for Advanced Process Technology for Urban Resource recovery (CAPTURE), Belgium [2] BIOMATH, Belgium [3] PaInT, Ghent University, Belgium; [4]FARYS|TMVW, Belgium; [4]CMET, Department of Biotechnology, Ghent University, Belgium)

PI_68. Multi-ion physical modeling of nanofiltration/low-pressure reverse osmosis with and without ozonation pretreatment
Penglin Fan, Anthony Szymczyk, Pierre-François Biard, Biard, Lydie Paugam (Univ Rennes, CNRS, ISCR Institut des Sciences Chimiques de Rennes, France)

PI_69. Food & Beverage Concentration Process Design and Optimization Using Aquaporin Forward Osmosis Membranes
Jafari Mehryar[1], Michelon Nicola[2], Ming Xie[1] ([1] University of Bath, United Kingdom; [2] Aquaporin A/S, Denmark)

Session: Multi-scale modelling in membrane science & engineering

PI_70. Modelling of CO2 Adsorption in PIM-1 Membranes using a Multiscale approach combining Monte Carlo technique, Molecular Dynamics and Reactive Vacancy Solution Theory
Alessandra Iazzolino[1], Alessio Caravella[1,2], Elena TOCCI[2] ([1]DIMES University of Calabria (UNICAL), Italy; [2] CNR-ITM, Italy)

PI_71. Ion diffusion in membranes: a multiscale modelling starting from molecular scale
Luque Di Salvo Javier[1], De Luca Giorgio[2], Cipollina Andrea[3], Luque Guillermina Leticia[1], Fuoco Alessio[2], Leiva Ezequiel Pedro Marcos[1], Micale Giorgio[3]([1]INFIQC, Universidad Nacional de Córdoba, CONICET, Ciudad Universitaria, Argentina; [2]CNR-ITM, Italy; [3] Università degli Studi di Palermo, Italy)

Session: Computational modelling in membrane science & engineering

PI_72. Characterisation of fouling behaviour during liposome sterile filtration using CFD modelling
Tan Ruijie, Johnson Thomas, Welsh John, Bracewell Daniel (University College London (UCL), UK)

PI_73. Effect of ion pairing on ion transfer through sub-nanometer CNTs
Neklyudov Vadim, Viatcheslav Freger (The Wolfson Faculty of Chemical Engineering, Israel)

PI_74. Establishing structure-property linkages for wicking time predictions in porous polymeric membranes by data processing workflows
Kunz Willfried, Altschuh Patrick, Selzer Michael, Nestler Britta (Institute of Digital Materials Science (IDM), Hochschule Karlsruhe - University of Applied Sciences, Germany Institute of Applied Materials Microstructure Modelling and Simulation (IAM-MMS), Karlsruhe Institute of Technology (KIT), Germany)

PI_75. Ion Transport Mechanisms in Polymers and Membranes
Jay Prakash Singh, Viatcheslav Freger (Israel Institute of Technology, Israel)

PI_76. Noncovalent interactions between SARS-CoV-2 spike and membrane polymer surfaces
De Luca Giorgio[1], Petrosino Francesco[2], Luque Di Salvo Javier[3], Chakraborty Sudip[2], Curcio Stefano[2]
([1]CNR-ITM, Italy; [2]D.I.M.E.S., University of Calabria, Italy; [3]NFIQC, Universidad Nacional de Córdoba, Facultad de Ciencias Química, Departamento de Química Teórica y Computacional, CONICET, Argentina)

Session: Artificial intelligence methodologies in membrane science & engineering

PI_77. The Determination of Equation of State (EoS) Parameters Using Machine Learning Methods for Membrane Gas Separation.
Ismaeel Hasan[1], Ricci Eleonora[2], De Angelis Maria-Grazia[1] ([1]University of Edinburgh, UK; [2]National Centre for Scientific Research Demokritos, Greece)

PI_78. Data-driven screening and discovery of polymer membranes for gas separation: from the molecular structure to the industrial performance

Eleonora Ricci[1] Maria Grazia De Angelis[2] ([1]National Centre for Scientific Research Demokritos, Greece; [2] University of Edinburgh, UK)

PI_79. Machine learning models to predict organic solute rejection in reverse osmosis and nanofiltration using molecular fingerprints.
Lee Sangsuk and Straub Anthony (University of Colorado Boulder, Colorado, USA)

Session: Membrane fouling, biofouling, scaling, ageing, cleaning& maintenance

PI_80. On the changes of boundary flux values adopting different control valves.
Kamran Alam, Marco Stoller (Sapienza University of Rome, Italy)

PI_81. Evaluation of NaCl pre-cleaning of PVDF based MF membrane fouled by skim milk.
Dirany Aya[1,2], Al Hage Nour [1,2], Nasser Ghassan[2], Rabiller-Baudry Murielle [1]([1]Univ Rennes, CNRS, ISCR France; [2]Universite Libanaise, Lebanon)

PI_82. Natural phenolic compounds as membrane modifiers to decrease biofouling problems.
Fareeha Kayani[1], Carita Sallomy[2], Tiina Rissanen[1], Tuomo Laitinen[2], Maija Lahtela-Kakkonen[2], Mari Kallioinen-Mänttäri[1] ([1]LUT University, Finland; [2]University of Eastern Finland, Finland)

PI_83. Investigation of the effect of immobilization media materials in quorum quenching membrane bioreactors for biofouling control.
Korkusuz Semanur[1,2], Ardic Rabia[1,2], Yılmaz Merve[1,2], Pekgenc Enise[1,2], Kose-Mutlu Borte[1,2], Koyuncu Ismail[1,2] ([1]Istanbul Technical University, Turkey; [2]Istanbul Technical University, Turkey)

PI_84. Controlling mineral scaling using electric fields
Yachnin Elina[1], Jassby David[2], Segal-Peretz Tamar[3], Ramon Guy Z.[1]. ([1,3]Technion - Israel Institute of Technology, Israel; [2]University of California, California, USA)

PI_85. Optimization of membrane cleaning for nanofiltration of kraft black liquor.
Battestini Vives Mariona[1], Xiao Xiao[2], Lipnizki Frank[1] ([1]Lund University, Sweden; [2]Harbin Institute of Technology, China)

PI_86. Exploring the membrane fouling effects and cleaning strategies in osmotically assisted reverse osmosis processes
Peters Christian D[a,b], Li Dan[b,c], Mo Zijing[b,c,d], Hankins Nicholas P.[a], She Qianhong[b.c] ([a]The University of Oxford, UK; [b] Nanyang Technological University, Singapore; [c]Nanyang Technological University, Singapore; [d]Nanyang Technological University, Singapore)

PI_87. Toward sustainable and cost-effective production of drinking water from eutrophic and micro-polluted water using the PAC-UF membrane hybrid process.
Denizer Didem[1], Bogunovi Minja[2], Panglisch Stefan[1], Ivana Ivančev-Tumbas[2] ([1]University of Duisburg-Essen, Germany; [2]University of Novi Sad, Serbia)

PI_88. Antimicrobial and antibiofouling electrically conducting laser-induced graphene spacers in reverse osmosis membrane modules

Pisharody Lakshmi [a], Thamaraiselvan Chidambaram [a], Manderfeld Emily[b], Singh Swatantra P.[a], Rosenhahn Axel[b], Arnusch Christopher J.[a] ([a]Ben-Gurion University of the Negev, Israel; [b]Ruhr University Bochum, Germany)

PI_89. Quantifying the Biofouling Potential of Polymer Films: A Combined AFM and QCM-D Study
Eskhan Asma, Johnson Daniel (New York University Abu Dhabi, The United Arab Emirates)

PI_90. Impact of enzyme stabilizers and formulation on the real efficiency of enzymatic detergent useful to clean polymer membrane fouled in skim milk ultrafiltration
Kavugho Mission Sophie[1,2], Javelle Alex[1], Rabiller-Baudry Murielle[1] ([1]Univ Rennes, CNRS, ISCR, France ; [2]Hypred (KERSIA group), France)

PI_91. Optimizing the technical parameters of AnMBR with commercial PES membrane
Nguyen Anh Dao[1], Tran Thi Thai Hang[1], Dao Van Tri[1], La Vinh Trung[1], Tran Le Luu1, Tran Hung Thuan[2], Nguyen Van Tuyen2, Chu Xuan Quang2, Francesco Galiano[3], Maria Francesca Vigile[3], Francesca Russo[3], Alberto Figoli[3] (1Vietnamese German University, Vietnam; [2]Center for Advanced Materials and Environmental Technology, Vietnam; [3]Institute on Membrane Technology (CNR-ITM), Italy)

Session: Pre-treatment methods for membrane processes

PI_92. Optimizing flocculation of digestate to increase circularity in the nutrient recovery from manure.
Marrit van der Wal, Kitty Nijmeijer, Zandrie Borneman (Eindhoven University of Technology, The Netherlands)

PI_93. Optimization of coagulant dosage and pre-treatment configuration to minimize ultrafiltration (UF) fouling. *Malkoske Tyler A., Bérubé Pierre R., Andrews Robert C. (University of Toronto, Canada; University of British Columbia, Canada)*

PI_94. Pre-treatment adsorption methods of reverse osmosis for removal of surfactants and organic compounds from electroplating wastewater.
Moutafis Ipek[a,c], Engstler Roxanne[b], Fahling Felix Johannes[b], Scoullos Iosif Marios[a], Kouvelos Evangelos[a], Favvas Evangelos[a], Barbe Stephan[b], Sapalidis Andreas[a] ([a]National Centre for Scientific Research "Demokritos", Greece; [b]TH Köln, Germany; [c]International Hellenic University, Greece)

Session: Membrane modules

PI_95. Better performing ultrafiltration membranes: from fundamental research to large-scale membrane modules.
Meulman Jordy, Ania Pacak (Pentair X-Flow,The Netherlands)

PI_96. Evaluation the effectiveness of physicochemical pre-treatment of flatsheet and hollow fiber membrane in anaerobic MBR for seafood processing wastewater treatment.
Tran Thi Thai Hang[1], Nguyen Anh Dao[1], Dao Van Tri[1], La Vinh Trung[1], Tran Le Luu[1], Tran Hung Thuan[2], Nguyen Van Tuyen[2], Chu Xuan Quang[2], Francesco Galiano[3], Maria Francesca Vigile[3],

Francesca Russo[3], Alberto Figoli[3]. ([1]Vietnamese German University, Vietnam; [2]National Center for Technological Progress, Vietnam; [3]CNR-ITM, Italy)

Session: Microfiltration, Ultrafiltration

PI_97. Development of a sustainable cellulosic membrane platform with green solvents.
Fahlberg S.[1,2], Schneider B.[1], Ulbricht M.[2] ([1]Sartorius Stedim Biotech GmbH, Germany; [2]Universität Duisburg-Essen,Germany).

PI_98. Utilization of commercial ultrafiltration membranes to recover phenolic compounds from wet olive pomace
Sánchez Arévalo Carmen, Awoyemi Victor, Vincent-Vela M. Cinta, Álvarez-Blanco Silvia. (Universitat Politècnica de València, Spain; Universitat Politècnica de València, Spain)

PI_99. REDWine: Chlorella's harvesting system using membrane technology
Uriburu-Gray María[1], Santos Edgar[2], Badenes Sara[2], Gomes Diana[2], Matencio Sonia[1], Boerrigter Marcel[1] ([1] LEITAT Technological Center, Spain, [2]A4F ALGAFUEL SA, Portugal)

PI_100. Modification of Ultrafiltration Membranes to Enhance the Adsorption of Cations and Anions.
Sarah Glass[1], Anthony Arvind Kishore Chand[1,2], Tomi Mantel[2], Muhammad Usman[2], Mathias Ernst[2], Volkan Filiz[1] ([1]Institute of Membrane Research, Germany; [2]Hamburg University of Technology, Germany)

PI_101. Dye removal with waste cotton textile and wood-based cellulose-rich ultrafiltration membranes: on the mechanism of dye removal
Lopatina Anastasiia, Nieminen Joona, Mänttäri Mika, Kallioinen-Mänttäri Mari (LUT University, Finland)

PI_102. Remediation of water from per- and polyfluoroalkyl substances: ozonation vs electrochemical oxidation after coagulation-microfiltration.
Fanjing Meng, Agnieszka Cuprys, Harsha Ratnaweera, Zakhar Maletskyi. (Norwegian University of Life Sciences, Norway)

PI_103. 1-(2'-hydroxyethyl)pyrrolidin-2-one as green solvent for poly(ethersulfone) ultrafiltration membrane fabrication
Gronwald Oliver, Adermann Torben, Poulton Simon. (BASF SE, Germany)

PI_104. Ultrafiltration as pre-treatment to improve vinasse biomethanation potential
Moreira Victor R.[1], Carpanez Thais G.[1], Amaral Míriam C. S.[1] ([1]Federal University of Minas Gerais, Brazil)

Session: Nanofiltration, Reverse Osmosis

PI_105. A New Insight on Heavy Metal Removal by Absorptive Nanofiltration Membranes
Abedi Fatemeh, Emadzadeh Daryoush, Dubé Marc A, Kruczek Boguslaw (University of Ottawa,Canada)

PI_106. On the transmission of organic solutes in RO of salty and sea waters
Charik Fatima Zohra [1,2], Alami-Younssi Saad [2], Rabiller-Baudry Murielle[1] ([1]Univ Rennes, CNRS, ISCR, France; [2]University Hassan II of Casablanca, Morocco)

PI_107. Characterization of membrane performance in whey nanofiltration.
Roselli Marco, Bandini Serena, Boi Cristiana. (University of Bologna, Italy)

PI_108. Novel interfacially polymerized polyamide-silicon quantum dots composite membranes for nanofiltration process
Shu-Hsien Huang[1,2], Po-Hao Lu[1] ([1]National Ilan University, Taiwan; [2]Chung Yuan Christian University, Taiwan)

PI_109. Polymeric fluorine-free ionomers and their complexes with polyelectrolytes for thin-film composite nanofiltration membranes
Sven Kroß, Ulbricht Mathias (Universität Duisburg-Essen, Germany)

PI_110. Effect of Relative Humidity on Nanofiltration Membrane Preparation via Phase Inversion
Swarna Sutapa Roy, Hosseinabadi Sareh Rezaei, Rutgeerts Laurens, Vankelecom Ivo F.J. (Membrane Technology Group (MTG)-Centre for Membrane Separations, Adsorption, Catalysis and Spectroscopy for Sustainable Solutions (cMACS), Belgium)

PI_111. Downstream processing of corn steep waters for biosurfactants recovery by nanofiltration technology
Martínez-Arcos Andrea[a],Takeshi Bruno[b,c], Figueira, Mariana[b,c,*], Reig Mònica[b,c] Cruz José M[a], Moldes Ana Belén[a], Cortina José Luis [b,c,d]; Valderrama César[b,c], Vecino, Xanel[a] ([a]University of Vigo, Spain; [b]Universitat Politècnica de Catalunya (UPC)-BarcelonaTECH, Spain; [c]Barcelona Research Center for Multiscale Science and Engineering, Spain; [d]CETaqua, Spain)

PI_112. Predictive modeling of arsenic and manganese removal by reverse osmosis by multi-linear regression
Lazarini Júlia X.[1], Moreira Victor R.[2], Santos Lucilaine V. S.[1,2], Amaral Míriam C. S.[2] ([1] Pontifical Catholic University of Minas Gerais, [2]Brazil; Federal University of Minas Gerais, Brazil)

PI_113. Interaction between brine discharge and catchment water of a seawater desalination plant: A case study of Mainis Algeria
Abdelghani Bouthiba[1], Mourad Amitouche[2], Hacene Mahmmoudi*[1], Mattheus F.A. Goosen[3] ([1]Hassiba Benbouali University of Chlef, Algeria; [2]M'Hamed BOUGARA University Boumerdes, Algeria; [3]Alfaisal University, Riyadh Saudi Arabia)

PI_114. Fractionation of biologically active compounds extracted from onion skin bio-waste
Olkiewicz Magdalena[1], Bajek Anna[2], Kowalczyk Oliwia[3], Navarro Oscar[1], Montornes Josep M.[1], Tylkowski Bartosz [1] ([1]Eurecat, Centre Tecnològic de Catalunya, Spain; [2] Ludwik Rydygier Collegium Medicum in Bydgoszcz Nicolaus Copernicus University in Torun, Poland; [3]Ludwik Rydygier Collegium Medicum in Bydgoszcz Nicolaus Copernicus University in Torun, Poland)

PI_115. Mixed Polymeric Substrate RO Membranes with Nanographene-Surface Modification

Shalaby Marwa[1], Abdallah Heba[1], Wilken Ralph[2], Schmüser Christoph[2], Shaban Ahmed[3], Gaber Marwa[1] ([1] National Research Centre, Egypt; [2]Fraunhofer Institute for Manufacturing Technologies and Advanced Materials (IFAM), Germany; [3]National Research Centre, Egypt)

PI_116. Polyelectrolyte multilayer membranes for the separation of lactose and salts in dairy streams
Achterhuis Iske, Balint Antonia, de Bouhid Aguaiar Izabella, de Grooth Joris, Schroën Karin, de Vos Wiebe (University of Twente, The Netherlands)

Poster Session II, Tuesday, 22 November 2022

Chairs: Pierre Aimar & Heiner Strathmann

Session: Organic solvent nanofiltration

PII_1. Toward precision molecular sieving in organic solvents using metal–organic frameworks and conjugated microporous polymers
Rifan Hardian, Gyorgy Szekely (PSE, King Abdullah University of Science and Technology (KAUST), Saudi Arabia)

PII_2. Graphene based membranes to massively reduce energy in the oil, gas and chemical industry
Zouaoui Taqwa, Livingston Andrew, Li Kang (Imperial College London, United Kingdom)

PII_3. Interactive organic solvent nanofiltration database and prediction tools
Gergo Ignacz, Gyorgy Szekely (King Abdullah University of Science and Technology, Saudi Arabia)

PII_4. Organic liquid mixture separation using fluorinated covalent organic framework (COFs) supported ceramic hollow fibers
Hao Boyuan; Livingston, Andrew; Li, Kang (Imperial College London, UK)

Session: Forward osmosis, pressure retarded osmosis

PII_5. Development and characterization of high molecular weight and high-osmotic pressure draw solute for forward osmosis applications
Atashgar Amirsajad, Emadzadeh Daryoush, Kruczek Boguslaw (University of Ottawa, Canada)

PII_6. Preparation of electrospun forward osmosis membranes for clean water production
Kinik Aylin, Nijmeijer Kitty, Borneman Zandrie (Eindhoven University of Technology, The Netherlands)

PII_7. New applications of biomimetic hollow fiber forward osmosis membranes
Luxbacher Thomas[1,2], Buksek Hermina[2], Stergar Janja[2], Ban Irena[2], Helix-Nielsen Claus[2,3], Petrinić Irena[2] ([1]Anton Paar GmbH, Austria; [2]University of Maribor, Slovenia; [3] Technical University of Denmark, Denmark)

PII_8. Membranes for pressure retarded osmosis as a new way for sustainable power generation from salinity gradients

Klaus Karlsen[1], Thomas Thostrup[1], Ziran Su[1], Magdalena Malankowska[1], Haofei Guo[2], Lars Storm Pedersen[2], Manuel Pinelo[1] ([1]Technical University of Denmark, PROSYS, Denmark; [2]SaltPower, Denmark)

PII_9. Development of a tool for understanding and design of forward osmosis processes
Greisner Bastian[1,2,3], Mauer[1], Rögener[2], Lerch[3] ([1]MionTec GmbH, Germany; [2]TH Köln, Germany; [3]Technische Univseriät Dresden, Germany)

PII_10. Thermo-responsive PAG based commercial draw solutions for forward osmosis
Jančič Natalija, Bukšek Hermina, Petrinić Irena (University of Maribor, Slovenia)

Session: Electromembrane processes

PII_11. Recovery of palladium through anion exchange membranes using electrodialysis
Tekinalp Önder[1], Zimmermann Pauline[2], Burheim Odne Stokke[2], Deng Liyuan[1] (Norwegian University of Science and Technology (NTNU), Norway; [2]Norwegian University of Science and Technology (NTNU), Norway)

PII_12. Carbon capture from water bodies by Membrane Capacitive Deionization (MCDI)
Sarić Marija, Popat Yaksh, Sumbharaju Raghavendra, Veldhuis Jakobert, Sluijter Soraya, Vente Jaap (TNO, Sustainable Technologies for Industrial Processes, The Netherlands)

PII_13. Valorization of saltwork brines for the production of acid and base using electrodialysis with bipolar membranes
Lòpez J.[1,2], Filingeri A.[3], Leòn T.[1,2], Culcasi A.[3], Tamburini A.[3], Micale G.[3], Cortin, J.L.[1,2,4], Cipollina, A.[3] ([1]EEBE, Universitat Politècnica de Catalunya (UPC), Spain; [2]Campus Diagonal-Besòs, Spain; [3]Università di Palermo (UNIPA), Italy; [4]CETaqua, Spain)

PII_14. Coupling electrodialysis with membrane contactor: a key approach to recovering nutrients and volatile fatty acids from food wastes towards higher concentrations and selectivity
Kotoka Francis, Cornelissen Emile, Gutierrez Leonardo (Ghent University, CAPTURE, Belgium)

PII_15. The concentration, potential, and pH profiles along a diluate channel of a miniaturized electrodialysis unit
Slouka Zdenek, Vobeckà Lucie, Kovář Petr, Jan Pagáč, Smoleň Michal (University of Chemistry and Technology, Czech Republic)

PII_16. Effect of ion exchange on the structure of heterogeneous ion-exchange membranes
Svoboda Miloš[1,2], Vobeckà Lucie[1], Beneš Jan[2], Slouka Zdenek[1,2] ([1]University of chemistry and technology, Czech Republic; [2]University of West Bohemia, Czech Republic)

PII_17. Ionic transport properties of charged mosaic membrane prepared from fabric structure of poly (vinyl alcohol) based charged-fiber
Higa Minato[1,2], Jikihara Atsushi[3], Shimizu Eriko[1], Kakihana Yuriko[1,2], Higa Mitsuru[1,2] ([1]Yamaguchi University, Japan; [2]Blue Energy Center for SGE Technology (BEST), Japan; [3]KURARAY CO., Japan)

PII_18. Relationship between anion permeability and membrane resistance in electrodialysis using monovalent selective anion exchange membrane

Hirotani Taishi[1], Higa Minato[1,2]; Sugimoto Yu[1,2], Nakamura Yuki[3], Doi Shoichiâ´, Kakihana Yuriko[1,2], Higa Mitsuru[1,2] ([1]Yamaguchi University; [2]Blue Energy Center for SGE Technology (BEST), Japan, [3]GODO SHIGEN Corp., Japan)

PII_19. Evaluation of power generation characteristics in reverse electrodialysis power generation using novel profiled ion-exchange membranes
Tanaka Manato[1], Saito Soma[1], Sugimoto Yu[1,2], Higa Mitsuru[1,2] ([1]Yamaguchi University, Japan; [2]Blue Energy Center for SGE Technology (BEST), Japan)

PII_20. Estimation of power generation of pilot-scale reverse electrodialysis (RED) based on performance of prototype RED stack
Kawasaki Hiroki[1], Shimogochi Yuki[1], Sugimoto Yu[1,2], Higa Misturu[1,2] ([1]Yamaguchi University, Japan; [2]Blue Energy Center for SGE Technology (BEST), Japan)

PII_21. Donnan dialysis performance of monovalent ion selective hollow fiber cation exchange membranes prepared by plasma graft polymerization
Ikeda Shuntaro[1], Kakihana Yuriko[1,2], Higa Mitsuru[1,2] ([1]Yamaguchi University, Japan; [2]Blue Energy Center for SGE Technology (BEST), Japan)

PII_22. Design of selective electro-driven membranes for ion resource recovery
Yan Zhao, Petric Marc Ruya, Lei Xia, Yang Xing (University of Leuven, Belgium)

PII_23. Synthesis and fabrication of low-cost proton exchange membranes for artificial photosynthesis
Depuydt Stef, Van der Bruggen Bart (KU Leuven, Belgium)

PII_24. Effect of operational parameters in the production of acid and base using a electrodialysis with bipolar membranes at pilot plant scale
J. Lòpez[1,2,3], M. Herrero-Gonzalez[3,4], G. Virruso[3], C. Cassaro[3], A. Tamburini[3], A. Cipollina[3], J.L. Cortina[1,2,5], R. Ibañez[4], G. Micale[3] *([1]EEBE, Universitat Politècnica de Catalunya, Spain; [2]Barcelona Research Center for Multiscale Science and Engineering, Spain; [3]Università degli Studi di Palermo, Italy; [4]Universidad de Cantabria, Spain; [5]CETaqua, Spain)*

PII_25. Assessment of reverse electrodialysis membranes performance in the presence of divalent ions
Sampedro Tamara, Gòmez-Coma Lucìa, Ortiz Alfredo, Ibañez Raquel, Ortiz Inmaculada (Universidad de Cantabria, Spain)

PII_26. Control of Hydrodynamics by Spacers to Enhance Electroconvective Instabilities
Dunkel Richard[1], Evdochenko Elizaveta[1], Köller Niklas[1], Linkhorst John[1], Wessling Matthias[1,2] ([1]Chemical Process Engineering AVT.CVT, RWTH Aachen University; [2]DWI – Leibniz-Institute for Interactive Materials)

Session: Membranes for energy, (microbial) fuel cells, batteries, electrolyzer

PII_27. Branched poly(aryl piperidinium) membranes for anion-exchange membrane fuel cells
Xingyu Wu, Nanjun Chen, Harm-Anton Klok, Young Moo Lee, Xile Hu (ISIC, Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland)

PII_28. Anion exchange membranes for the electrochemical reduction of CO_2
Ter Weel Woutje, Houben Menno, Borneman Zandrie, Nijmeijer Kitty (Eindhoven University of Technology, The Netherlands)

PII_29. High-performance anion exchange membrane water electrolyzers with a current density of 7.68 A cm-2 and a durability of 1000 hours
Nanjun Chen[1], Ju Yeon Lee[2], Jong Hyeong Park[1], So Young Lee[2], Young Moo Lee[1] ([1]Hanyang University, Korea; [2]Fuel Cell Research Center, KIST, Korea)

PII_30. Recovery of ammonium ion by Donnan dialysis using sulfonated polyether sulfone-based cation-exchange membranes
Kakihana Yuriko[1,2], Komuta Keiko[1], Higa Mitsuru[1,2] ([1]Yamaguchi University, Japan; [2]Blue Energy Center for SGE Technology (BEST), Japan)

PII_31. Examination of operating conditions with high current efficiency in acid / alkali production using bipolar membrane electrodialysis
Hirao Souichiro[1], Kakihana Yuriko[1,2], Higa Mitsuru[1,2] ([1]Yamaguchi University, Japan; [2]Blue Energy Center for SGE Technology (BEST), Japan)

PII_32. H_2 production by water electrolysis using cation exchange membrane: insights on build a PEM electrolytic cell
Longo Mariagiulia[1], Esposito Elisa[1], Fontananova Enrica[1], Jansen Johannnes Carolus[1], Minotti Angelo[2], Figoli Alberto[1] ([1]Institute on Membrane Technology, CNR-ITM, Italy; [2]MIPRONS srl, Italy)

PII_33. Role of tungsten trioxide in enhancing PEM performance for fuel cell at intermediate temperature
Selim Asmaa, Szijjarto Gabor, Romànszki Loránd, Tompos Andràs (Institute of Materials and Environmental Chemistry, Research Centre for Natural Sciences)

PII_34. A Facile Approach to Fabricate Ion Exchange Membranes for Fuel Cell and Electrolysis Applications
Sang Yong Nam, Kwang Seop Im, Do Hyeong Kim, Vijayakumar Vijayalekshmi (Gyeongsang National University, Korea)

Session: Membrane contactors: membrane condenser, membrane dryier

PII_35. Beverage dealcoholization by stripping gas-liquid membrane contactor
Mericq Jean-Pierre[1], Zurob Elsie[1,2], Belleville Marie-Pierre[1], Sanchez-Marcano José[1] (*[1]IEM, CNRS, Université de Montpellier, France. [2]LabProSeM, University of Santiago de Chile, Chile)*

PII_36. Nitrogen recovery from effluents with high ammonium concentration using membrane contactor
Cifuentes-Cabezas Magdalena[1], Lujàn-Facundo Marìa-José[1,2], Cuartas-Uribe Beatriz[1,2], Iborra-Clar Alicia[1,2], Mendoza-Roca José-Antonio[1,2] ([1]IEM, CNRS, Université de Montpellier, France. [2]LabProSeM, University of Santiago de Chile, Chile)

PII_37. Investigation of ceramic membrane contactors regarding their suitability for nutrient recovery from digestate
Pflieger Christian[1], Weyd Marcus[1], Fischer Gundula[1], Wufka André[2], Trepte Sarah[2], Faßauer Burkhardt[2], Voigt Ingolf[1] ([1]Fraunhofer IKTS, Germany; [2]Fraunhofer IKTS, Germany)

PII_38. New concepts for the use of membrane contactors in treatment of (waste) water and their impact to climate change
Selzer Norbert, Ulbricht Martin (3M Deutschland GmbH Separation and Purification Sciences Division, Germany)

PII_39. Preparation of microcapsules based on polyelectrolyte complexes by membrane emulsification
Victoriia Konovalova, Iryna Kolesnyk (National University of Kyiv-Mohyla Academy, Ukraine)

PII_40. The production of PCL nanoparticles by membrane emulsification/solvent diffusion and membrane nanoprecipitation: a comparative analysis
Piacentini Emma, Bazzarelli Fabio, Giorno Lidietta (Institute on Membrane Technology (CNR-ITM), Italy)

PII_41. O/W emulsions stabilized with lesser mealworm (A. diaperinus) protein concentrate: studies of droplet coalescence with microfluidics
Jayakumar Jitesh[1], Vernet Anton[2], Pallarès Jordi[2], Ballon Aurélie[1], De Lamo-Castellvì Sìlvia[1], Güell Carme[1], Ferrando Montserrat[1] (ETSEQ, Universitat Rovira i Virgili, Spain; [2]ETSEQ, Universitat Rovira i Virgili,Spain)

PII_42. Maximize bioactive molecules encapsulation by pulsed back-and-forward Membrane Emulsification
Russo Beatrice[1], Piacentini Emma[1], Bazzarelli Fabio[1], Calderoni Gabriele Maria[2], Vacca Paolo[2], Figoli Alberto[1], Giorno Lidietta[1] ([1]CNR-ITM, Italy; [2]SAES Getters S.p.A., Italy)

PII_43. Membrane ionotropic gelation as scalable method for nanoparticles production
Russo Beatrice[1], Piacentini Emma[1], Bazzarelli Fabio[1], Calderoni Gabriele Maria[2], Vacca Paolo[2], Figoli Alberto[1], Giorno Lidietta[1] ([1]CNR-ITM, Italy; [2]SAES Getters S.p.A., Italy)

PII_44. A numerical demystification of the pre-mix membrane emulsification process: a dynamic flow architecture-impregnated distributed mass balance-based multi-phase modeling
Deb T. K.[1,2], Mike M.[3] ([1]Tremplin A., France. [2]Last position hold: Marie Skłodowska-Curie ESR, Erasmus Mundus Fellow (Membrane Engineering in the framework of EM3E-4SW, 2013-2015) [3]Interpol, France)

PII_45. PVDF-chitosan hybrid membranes for membrane emulsification with olive oil
Zuzanna Flanc, Joanna Kujawa, Wojciech Kujawski (Nicolaus Copernicus University, Poland)

Session: Membrane distillation, osmotic distillation, membrane crystallization

PII_46. Hydrodynamics (Reynold's number) are critical to limiting surface scaling in membrane distillation (and membrane distillation-crystallisation)

Jikazana Aphiwe, Campo Moreno Pablo, McAdam Ewan (Cranfield University, UK)

PII_47. Energetic penalty for water recovery from mining brine by direct contact membrane distillation
Grosso Valentina, Pantuso Elvira, Donato Laura, Di Profio Gianluca, Fontananova Enrica (Institute on Membrane Technology (CNR-ITM), Italy)

PII_48. Laboratory-scale evaluation of petroleum industry wastewater dewatering through membrane distillation
Bottino Aldo, Comite Antonio, Pagliero Marcello, Rizzardi Ilaria, Boero Federica, Cattaneo Claudia, Tagliabue Marco (Università di Genova, Italy; TICASS S.c.r.l., Italy; Environmental and Bological Laboratories, Eni S.p.a., Italy)

PII_49. Evaluation of membranes performance for the treatment of high salinity sources with membrane distillation
Andrés-Mañas Juan Antonio, Requena Isabel, Aparicio Fernando, Zaragoza Guillermo (CIEMAT-Plataforma Solar de Almerìa, Spain)

PII_50. Recovery and control of lactose crystals using membrane crystallization
Shirazi, Mohammad Mahdi; Ali, Aamer; Quist-Jensen, Cejna Anna (Center for Membrane Technology, Denmark)

PII_51. Membrane distillation for produced water treatment
Macedonio Francesca, Alessandro Francesca, Drioli Enrico (Institute on Membrane Technology (CNR-ITM), Italy)

PII_52. Photothermal nanocomposite membranes for solar membrane distillation processes
Mella Javiera[1,2], Quintero Yurieth M.[1,3], Giraldo Mejia Hugo[1], Quero Frank[2], Araya Rodrigo[4], Garcìa Andreina[1,5] ([1]AMTC, Universidad de Chile, Chile; [2]FCFM, Universidad de Chile, Chile; [3]Universidad Tecnologica Metropolitana (UTEM), Chile; [4]Universidad Tecnològica Metropolitana (UTEM), Chile; [5]FCFM, Universidad de Chile, Chile)

PII_53. Direct contact membrane distillation applied to the simultaneous treatment of two saline streams
Criscuoli, M.C. Carnevale (Institute on Membrane Technology (CNR-ITM), Italy)

PII_54. Enhanced nutrient recovery from mariculture wastewater via air gap membrane distillation process
Duyen Phuc-Hanh Tran, Dian Qoriati, Ya-Fen Wang, Sheng-Jie You (Chung Yuan Christian University, Taiwan)

PII_55. Improving structural homogeneity, hydraulic permeability, and mechanical performance of sustainable skinny asymmetric monophasic cellulose acetate/silica membranes: spinodal decompositions mix
Zare Fahimeh[1,2], Gonçalves Sérgio[3], Narciso Joana[1], Faria Mònica[4], Gonçalves M. Clara[1,2]([1]Universidade de Lisboa, Portugal; [2]Centro de Quìmica Estrutural (CQE), Portugal; [3]IDMEC, Universidade de Lisboa, Portugal; [4]CeFEMA, Universidade de Lisboa,Portugal)

PII_56. Multipass hollow fiber membrane modules for membrane distillation
Aamer Ali[1], Jheng-Han Tsai[2], Cejna Quist-Jensen[1] ([1]Aalborg University, Denmark; [2] Department of Chemical Engineering and Biotechnology, UK)

PII_57. Membrane distillation for phenolic compounds removal from surface water
Ramos Ramatisa L., Lebron Yuri A. R., Moreira Victor R., Martins Mateus F., Santos Lucilaine V. S., Amaral Miriam C. S. (Federal University of Minas Gerais, Brazil)

PII_58. Ceramic membranes to achieve zero liquid discharge through Percrystallization
Nielsen Morten H., Quist-Jensen Cejna , Ali Aamer (Aalborg University, Denmark)

Session: Membrane reactors, photocatalytic membrane reactors

PII_59. Stability-time performance study of methanol conversion to dimethyl ether on a α-Al_2O_3 catalytic membrane
Elisa Avruscio[1], Adele Brunetti[1], Giuseppe Barbieri[1] (CNR-ITM, Italy)

PII_60. Reusable nanocomposite-filters for arsenite and arsenate dual real effluents remediation in an up-scaled membrane reactor
Martins Pedro, Salazar Hugo, Fernandes Margarida, Costa Pedro, Ferdov Stanislav, Botelho Gabriela, Lanceros-Mendez Senentxu (University of Minho, Portugal; BCMaterials, Basque Center for Materials, Applications and Nanostructures, UPV/EHU, Spain; IKERBASQUE, Spain)

Session: Membrane bioreactors, submerged MBR

PII_61. Vinasse treatment by anaerobic membrane bioreactor in soudano-sahelian climate: effect of SRT on biogas production and membrane fouling.
Abdoul Wahab Nouhou Moussa[1], Boukary Sawadogo[1], Yacouba Konaté[1], Sayon Sidibe[1], Marc Heran[2] ([1]Institut International d'Ingènierie de l'Eau et de l'Environnement (2iE), LEHSA, Burkina Faso. [2]Université de Montpellier, France)

PII_62. Anaerobic membrane bioreactor for vinasse recycling and by-products recovery: water, biomethane, and fertilizer
Moreira Victor R.[1], Carpanez Thais G.[1], Amaral Mìriam C. S.[1] ([1]Federal University of Minas Gerais, Brazil)

PII_63. Reuse4Hos: Hospital Wastewater Treatment and Reuse by Solar-Powered Membrane Bioreactor
De Sneha[1,2,3], Mathieu Coutard[4], Figoli Alberto[2], Gabriele Bartolo[1], Hoinkis Jan[3] ([1] University of Calabria, Italy; [2]Institute on Membrane Technology (CNR-ITM), Italy; [3]CAR, Karlsruhe University of Applied Sciences, Germany; MMT, Karlsruhe University of Applied Sciences, Germany)

PII_64. Unraveling pH effects on UF membrane fouling by extracellular polymeric substances: Adsorption and conformation analyzed with localized surface plasmon resonance
Ran Noya, Sharon-Gojman Revital, Larsson Sara, Gillor Osnat, Mauter S. Meagan, Herzberg Moshe (Ben-Gurion University of the Negev, Israel)

Session: Pervaporation, vapor permeation

PII_65. The potential of vapor-phase membrane separation of organic components from biomass treatment products
Alina Kozlova, Maxim Shalygin, Vyacheslav Zhmakin, Alexander Netrusov, Vladimir Teplyakov (TIPS RAS, Russia)

PII_66. Pervaporative separation of methanol – dimethyl carbonate system with PDMS based mixed matrix membrane with pristine and functionalized MIL-53(Al)
Katarzyna Knozowska[1], Joanna Kujawa[1], Wojciech Ogiegło[2], Wojciech Kujawski[1] ([1]Nicolaus Copernicus Universityin, Poland; [2]King Abdullah University of Science and Technology (KAUST), Saudi Arabi)

PII_67. On the use of pervaporation for the recovery of pharmaceutical solvents
Martì-n-Quijano Álvaro, González-Revuelta Daniel, Gorri Daniel (University of Cantabria, Spain)

Session: Membranes in gas and vapor separation

PII_68. Synthesis and characterization of membranes based on graphen oxide and carbon nanotubes for H_2 separation from gas mixtures
Gardenö Dániel[1], Bouša Daniel[2], Sofer Zdeněk[2], Vlastimil Fila[3], Karel Friess[1]([1] Department of Physical Chemistry, UCT Prague [2] Department of Inorganic Chemistry, UCT Prague [3] Department of Inorganic Technologies, UCT Prague)

PII_69. Tuning ultramicropore dimensions of carbon molecular sieve membranes via electron Irradiation
Banseok Oh, Dong-Yeun Koh (KAIST)

PII_70. Advanced GNPs/CA asymmetric membrane manufacturing via electrohydrodynamic emission
Alkandari Sharifah, Perera Semali, Castro-Domingueza Bernardo (University of Bath, United Kingdom)

PII_71. Modified supports to induce the synthesis of high performance ZIF-8 membranes
Shan Yongjiang, Wang Yifei, Wang Xingqian, He Mingliang, Zhang Fei, Chen Xiangshu (Jiangxi Normal University, P. R. China)

PII_72. Layer by Layer assembly (LbL) of polyelectrolyte thin-membranes for gas separation
Signorini Virginia[1], Giacinti Baschetti Marco[1], Minelli Matteo[1], Oldani Claudio[2] ([1]DICAM, Alma Mater Studiorum, Italy; [2]Solvay Specialty Polymers Italy S.p.A., Italy)

PII_73. Preparation and characterization of newly developed gas separation membranes made from aramid polymers
Hyunho Choi, Wonseong Song, Kyungeun Park, Sang Yong Nam (Gyeongsang National University, Republic of Korea)

PII_74. Effect of water vapor, pressure and temperature on the separation performance of mixed matrix iongel membranes containing azo-porous organic polymers
Nabais Rita[1], Ortiz-Albo Paloma[1], Mecerreyes David[2], Zhou Jin-Xiu[2], Crespo João[1], Tomé Liliana[1], Neves Luìsa[1] ([1]LAQV-REQUIMTE, Universidade NOVA de Lisboa, Portugal; [2]POLYMAT, University of the Basque Country UPV/EHU, Spain)

PII_75. Thin film composite membranes based on PIM/Matrimid® blends for gas separation
Esposito Elisa[1], Ferrari Maria-Chiara[2], Lasseuguette Elsa[2], Fuoco Alessio[1], Longo Mariagiulia[1], Monteleone Marcello[1], Tocci Elena[1], McKeown Neil B.[3], Jansen J.C.[1] ([1]CNR-ITM, Italy; [2]University of Edinburgh, UK; [3]EaStCHEM, University of Edinburgh, UK)

PII_76. Optimization of pretreatment and humidification methods of heterogeneous anion exchange membranes for direct air capture of CO_2
Wang Xueru, Jia Ni, Deng Liyuan, Liangliang Zhu ([1]Norwegian University of Science and Technology (NTNU), Norway; [2]SV LAB, Xi'an Jiaotong University (XJTU), China; [3]Northwest University, China)

PII_77. The impact of water vapor on the performance of mixed matrix membranes containing ionic liquids for gas separation
Ortiz-Albo P.[1], Tinevez J.[2], Sequeira I.[3], Baptista A. C.[3], Ferreira I.[3], Kumakiri I.[4], Crespo J.[1], Neves L.[1] ([1]LAQV/REQUIMTE, Universidade NOVA de Lisboa, Portugal; [2]ITECH Lyon, France; [3]CENIMAT/I3N, Universidade Nova de Lisboa, Portugal; [4]Yamaguchi University, Japan)

PII_78. Flat sheet and hollow fiber CO_2/CH_4 selective membranes based on green chemistry
Theodorakopoulos George[1,2], Karoussos Dionysios[1], Sapalidis Andreas[1], Favvas Evangelos[1] ([1]National Center for Scientific Research "Demokritos", Greece; [2]National Technical University of Athens, Greece)

PII_79. Simulation study of single- and multi-stage membrane system based on CO_2-philic composite hollow fiber membrane modules
Peter Jakub[1], Pientka Zbynek[1], Medeiros, Santos Gbriela[1], Marsalek Jiri[2] ([1]Institute of Macromolecular Chemistry CAS, Czech Republic; [2]MemBrain s.r.o., Czech Republic)

PII_79a. Separation of close-boiling point F-gas mixtures with hollow fiber membranes coated with a defect- free Pebax®1657/ Ionic Liquid composite layer
Gutiérrez Sergio V, Ibañez-Lipatova Anna, Pardo Fernando, Zarca Gabriel, Urtiaga Ane (Universidad de Cantabria, Spain)

Session: Membranes in hydrogen production and separation

PII_80. A combined molecular-level simulation and experimental approach to develop a thin-film polymer barrier for high-pressure hydrogen storage tanks
Méryll Barraco[1,3], Sylvie Neyertz[1], Farzaneh Radmanesh[2], Nieck E. Benes2, David Brown[1] ([1]Univ. Savoie Mont Blanc, Univ. Grenoble Alpes, CNRS, France; [2]University of Twente, r; [3]DPI, The Netherlands)

PII_81. ZrVTi sustainable multi-layered membranes prepared by HiPIMS
Fasolin Stefano[1], Barison Simona[1], Agresti Filippo[1], Battiston Simone[1], Fiameni Stefania[1], Isopi Jacopo[1], Armelao Lidia[2,3] ([1]Institute of Condensed Matter Chemistry and Technologies for Energy (CNR-ICMATE), Italy; [2]Dept. Chemical Sciences and Materials Technology (CNR-DSCTM), Italy; [3]University of Padova, Italy)

PII_82. Comparative Analysis of ZIF-8 and ZIF-90 Matrimid® mixed matrix membranes for hydrogen recovery from industrial waste streams
Moral Gonzalo, Ortiz Alfredo, Gorri Daniel, Ortiz Inmaculada (Universidad de Cantabria, Spain)

PII_83. Development of membranes for alkaline water electrolysis
Gupta Ruchika (Indian Institute of Technology Bombay, India)

PII_84. Application of facilitated transport membranes for hydrogen separation in HT-PEM Fuel cell plants
Shivakumar Tarun[1], Loreti Gabriele[2], Facci Andrea Luigi[2], Ansaloni Luca[3], Peters Thijs[3], Ferrari Maria-Chiara[1] ([1]University of Edinburgh, UK. [2]DEIM, University of Tuscia, Italy. [3]SINTEF Industry, Norway)

Session: Membranes for CO$_2$ capture

PII_85. Carbon capture via high performance ZIF-302/PSF interfacial composite membranes
Sarfraz, Muhammad Sarfraz (University of Engineering and Technology, Pakistan)

PII_86. Innovative Direct Air Capture (DAC) technology based on non-dispersive CO$_2$ absorption using membrane contactors and ionic liquids
Vadillo Jose Manuel, Martinez-Ramil Jacobo, Dìaz-Sainz Guillermo, Gomez-Coma Lucìa, Garea Aurora, Irabien Angel (Biomolecular Universidad de Cantabria ETSIIyT, Spain)

PII_87. CCSEL Research Infrastructure for evaluation of membranes and barrier materials relevant within the CCUS value chain
T.A. Peters, L. Ansaloni, M. Minelli, V. Röhling, S. Quale (SINTEF Industry, Norway; Alma Mater Studiorum-Università di Bologna; ECCSEL ERIC, Norway)

PII_88. H$_2$/CO$_2$ separation using polyPOSS-imide and hybrid silica based ceramic multi-channel membranes
Nikbakht Fini Mahdi, Sumbharaju Sumbharaju, van Tuel Marc, van Delft Yvonne, Dhaler Didier, Ansaloni Luca, Peters Thijs (TNO, Unit Energy Transition, The Netherlands; CTI Alsys-Group, Salindres, France; SINTEF Industry, Norway)

PII_89. Facilitated direct air capture of CO$_2$ in polysulfone membrane contactors by improving stability and hydrophobicity of amine additives
Alireza Zare[1], Ahmed Khatib Boukalfa[1], Adrianna Nogalska[2], Alberto Puga[1], Pierfrancesco Cerruti[3], Bartosz Tylkowski[2], Borja Pascual-Jose[4], Amparo Ribes-Greus[4], Marta Giamberini[1] ([1]Universitat Rovira I Virgili, Spain; [2]Eurecat, Centre Tecnològic de Catalunya, Spain; [3]Institute of Polymers, Composites and Biomaterials, (CNR-IPCB), Italy; [4] ITM, Universitat Politècnica de València (UPV), Spain)

PII_90. Postcombustion carbon capture via nanoporous graphene membranes: a techno-economic analysis
Micari Marina, Agrawal Kumar Varoon (Ecole Polytechnique Federale de Lausanne (EPFL), Switzerland)

PII_91. (Per-)fluorinated MOF-based MMMs for CO_2 capture
Esposito Elisa[1], Tocci Elena[1], Calucci Lucia[2], Taddei Marco[3], Lessi Marco[3], Morelli Venturi Diletta[4], Marmottini Fabio[4], Costantino Fedinando[4], Guiotto Virginia[5], Signorile Matteo[5], Crocellà Valentina[5], Fuoco Alessio[1] ([1] CNR-ITM, Italy; [2]Institute of chemistry of organometallic compounds, (CNR-ICCOM), Italy; [3]University of Pisa, Italy. [4]University of Perugia, Italy. [5]University of Torino, Italy)

PII_92. Membrane, gas hydrate and hybrid technologies for CO_2 capture from flue gases
Vorotyntsev Ilya, Atlaskin Artem, Petukhov Anton (Mendeleev University of Chemical Technology of Russia, Russia)

Session: Membranes in wastewater treatment

PII_93. Wastewater treatment and resource recovery in the food industry by membrane technology
Pervez Md. Nahid[1], Mishu Monira Rahman[2], Mahboubi Amir[3], Stylios George K.[4], Buonerba Antonio[1], Hasan Shadi W.[5], Cai Yingjie[6], Zhao Yaping[7], Zarra Tiziano[1], Belgiorno Vincenzo[1], Taherzadeh Mohammad J.[3], Naddeo Vincenzo[1] ([1]SEED, University of Salerno, Italy; [2]Patuakhali Science and Technology Universit,, Bangladesh; [3]University of Borås, Sweden; [4]Heriot-Watt University, UK; [5]CMAT, Khalifa University of Science and Technology, UAE; [6]Wuhan Textile University, China; [7]East China Normal University, Institute of Eco-Chongming, China)

PII_94. Polystyrene nanoplastics removal from urban wastewater by aerobic membrane bioreactor
Anamary Pompa-Pernìa[1,2], Serena Molina[1], Junkal Landaburu-Aguirre[1] ([1]IMDEA Water Institute, Spain; [2]University of Alcalà, Spain)

PII_95. Validation of recycled nanofiltration and anion-exchange membranes for the treatment of high salinity urban wastewaters for crop irrigation
Anamary Pompa-Pernìa[1,2], Serena Molina[1], Amaia Lejarazu-Larrañaga[1], Junkal Landaburu-Aguirre[1], Eloy Garcìa Calvo[1,2] ([1]IMDEA Water Institute, Spain; [2]University of Alcalà, Spain)

PII_96. Reusable composite membranes for highly efficient chromium removal from real water matrixes
P. M. Martins, Joana M. Queiròs, H. Salazar, A. Valverde, G. Botelho, R. Fernàndez de Luis, J. Teixeira, S. Lanceros-Mendez (CF-UM-UP, University of Minho, Portugal; LaPMET, University of Minho, Portugal; Centre of Molecular and Environmental Biology, University of Minho, Portugal; Centre/Department of Chemistry, University of Minho, Portugal; Institute for Research and Innovation on Bio-Sustainability, University of Minho, Portugal; BCMaterials, Spain; IKERBASQUE, Basque Foundation for Science, Spain)

PII_97. The gases emissions by raw and membrane-treated vinasse applied into soil
Carpanez Thais G.[1], Moreira Victor R.[1], Amaral Mìriam C. S.[1] ([1]Federal University of Minas Gerais, Brazil)

Session: Membranes in drinking water

PII_98. Toward sustainable and cost-effective production of drinking water from eutrophic and micro-polluted water using the PAC-UF membrane hybrid process
Denizer Didem[1], Bogunović Minja[2], Panglisch Stefan[1], Ivančev-Tumbas Ivana[2] ([1]University of Duisburg-Essen, Germany; [2]University of Novi Sad, Serbia)

PII_99. Development of an innovative PVDF based hollow fiber membrane with incorporated graphene oxide to combine ultrafiltration and adsorption
W.Marechal, O. Lorain (Polymem, France)

PII_100. Effectiveness of hybrid adsorption/coagulation/ceramic MF towards contaminants of current and emerging concern in drinking water
Campinas Margarida[1], Viegas Rui M.C. [1], Coelho Rosàrio[2], Lucas Helena[2], Rosa Maria João[1] ([1]LNEC-National Laboratory for Civil Engineering, Portugal; [2]AdA-Águas do Algarve S.A., Portugal)

PII_101. Decentralized water treatment by low pressure recycled reverse osmosis
Moreira Victor R.[1], Lebron Yuri A. R.[1], Santos Lucilaine V. S.[2], Amaral Mìriam C. S.[1] ([1]Federal University of Minas Gerais, Brazil; [2]Pontifical Catholic University of Minas Gerais, Brazil)

PII_102. F-rich groundwaters in Calabria Region (Southern Italy): from sources to remediation
Fuoco Ilaria[1,2], Apollaro Carmine[1], De Rosa Rosanna[2], Gabriele Bartolo[3], Criscuoli Alessandra[2], Figoli Alberto[2] ([1] DiBEST, University of Calabria, Italy; [2]Institute on Membrane Technology (CNR-ITM), Italy; [3]LISOC, University of Calabria, Italy)

PII_103. Graphene oxide membrane on Nylon support for PFAS removal from water by filtration
Pla Raùl, Vàzquez Andrea, Baeza Jose A, Calvo Luisa, Alonso-Morales Noelia, Gilarranz Miguel A. (Universidad Autònoma de Madrid, Spain)

PII_104. Prepare of layer by layer polymer membrane for high permeation rate separation membrane for water purification
Sang Yong Nam[1], Kwang Seop Im[1], Seong Heon Kim[1], Seung Moon Woo[2], Youn Suk Chung[2] ([1]Gyeongsang National University, Republic of Korea; [2]PureMem Co., Republic of Korea)

PII_105. Laboratory pilot of SophiA water technologies for drinking and deionised water
De Sneha[1,2,3], Duc N. Dinh[3], Oliver Schmid[3], Michael Kauffeld[3], Gabriele Bartolo[1], Figoli Alberto[2], Hoinkis Jan[3] ([1]University of Calabria, Italy; [2]CNR-ITM, Italy; [3]IKKU, Karlsruhe University of Applied Sciences, Germany)

Session: Membranes for (bio)artificial organs and controlled delivery/release

PII_106. Perspectives of the extracorporeal membrane oxygenation from the mathematical modelling

Mondal Sourav (Indian Institute of Technology Kharagpur, India)

PII_107. Characterization under dynamic conditions of cellulose acetate-based monophasic hybrid membrane for improved blood purification devices
Rodrigues Flàvia S.C., Janeca Adriana, Pires Rita F., Faria Mònica (CeFEMA, LaPMET, Universidade de Lisboa, Portugal)

PII_108. Controling polylactide layer parameters covered hyaluronic acid-alginate structures in drug carriers
Trusek Anna, Nowak Mariusz, Kijak Edward (Wroclaw University of Science and Technology, Poland)

Session: Integrated membrane processes, hybrid processes, new trends in membrane science and technology

PII_109. Hydrogen production by ammonia scission
Zucca Tullia (Chemical Engineering Free Consultant, Italy)

PII_110. Monophasic coupling of nanofiltration and O_3/H_2O_2 advanced oxidation process
Loulergue Patrick[1], Ouali Sara[1,2], You Rukun[1], Biard Pierre-François[1], Nasrallah Noureddine[2], Szymczyk Anthony[1] ([1]Univ Rennes, CNRS, France; [2]Université des Sciences et de la Technologie Houari Boumediene, Algérie)

PII_111. Life-cycle impact assessment of end-of-life reverse osmosis membranes: method selection
Grossi Luiza, Neves Eric, Silva Bruna, Lange Lisete, Amaral Miriam (Universidade Federal de Minas Gerais, Brazil)

PII_112. Plasmonics phenomena in membrane technology
Alessandro Francesca, Macedonio Francesca, Drioli Enrico (CNR-ITM, Italy)

PII_113. A tunable fabrication method towards catalytic polymer-based porous multi-composite membranes with promising properties for pollutant degradation and hydrogen production
Volz Anna, Fischer Lukas, Ulbricht Mathias (Universität Duisburg-Essen, Germany)

PII_114. Intensification of the antisolvent crystallization process using membrane technology
Chergaoui Sara[1], Leyssens Tom[2], P. Debecker Damien[2], Luis Alconero Patricia[1] iMMC-IMAP, UCLouvain, Belgium; [2]IMCN, UCLouvain, Belgium)

Additional list

PIII_AL_1. Continuous and intensified membrane crystallization process: Quality control of an active pharmaceutical ingredient
Kalalech Carla [1], Charcosset Catherine[1], Monnot Mathias [2], Mangin Denis[1], Gagnière Emilie[1], Habanon Élodie [1]([1] University, LAGEPP, France [2] Aix Marseille University, CNRS, France)

Preparation and characterization of semi-alicyclic homo-, co- and blend polyimides membranes using alcyclic dianhydrides and their hydrogen separation properties

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Aromatic polyimides are promising membrane materials for gas separation due to their excellent hydrogen separation properties. Herein, two soluble semi-alicyclic polyimides were synthesized via a one-step thermal imidization process with two alicyclic dianhydrides possessing asymmetric kink structures: bicyclo [2,2,2] oct-7ene-2,3,5,6-tetracarboxylic dianhydride (BCDA) and 5-(2,5-dioxotetrahydrofuryl)-3-methyl-3-cyclohexene-1,2-dicarboxylic anhydride (DOCDA), and a flexible aromatic diamine: 4,4'-Oxydianiline (ODA). Their homo-, co- and blend polyimide membranes were prepared in various mixing ratios (100/0, 75/25, 50/50, 25/75, and 0/100) and their gas permeation properties were investigated for five gases (H_2, CO_2, O_2, N_2, and CH_4) and five gas pairs (H_2/CH_4, H_2/N_2, CO_2/CH_4, CO_2/N_2, and O_2/N_2). The homopolyimides (BCDA-ODA, DOCDA-ODA) exhibited amorphous structure without crystallinity and good solubilities in the casting solvents. BCDA-ODA showed higher d-spacing, FFV values, gas diffusivities and gas solubilities than DOCDA-ODA, resulted in higher gas permeabilities and lower gas selectivity for the five gas pairs, which were severely affected by the gas temperature in feed. Also, the gas permeability and selectivity of the co- and blend polyimide membranes were dependent upon the BCDA/DOCDA mole ratio. The excellent gas separation properties of homo-, co- and blended membranes were observed for the hydrogen over other gases, which showed comparable to or better than those of commercial membrane materials such as P84®, PSF, and Matrimid®.

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Towards sustainable manufacturing of electrospun PVDF mixed matrix membranes for water treatment and purification

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Chemical separations are critical to many aspects of our daily lives, from the energy we use to the medications we take. Separations consume 10-15% of the total energy use worldwide, with membranes having the potential to make separations 10x more energy efficient, saving >Mt CO_2 emissions annually. However, current membrane manufacture is highly unsustainable, with development based on trial-and-error approaches and fabrication using highly toxic solvents prone to regulatory restrictions and banning. This work lays the foundations for a novel approach to membrane discovery and manufacture based on the use of green solvents to electrospin PVDF-graphene-based mixed matrix membranes for water treatment and purification. Water treatment is an area where performance improvement can have dramatic impacts in both developing and developed countries, e.g. by providing access to potable water or reducing the energy consumption of water treatment.

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Electrospun Poly(vinylidene fluoride-hexafluoropropylene) (PVDF-HFP)-CNT-GO nanocomposite membranes for Membrane Distillation Applications

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Nanotechnology has made remarkable progress in terms of nanofiber manufacturing and practical applications in recent years. Electrospinning is a simple, versatile, and cost-effective technology that uses electrostatic repulsions between surface charges to fabricate scalable assemblies from a variety of raw materials (polymers, ceramics, nanoparticles, carbon nanomaterials, gels, and so on.) with diameters down to the macro-nano scale. The PVDF-HFP polymer was dissolved in a mixture of N,N-dimethylformamide (DMF)/ acetone and successive quantities addition of graphene oxide (GO) and carbon nanotube (CNT) nanoparticles. The obtained polymer/GO-CNT solutions were used to make the Electrospun Nanofiber Membranes. Constant working conditions are used, including voltage, needle and plate distance, flow rate, and so on. The effects of hot pressing and variation of fiber diameter on pore size, porosity, contact angle, and liquid entry pressure (LEP) of the fabricated membranes were all studied. It was noted that increasing the heat post-treatment to 140°C resulted in the formation of an improved membrane's mechanical and physical properties. This was demonstrated by examining the thermal properties, crystal structure and phases by differential scanning calorimetry (DSC), X-ray diffraction (XRD) and FTIR spectroscopy. Scanning Electron Microscopy (SEM), Atomic Force Microscope (AFM), and Energy Dispersive X-ray Analysis (EDX) results showed that grafting PVDF-HFP with varied concentrations of nanoparticles and heat post-treatment improved the morphological structure of the electrospun membranes. The electrospun PVDF-HFP nanocomposite membranes were tested for the treatment of brine solutions (60 g/L) by direct contact membrane distillation (DCMD) and a water flux value from 30 to 48 kg. h^{-1} .m^{-2} were obtained with a salt rejection higher than 99.5%. The developed membranes are hydrophobic and the measured contact angle was between ~120°-130°. The electrospun nanocomposite membranes can provide an innovative and easy way to obtain more efficient, high-performance membranes in water treatment.

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Fabrication of Poly(vinylidene fluoride-hexafluoropropylene) (PVDF-HFP) nanofiber membranes by electrospinning process for Membrane Distillation Applications

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Among several membrane-based techniques, membrane distillation (MD) technique has various advantages. MD advantages include (i) high salt rejection, (ii) lower fouling sensitivity, and (iii) less vulnerability to feed salinity [1]. Membrane distillation (MD) process is based on the difference of vapor pressure between feed and permeation solutions which is created by the temperature gradient [2]. Polyvinylidene fluoride (PVDF) is a semi-crystalline fluorocarbon polymer with special properties such as excellent chemical resistance, hydrophobicity, thermal and mechanical properties [3]. In this study, electrospinning process was used for the fabrication of hydrophobic Poly(vinylidene fluoride-co-hexafluoropropylene (PVDF-HFP) nanofibrous membranes for the treatment of brine from Reverse Osmosis desalination plant. The morphologies of membranes were studied by SEM and AFM. The crystallinity of the electrospun PVDF-HFP was studied by DSC, FTIR and XRD. The contact angle was used to evaluate the hydrophobic character of the electrospun membranes. The electrospun membranes were characterized in term of porosity, liquid entry pressure (LEP), and pore size measurement. SEM and AFM images show that different nanofiber diameters and physical properties, were obtained by changing solvent and varying polymer concentration. The FTIR and DSC results found that all membranes have lower crystallinity (4 to 10%), and the most abundant crystals were polar β and γ-phases. The water contact angles were studied of fabricated membranes and found for all membranes above 125° which means the hydrophobic membranes were successfully created. Optimized membranes were obtained with mean pore size, porosity, contact angle and LEP of 0.5 ± 0.1 μm, 70 ± 5%, 138° ± 2 and 2.5 ± 0.1 bar, respectively. Electrospun PVDF-HFP membranes were tested for the treatment of brine (60 g/L) by direct contact membrane distillation (DCMD) and a water flux value from 20 to 45 kg. h^{-1} .m^{-2} were obtained with a salt rejection up to 99.5%.

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Highly permeable polyamides of intrinsic microporosity (PAIMs) with tunable molecular weight cut-offs for organic solvent nanofiltration (OSN)

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While majority of existing organic molecules are only soluble in organic solvent media, selective organic solvent purification/concentration processes have become predominantly crucial in food and pharmaceutical industries targeting different solutes [1]. Diverse types of solvent-stable polymeric membranes have been reported with hierarchical molecular weight cut offs (MWCF), yet performances of low MWCF polymeric membranes are limited by low permeability and/or permeances. Here in this work, we present synthesis of and polyacrylonitrile-based thin-film composite membranes from a series of polyamide matrices constructed from extraordinarily contorted, rigid norbornene-based small molecules. Novel polyamide films showed at least 24 folds higher BET surface area, 13 folds higher of micropore volume, plus >200 times higher permeabilities compared with conventional MPD-TMC polymeric membranes. Under 10 bar and 80L/hr liquid flow rate, with a MWCF at ~ 910 Dalton PAIMs-3-PAN TFC membranes showed more than 32 L hr-1 m-2 bar-1 acetone solvent permeance, coupled with sub-nanometer skin layer thickness. Compared with PAN membranes, TFC membranes with PAIMs skin layers had trivial flux decrease. We concluded our crosslinked PAIMs TFC membranes have more sustainable performances over long operation span compared with phase-inverted membranes. It was also discovered that free-interface interfacial polymerization (IP) of porous polyamides yields thicker films ~30nm to ~1700nm, while free-interface IP of previously reported MPD-TMC matrix often yields films of <30nm in thickness [2]. All PAIMs have no adsorption towards neutral and negatively charged dyes. We are able to tune MWCFs by controlling monomer geometry towards molecular self-assembly. In conclusion, we believe the up-scalable chemistry and materials preparation will open up new windows to designing next-generation highly selective OSN membranes.

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Control of Graphene Oxide Membrane Performance in Water Filtration by Compaction During Fabrication

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Membranes fabricated from graphene materials have attracted huge interest due to the unique properties of these materials and ease of functionalization, which makes possible to adjust water permeation and rejection of species dissolved in water [1-3]. Graphene oxide (GO) membranes were prepared on Nylon support using different GO loads 200-600 mg/m^2 and GO suspension concentrations 2.5-40 mg/L. The compaction of graphene oxide (GO) membranes was tuned in a facile and scalable way by adjusting these fabrication conditions. The GO membranes were characterized by XRD, SEM, TEM, electron diffraction, AFM and tested in a cross-flow filtration system with aqueous solutions of pyridine, phenol and $NaNO_3$. GO membranes with higher GO load showed a higher compaction of GO sheets and lower interlayer spacing, with no-linear dependence of thickness membrane on GO load. GO membranes fabricated with GO suspensions of lower GO concentration of the suspension showed higher compaction. GO membranes presented heterogeneity in the structure, resulting from differential pressure employed in their fabrication by filtration. Thus, the most internal part of the membrane cross-section showed a much higher compaction. On other hand the outer layers of the membrane showed a structure of less-ordered GO nanosheets associated to agglomeration of GO nanosheets in the suspension during fabrication. The membranes fabricated with the highest GO load and the lowest GO concentration highly improved the rejection rate of all the solutes tested, from 7 to 35% for pyridine, from 5 to 45% for phenol, and even for $NaNO_3$, the rejection rate increase from 3 to 20%, while permeate flux ranged 1.6-26.1 L/(m^{2}hbar). The filtration behavior was related to compaction of the GO membranes, with the most internal layer of the membrane controlling rejection and flux, even when high compaction only affected few nanometers of the most internal layers of the cross-sectional area.

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Engineering Superior Multi-Functionalized Holey Graphene toward Ultra-Permeable Membrane for Emulsified Oil/water Separation

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The direct discharging of industrial oily wastewater into the environment with inadequate treatment is causing pollution issues to aqueous system. Membrane filtration process is considered one of the most effective solutions for treatment of oily wastewater, due to its low cost, energy efficiency and sustainability. In this work, we report the fabrication of novel hydrophilic multi-functionalized holey graphene (HG) membranes for oil-in-water emulsion separation using a facile vacuum filtration approach. Different loadings of the coating were utilized to optimize the membrane fabrication process. The fabricated membranes were thoroughly analyzed, and the performance of membranes were assessed in terms of flux and oil removal %. The modified HG membrane has high water flux with more than 8-fold, compared to graphene membrane. While the efficiency of oil-in-water emulsion separation was enhanced from 74 to 97% for control and modified membrane, respectively. This eco-friendly, facile, and versatile coating-based HG membrane may open a golden opportunity to be applied for the efficient treatment of industrial oily wastewater.

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Effect of graphene oxide on liquid water-based waterproofing bituminous membranes

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Bituminous membranes are widely used in many countries as a waterproofing layer to create a barrier to water and humidity for the protection of roofs, terraces, retaining walls, and any other kind of civil and industrial surfaces. However, the exposure to atmospheric conditions causes oxidative aging phenomena that compromise the performance and the useful life of the membranes. Several polymers have been used to improve membrane performance. However, some limitations were found such as low resistance to aging and high costs. The advent of advanced materials, such as nanomaterials, due to their unique properties, has opened new frontiers in the production of materials with new properties and functions. However, high production costs are the main limitation in large-scale use. In this work, small quantities of multilayer GO were used to produce high-performing liquid bituminous membranes. Significant improvements were observed in terms of mechanical strength, chemical stability, and water vapor barrier. The GO concentration was selected in order to have a low impact on production cost and, thus, to be competitive in the target market. In conclusion, the introduction of GO allowed us to produce membranes that are mechanically resistant; stable to drastic conditions of oxidation, such as heat and UV rays; highly waterproof; and with a very prolonged useful life. This has a significant environmental and economic impact, since these properties can be associated with a reduced number of interventions to restore the waterproofing material and, therefore, reduce the use of raw materials and the production of special waste.

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Changes of mechanical and spectroscopical characteristics of glassy polymeric membranes due to physical ageing

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The mechanical stability of membranes is the crucial property for their transferability into industrial practice. However, while mechanical characteristics of pure polymers are usually available, more practical long-term mechanical stability under relevant conditions does not commonly appear in literary sources. In this study, Raman spectroscopy and dynamic mechanical analysis (DMA) were used to assess and quantify the solvent-polymer interactions and polymer ageing by comparing the characteristics of newly prepared and aged membranes. Our focus was on three glassy polymers commonly used in membrane separation processes – cellulose triacetate (CTA), poly[1-(trimethylsilyl)-1-propyne] (PTMSP) and the archetypal polymer of intrinsic microporosity (PIM-1). The experiments were conducted with polymers equilibrated with the partially saturated vapours at 40 °C using a self developed DMA setup [1]. Methanol (MeOH) and dichloromethane (DCM) were chosen as the typical thermodynamically poor and good solvents for all three studied polymers, and p-xylene was additionally studied as a good solvent for PTMSP and PIM-1. For most polymer/solvent combinations, strong antiplasticization was observed for the less saturated vapours of good solvents, while plasticization prevailed for highly saturated vapours. Besides that, PIM-1 and PTMSP presumably underwent the solvent induced glass transitions in the p-xylene vapour saturated to approx. 80 %. Neither of the polymers underwent the glass transition upon the exposure to similarly saturated vapours of DCM, although these solvents showed similar mass sorption uptakes when using supporting vapour sorption microgravimetry measurements. Overall, the deterioration of the mechanical properties of the studied polymeric films in vapours of organic compounds appeared strongly compound-specific. This study, therefore, brings a better understanding of the interactions of polymers with solvents. In the future, such observations could facilitate the choice and the preparation of suitable separation membranes.

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Properties of polyhydroxyalkanoate membranes formed using non-halogenated solvents

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Though membrane technologies are well-established with ease of use and low energy consumption, the fabrication process needs to be more environmentally friendly and sustainable. Toxic solvents and petrol-based polymeric materials are typically used in the conversion, and this contributes to risks of negative environmental and health impacts. In this research, we use biopolymers from the family of polyhydroxyalkanoates (PHAs) as an alternative for petrol-based polymer membranes.[1] PHAs are fully biobased and biocompatible materials that can contribute to enhancing environmental sustainability and ongoing value in membrane applications. Polyhydroxybutyrate (PHB) and poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) membranes were prepared using evaporation-induced phase separation. So-called PHA-poor non-halogenated solvents, dimethyl carbonate and higher alcohols, were applied as environmentally more sustainable options compared to commonly used chloroform. For PHA-poor solvents, the solubility of PHAs is a function of temperature, concentration, and time. The critical temperatures and times for solvent solutions including solubility and gelation were evaluated systematically by differential scanning calorimetry. The temperature/time criteria were then applied as the key parameters to tailor membrane properties during phase separation. The nature of polymer-solvent interaction and solution concentration were considered and linked to the obtained membrane morphology as well as to the resulting mechanical and thermal properties.

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Understanding the non-solvent induced phase separation (NIPS) parameter effect during the fabrication of microporous DOCDA membranes

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DOCDA-ODA (5-(2,5-dioxotetrahydrofuryl)-3-methyl-3-cyclohexene-1,2-dicarboxylic anhydride- 4,4’-diaminodiphenylether) membranes were prepared by employing a non-solvent induced phase separation (NIPS). The key factors affecting the morphology of NIPS membrane include the solution parameters (i.e. polymer type and concentration, type of solvent, and additives) and the operating parameters (i.e. exposure time, relative humidity, dissolution temperature, and vapor temperature). In this study, we prepared membranes by changing THF/NMP ratio of solvent and exposure time. After membrane fabrication, we confirmed the membrane morphology how to change according to THF/NMP ratio and exposure time, and measured the gas permeance and H_2/CH_4 selectivity of each membrane.

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Dope temperature influence on structure and performance parameters in an industrial PES UF NIPS hollow fiber process

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Within the Biopharmaceutical industry micro- and ultrafltration membranes play a crucial role in the downstream processes of existing and future biopharmaceuticals. Hereby hollow fiber membranes are of great importance due to their lower shear forces applied on the substances in contrast to flat-sheet membranes. This is especially visible during the Covid19 pandemic for shear sensitive biopharmaceuticals like vector viruses or mRNA. However, besides the lower shear forces hollow fiber membranes are characterized by their structural and performance parameters. This work is focused on ultrafiltration (UF) membranes so that the main structural parameters were the inner fiber diameter, the wall thickness and the ovality. Furthermore, the main performance parameters are the retention behavior for dedicated Polyvinylpyrrolidones (PVP`s) and the permeability. These structural and performance parameters are influenced by the dope and bore composition as well as the overall production process. One interesting process parameter in this non-solvent induced phase separation (NIPS) process is the dope temperature with which the dope is conveyed from the nozzle into the Air Gap. This process parameter determines the viscosity of the dope and thus the kinetics on one hand as well as the thermodynamics of the precipitation process in the Air Gap on the other. Furthermore, the viscosity determines the wall thickness and the inner fiber diameter if all other process parameters are kept constant. This parameter could therefore be used as a tool in changing the structure and performance in a way that the desired UF hollow fiber membranes can be achieved. This work will therefore show how and to what extent this process parameter affects the above mentioned structural and performance parameters in an industrial Polyethersulfone (PES) hollow fiber UF membrane process.

Study of spinning parameters of polyvinylidene fluoride (PVDF) hollow fiber membranes preparation for membrane distillation

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Nowadays, the problem of freshwater shortage remains sharp, as freshwater resources are insufficient to meet the human and environmental demands. Recent water treatment technologies and particularly desalination methods confront sufficiently the freshwater depletion. In particular, membrane distillation (MD) is an emerging and state-of-the-art membrane-based separation process, which permits only vapor molecules of a hot feed to cross through a microporous hydrophobic membrane. Hence, the driving force is the differential vapor pressure across the membrane, while mass and heat transfer phenomena happen simultaneously. Polyvinylidene fluoride (PVDF) is one of the most attractive polymers in membrane fabrication due to its excellent chemical resistance, good thermal stability, high mechanical strength and good hydrophobicity due to low surface energy. Asymmetric PVDF hollow fibers (HFs) were fabricated in a spinning set-up by applying the dry-jet wet or wet phase inversion process, which are based on the spinodal decomposition principle [1]. During fiber spinning, instabilities or deformations are induced by the flow rate and the internal coagulant fluid composition affecting the geometrical shape of HFs and in turn their performance [2]. The wall thickness of HFs is non-uniform and their circular cross-sectional shape is deprived. The effect of air gap, bore fluid composition, flow rates of the dope solution and bore fluid, as well as take-up speed during the HF spinning process on HFs morphological properties and performance were evaluated. Specifically, the developed membranes were characterized by several techniques such as SEM, water contact angle, liquid entry pressure, porosity and tensile strength. Also, their performance was evaluated at different lab-scale membrane distillation processes such as direct contact and sweeping gas, employing saline water as feed. The results showed that the membranes present permeate flux above 10 L*m-2*h-1 and salt rejection above 99.9 %.

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Development of hydrophilic Polyvinylidene fluoride-based membrane by blending with Alkali Lignin

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Oil-wastewater produced by oil refining has become an urgent problem. Nowadays, special attention is paid in searching for eco-friendly treatment techniques. Membrane separation represents a suitable technology. Attempts are made towards two main directions: petrochemicals to biopolymers substitution and fouling reduction to increase membrane lifetime. One of the most used polymers in membrane manufacture is polyvinylidene fluoride (PVDF), which although boasting unique thermal-chemical-physical properties tends to easily get fouled when contacting non-polar compounds such as oil-based. The aim of this work was the development of hydrophilic membrane with minimized non-biodegradable content having reduced fouling tendency. To do this, Alkali Lignin (AL) was used as a hydrophilizing agent in a blend with PVDF, from which porous membranes were developed by Non-Solvent Induced Phase separation (NIPS) technique. Differential Scanning Calorimetry analysis showed the excellent miscibility of the new system PVDF/AL. The presence of AL in membrane substantially changed the membrane structure (analyzed by Scanning Electron Microscopy), the mechanical properties (86% Young's modulus increase), and increased the hydrophilic properties with beneficial effects on water permeance (7-fold increase), antifouling properties and cleaning efficiency (70% of pure water permeance restored and kept at a constant value). The PVDF/AL 1:1 membrane was able to fully retain oil droplets (of about 142 nm in diameter) from oil-water emulsion used as model solution of oily wastewater. The membrane maintained its high performance (rejection and flux) during the multiple cycles performed.

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Water selective membranes for use in electrocatalytic CO_2 reduction to syngas

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The CO2SMOS projects aims to convert biogenic CO_2 emissions into sustainable biochemicals and biomaterials. One of the CO_2 conversion technologies used is the electrocatalytic reduction of CO_2 to syngas. This is done in an intensified electrocatalytic membrane (eCMR) reactor that integrates three components: 1) A high-temperature proton exchange membrane (PEM) electrolyser; 2) A reverse water gas shift catalyst; and 3) A water selective membrane. The design of the integrated reactor enables an efficient heat and energy management and controls the flow path and the reaction contact time, which allows tuning the H_2/CO ratio. It is crucial for the produced water to be captured in order to ensure longevity of the PEM. The syngas will be further converted into valuable aromatics (benzene, toluene, ethylbenzene, xylene and paraxylene) using a catalytic process. In this work we present the development of the water extracting membrane within the CO2SMOS project, which needs to operate at elevated temperatures (150-200°C) and high pressure. We will present the chosen approach, the materials selected and screened and the first results obtained in the development of this membrane, including its gas and water vapor separation performance.

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Molecular layer deposition for the fabrication of desalination membranes with tunable metrics

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The recent advancement of semiconductor devices to the near-atomic scale necessitated the development of atomic layer processing methods, including molecular layer deposition (MLD). This gas-phase deposition technique creates semipermeable polymer films with precise control of composition and thickness. Herein, MLD was used to produce thin-film composite reverse osmosis membranes. Aromatic polyamide films as thin as 0.5 nm were applied to NF270 nanofiltration membranes using m-phenylenediamine and trimesoyl chloride. Within two molecular layers, desalination performance was affected. As film thickness increased to 15 nm (48 MLD cycles), performance progressed from nanofiltration to reverse osmosis metrics in terms of salt rejection and water permeance. With film thickness > 5 nm, rejection values exceeded a small sampling of commercial membranes. In all cases, a tradeoff between rejection and permeance was observed. Atomic force microscopy measurements indicate that MLD enhancement led to removal of small-scale roughness features and resulted in a root mean square roughness difference of <0.1 nm from the substrate. These initial MLD studies represent a novel processing approach that offers a potential pathway for the fabrication of membranes with finely tailored properties.

Fabrication of high-performance CO_2-philic mixed matrix membrane via interfacial blueprinting of the MOFs and polymer matrix for CO_2 capture applications

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The post-combustion CO_2 capture represents an essential contribution to climate change mitigation. Reducing CO_2 emissions by membrane technologies is considered clean and cost-effective compared to other techniques. As a hybrid class of materials, newly invented mixed matrix membranes (MMMs) possess both the inorganic and polymeric materials in a unique assembly applicable for the effective CO_2 separation from gas mixtures. Among several copolymers, Pebax with a rigid polyamide (PA) and a flexible polyethylene oxide (PEO) segments has shown high selectivity toward CO_2 due to the high PEO affinity towards CO_2. Similarly, the metal-organic framework (MOF) nanoparticles demonstrated the potential as an efficient material for improving the selectivity of MMMs without the loss of permeability. Among the countless MOF species, the reported MOF nanoparticles UiO-66-NH_2 provide severe basic nitrogen atoms as the binding sites of polarizable molecules of CO_2 via the base-acid and dipole-quadrupole interactions. Herein, we report the successful synthesis, characterisation and application of mixed matrix membrane based on Pebax1657 and MOF (UiO-66-NH_2-Pebax-1657) for effective CO_2 separation from CO_2/N_2, CO_2/H_2, and CO_2/CH_4 gas pairs. The addition of MOF particles with a loading amount up to 25 wt.% to the membrane matrix caused a significant enhancement of the permeability and selectivity by exceeding the Robeson upper bounds [1] for the targeted gases. With only 5 wt. % of MOF particles, the CO_2/N_2 selectivity increased by around 30 % with the improvement in CO_2 permeability compared to pure Pebax[2]. This was due to the observed uniform embedding of the MOF, CO_2-philic behavior of the chosen polymer and MOF, and their strong interfecial intraction of MOF with the membrane matrix via the bridging linkers. This method has proven to be blueprinting technique for reducing CO_2 emissions and carbon capture and storage applications.

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Interfacial engineering of electrospun polyvinyl alcohol/carbon nanotube mats into dense films for sustainable alcohol dehydration

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Pervaporation is a green process that consumes low energy and does not require solvents for separation. In this work, interfacial engineering of polyvinyl alcohol (PVA)/carbon nanotube (CNT) electrospun fibers was attempted to fabricate dense membranes for pervaporation. Ethanol and water were used as nonsolvent and solvent, respectively, to alter the nanoscopic configuration of the fibers through interfacial solvent interactions. PVA and PVA/CNT mats with micron-sized pores (>100 μm) were prepared by optimizing electrospinning conditions. The mats were compacted via immersion in ethanol. They exhibited asymmetric close-packed top and bottom layers and porous intermediate layers. Further consolidation was achieved using water to fuse the fibers. The mats with more than 1.5 wt% CNT loading were stable against water owing to the crosslinking between the CNTs and PVA. Because of the uniform dispersion of CNTs into the PVA matrix, the PVA/CNT dense films exhibited higher water/ethanol selectivity and water flux than those exhibited by similar films prepared by solution casting. Thus, the simple interfacial engineering of electrospun mats presented in this work successfully generates high-performance PVA/CNT membranes. A high alcohol dehydration performance and time-efficient membrane fabrication are achieved.

Selenium modified graphitic carbon nitride membrane for antibacterial properties

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Most South African communities rely on untreated water from rivers, streams, and dams that are constantly contaminated with pathogenic bacteria. Ingestion of water containing pathogenic bacteria such as V. cholera, E. coli, Salmonella, and S. aureus results in many diseases such as diarrhoea and shigellosis [1]. The use of nanocomposite membranes has been well received since it addresses the limitations of traditional methods. In this study, the prepared nanocomposite was incorporated into a polyethersulfone (PES) matrix through phase inversion method and used in the deactivation of B. cereus and E. coli. The synthesized membranes were characterized using scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDX), contact angle analysis, water intake capacity and water flux. The antibacterial properties of the membranes against E. coli and B. cereus were investigated with the disc diffusion assay, growth rate inhibition and Colilert-18 procedure. The modified nanocomposite membranes displayed improved antibacterial properties against B. cereus and E. coli achieving an inhibition of growth rate of approximately 100% at the highest concentration (0.9%). It was also found that the modified nanocomposite membrane has an inhibition zone range from 6-12 nm against the two bacteria strains tested. The SEM of the modified membrane results show that the cell's morphology of the B. cereus and E. coli was damaged and lost integrity of membrane, and this was due to the bactericidal effect of the modified membrane.

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Modification of Cellulose acetate Ultrafiltration Membrane with Zeolitic Imidazole framework-8

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Developing biodegradable polymers to fabricate filtration membranes is one of the main challenges of membrane science and technology. Cellulose acetate (CA) membranes, due to their excellent film-forming property, high chemical and mechanical stability, high hydrophilicity, eco-friendly, and suitable cost, are extensively used in fabrication of membranes for water and wastewater treatment. The possibility of modification of CA to improve permeability and stability has attracted the researcher's attention. In this study, the CA-based ultrafiltration membranes modified with Zeolitic imidazole framework-8 (ZIF-8) nanoparticles were prepared with various ZIF-8 contents. SEM, contact angle, porosity, pore size, AFM, and roughness tests were applied to the membranes in order to perform a detailed morphological analysis of the produced membranes. For the performance determination of the membranes, removal efficiency studies were carried out with bovine serum albumin (BSA) and two different dyes (Reactive Black 5 and Reactive Red 120). In addition, the antifouling performances of the membranes were also investigated. With the addition of ZIF-8, the pure water flux of the membranes increased, besides, the FRR (%) value was approximately 85% for the bare CA membrane, while it increased to 90% and above for the membranes with ZIF-8 addition. It was observed that the dye removal performance increased with the addition of nanoparticles. Notably, dye separation performance increased up to 98%. Overall, CA membranes were successfully modified by incorporating ZIF-8 nanomaterial and better-quality wastewater treatment performance was obtained.

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Mixed matrix membranes with MOFs for removal heavy metals from water

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Water contamination by natural and anthropogenic contaminants represents one of the most serious problems for the environment and human health [1]. The development of innovative methods for the removal of these pollutants from aquatic matrices is an ever increasing-demand. Membrane processes are among the most efficient technologies applied in this field, but are not yet sufficiently selective to remove heavy metals down to sufficiently low levels for drinking water. Incorporation of metal organic frameworks (MOFs) in membranes is a strategy to improve their efficiency, exploiting their high affinity for heavy metals [2]. In this work, novel biocompatible MOFs (bioMOFs), rationally designed to have a high affinity and high specificity, are embedded in porous membranes based on two different polymers: polyacrylonitrile (PAN) and Cellulose Acetate (CA). The employed filler materials consist of chiral 3D MOFs, with formula $\{CaIIZn_6II[(S,S)\text{-}L]_3(OH)_2(H_2O)\} \cdot 16H_2O$ (1), exhibiting functional channels decorated with – CH_2SCH_3 (L = mecysmox) and – $CH_2CH_2SCH_3$ (L = methox) thioalkyl chains derived from the residues of the natural amino acids L-Methylcysteine and L-methionine, respectively. Neat polymer membranes were prepared by nonsolvent-induced phase inversion (NIPS), optimizing the relevant parameters (e.g. polymer concentration, composition of the coagulation bath, additives, exposure to the air) in order to obtain membranes with the most suitable, sponge-like morphology. Mixed matrix membranes (MMMs) with various MOF loadings were prepared under the optimized conditions, and they were characterized in terms of morphology, hydrophobicity, water permeability. The metal retention was measured both in static and in dynamic mode, using water enriched with metal ions and/or other contaminants to simulate the real conditions of the purification process. The results of this study provide new insight into the MMM/heavy metal interaction, and will pave the way towards the development of more efficient membranes for drinking water production.

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Study of nanocarbon-based mixed matrix flat sheet membranes preparation for CO_2/CH_4 separation

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In recent years, gas separation, such as N_2 production, H_2 recovery, CO_2 capture, natural gas sweetening, is achieved with polymeric membranes, an efficient process competing sufficiently with well-established separation processes such as adsorption, extraction, cryogenic distillation. Membranes offer easy fabrication and scalability, low capital and operating cost, operation simplicity, low energy consumption and maintenance, mechanical reliability and small footprint. However, the gas separation performance of polymeric membranes is frequently restricted by Robeson’s trade-off upper bound [1]. Hence, nanoporous carbon-based nanofillers are incorporated into polymer matrices as a dispersed phase forming mixed matrix membranes (MMMs), which subsequently can provide an increase in selectivity and permeability simultaneously [2]. In current work, Pebax-1657, a commercial multiblock copolymer (Poly(ether-block-amide)), constituted by 40% rigid amide (PA6) groups and 60% flexible ether (PEO) linkages, was selected as the base polymer of MMMs. Versatile carbon nanofillers, in specific, raw and treated (plasma or oxidized) multi-walled carbon nanotubes (MWCNTs) and graphene nanoplatelets (GNPs) were incorporated into the polymeric matrix in order to improve the gas separation performance and polymer’s structural properties. The MMMs were prepared by the casting method. A treated at 60°C in an oven for 3 h was followed in order to evaporate any remaining traces of the residual solvent. The developed membranes were characterized by SEM, FTIR and their mechanical properties were evaluated by tensile testing. The tensile strength of MMMs was enhanced by 18.4% compared to the pure polymeric membrane (9.2 MPa). In addition, the effect of nanofiller type, structure and amount to real binary CO_2/CH_4 (10/90 vol.%) mixture separation performance was evaluated under elevated pressure conditions. Specifically, a CO_2/CH_4 separation factor of 20.7 was achieved (CO_2 permeance 2.2-3.5 GPU). Overall, MMMs exhibited enhanced gas permeabilities (up to threefold values) without sacrificing gas selectivity compared to the corresponding pure polymeric membrane.

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Addition of different powder activated carbon (PAC) in non-solvent induced phase-inversion membranes: effect and characterisation.

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Secondary and tertiary treatments applied to wastewater normally allow its discharge into natural watercourses. However, additional treatment should be implemented to reuse it. Membrane processes are an accurate and thorough solution to getting recyclable water, but their main limitation is associated to fouling and adsorption of organic materials to the surface, affecting membranes performance. Liu et al. (2018) already used PAC in polyvinylidene fluoride (PVDF) ultrafiltration membranes, showing excellent resistance to fouling [1]. In this study, three different PAC -noted as PANREAC, NORIT R and NORIT PK- have been characterised obtaining Pore Size Distribution (PSD), specific surface area, as well as thermal stability. Afterwards, non-solvent induced phase separation (NIPS) membranes were produced with PES/DMA solution, and different concentrations of the different PAC were dispersed in DMA. Membranes were characterized and compared in terms of their performance: filtration tests as well as FESEM analysis showed different membrane behaviours depending on both PAC type and its percentage in the membrane active layer. The highest PEG rejection among the performed membranes was NORIT R 0.5% wt., reaching 80.34% in rejection index. In general, PAC membranes led to bigger pore sizes than those from common NIPS membranes.

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Impact of layered perovskite oxide on pervaporation performance of polymer membranes

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Mixed matrix membrane is considered a great attempt to fabricate membrane with superior permeability and selectivity, excellent durability, high mechanical stability, and economic viability. Currently, inorganic membranes based on perovskites are widely used for oxygen purification in catalytic membrane reactors and there are several attempts to use exfoliated perovskite nanosheets as membrane material. However, there is few data on the use of perovskites as modifiers for polymer membranes for pervaporation. Perovskite-type layered oxides seem promising materials for incorporation into membranes due to combination of two factors: i) the possibility of creating selective transport channels through the interlayer space of the perovskite structure; the presence of ionic compounds in the polymer matrix leads to an increase in the solubility of polar molecules and a decrease in the solubility of non-polar media, and ii) the possibility of increasing the distance between polymer chains, which should increase the permeability of the membranes due to the frame of inorganic materials that can prevent the tight packing of polymer. This work aims to develop a method for manufacturing membranes based on composites with perovskite-type layered oxides for pervaporation separation. The membrane morphology was studied by scanning electron microscopy. The thermal stability of the membranes was studied using thermogravimetric analysis. The glass transition temperature of the membranes was obtained by differential calorimetry. The membranes were also characterized in terms of contact angle, Fourier trans- formed infrared spectroscopy, X-Ray diffraction and sorption degree. The transport characteristics were studied by carrying out pervaporation experiments with organic media. The developed membranes demonstrates increase the flux with good separation effectivity, which indicates that perovskite-type layered oxides can be considered as candidates of novel fillers for polymer membranes to be explored in pervaporation separation.

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Organic solvent separation using a hybrid of size-controlled porous organic polymer and polyethersulfone membrane

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Materials research for fabricating Mixed matrix membranes (MMM) are considered a promising strategy for both aqueous solutions and organic solvents separations. Traditional polymeric membranes are commercially used for solute-solvent separations and are susceptible to low chemical stability when exposed organic solvent separation [1,2]. Separating alkane isomers, which is extremely difficult due to their similar physiochemical properties, is of great benefit to the production of gasoline [1]. This has prompted researchers to construct rigid and tunable pore size materials for incorporation into membrane matrix to create advanced membranes. Here, we report the synthesis and incorporation of a new interdigitated two-dimensional (2D) porous coordination polymer: $[Zn(1,3\text{-}H_2BDP)_2(BDC)_2]DMF$ (PCP) into a polyethersulfone matrix via phase inversion method. The loadings of PCP were varied from 0 and 8% to produce MMM0-MMM8. The membrane surface and cross-sectional pores increased as loadings of PCP increased. The contact angle measurements also revealed an increase in hydrophilicity as the PCP loadings increased. The membrane flux and selectivity were performed using a dead-end filtration cell as shown in the scheme 1. The gas chromatography (GC) was used to quantify the hexanes in the permeate solutions. The selectivity coefficients showed a preference for linear hexane (nHx) over 2,3-dimethylbutane (2,3DMB) hexane, with selectivity coefficients ranging between 1.0 and 1.2 in all membranes (MMM0-MMM8).

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Synthesis and Gas Permeation Properties of the 2D ZIF-8 and PEBAX Mixed Matrix Membranes

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ZIF-8 and its derivates have attracted attention as promising nanofillers in mixed matrix membranes (MMMs) for gas separation due to thier tunable pore size, high specific surface area, and excellent physical and chemical properties. However, the filler agglomeration and the poor interfacial compatibilities between ZIF-8 nanoparticles and polymers are recognized as main drawbacks of enhancing gas separation performance. Controlling the ZIF-8 morphology to two-dimensional shape is expected to overcome those hurdles. In this research, we synthesized two-dimensional ZIF-8 (2D ZIF-8) nanosheets and incorporated those fillers into the rubbery polymer (PEBAX) with different contents. The fabricated 2D ZIF-8/PEBAX MMMs showed the highly oriented 2D ZIF-8 sheets in the polymer matrix and improved gas permeability and selectivity.

Development and Characterization of High Porosity Recrystallized Silicon Carbide Membrane Substrate

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Recrystallized silicon carbide (R-SiC) is a high purity grade of SiC sintered through a sublimation-condensation process at temperature above 2000 degC It has been used to manufacture porous structure ceramic membranes for liquid filtration due to its enhanced permeate flux, superior thermal and chemical resistance and high thermal shock resistance. It has been reported that R-SiC porous ceramic filters have porosity around 40% in general [1]. The higher the porosity, the higher the water permeability, therefore higher production capacity. In this study, a new type of R-SiC ceramic filter with porosity higher than 50% was obtained with a type of carbonhydrate based pore former. The porosity of R-SiC filters is tailored by adjusting the weight content of varies organics that were chosen as pore formers. In this case, 5wt%, 7.5wt% and 10wt% were examined in finding the optimal recipe to high porosity R-SiC. Corn, potato and ultra refined cellulose were chosen as pore formers for this study. All samples were placed in cruisibles with the same dimension and sintered under the same sintering program. Interestingly, the carbon residues generated from the carbonhydrate facilitated recrystallization process. The results showed that using cellulose as a pore former yields a porosity higher than 50% in comparison to the corn and potato starch. The pore size was measured to be 12 – 14 µm. The mechanical strength tested by four-point bending was measured to be 30 - 35 MPa. After sintering, the SEM microstructure showed that adding organic as pore formers did not change the morphology of microstructure. The SEM microstructure of corn, potato and cellulose showed strong neck formed between SiC particles. However, using cellulose as a pore former yielded a more porous structure than the others, given that it had the highest porosity.

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Scaling-up of zeolite ZSM-5 membranes preparation for natural gas treatment

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Zeolite ZSM-5-membranes are ideal for separating organic solvents from water [1], branched and non-branched hydrocarbons [2] or higher hydrocarbons from natural gas [3]. However, for industrial application the synthesis of MFI membranes have to be scaled-up in terms of membrane geometry and manufacturing technology. ZSM-5-membranes (Si/Al = 300) were prepared by slurry coating for seeding (MFI powder Clariant), hydrothermal crystallisation and detemplating at 450°C [1] on ceramic support tubes with supporting layer of 100 nm pore diameter. In first step ceramic supports with a length of 250 mm and an increasing number of channels (1, 4, 7, 19 channels) were used. The success of the synthesis was checked by PV-separation of ethanol from water (5 wt% ethanol) at 40°C. In a second step, 15 membranes with a length of 600 mm and 19-channel geometry were synthesized in a single batch. Two batches were run. The membranes were examined using permporometry. In PV tests with ZSM-5 membranes prepared on ceramic supports with different numbers of channels all membranes showed a clearly hydrophobic separation behavior and were able to prove their functionality. Two 600 mm long membranes in 19-channel geometry from each synthesis batch of 15 membranes were examined using permporometry. It was found that all membranes achieved a relative permeance, i.e. a flux through membrane defects, of <1%. With three membranes, the value was even 0.1%. 30 membranes were tested in natural gas of 90.4 % CH_4, 4.4 % n-butane, 2.7 % ethan, 0.5% propane and 2 % higher hydrocarbons at 2 bar feed and 250 mbar permeate pressure. Everage n-butane/CH_4-selectivities of 24 with best values up to 44 and everage permeances of 3.7 $m^3/(m^2hbar)$ = 4.6 10-7 $mol/(m^2sPa)$ were achieved. This was the first time that the synthesis of ZSM-5 membranes had been successfully scaled up, both in terms of geometry and manufacturing technology.

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A New Through-the Membrane-Flow Atomic Layer Deposition Reactor Design Used for the Development of Ceramic Nanofiltration Membranes

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Atomic layer deposition (ALD) is performed using one of two reactor designs, different in precursor [1]. Firstly, temporal ALD (t-ALD) reactors are widely used because of their excellent deposition performance, while needing long cycle times to allow proper evacuation of the precursors. Secondly, spatial ALD (s-ALD) is popular in commercial applications for its high throughput, but deposition is limited to planar samples. Ideally, an ALD reactor for the modification of ceramic membranes would be able to fit and modify membranes of various shapes and sizes, with as low cycle times as possible. In this work, a new Through-the Membrane-Flow ALD (TMF-ALD) reactor design for atomic layer deposition is introduced. The flow scheme was designed to direct the precursors through the pores of ceramic membranes under tunable pressure up to 5 bars, with the exhaust of the setup at atmospheric pressure. Preliminary results show that a pressure drop of 0.2 bar over the membrane allows for a pore size reduction of an α-alumina membrane from 20 nm to 14 nm. Furthermore, pressureless flow-type operation can decrease the pore size of 5 nm γ-alumina membranes to the nanofiltration (<1 nm) range. For example, 25 cycles of $TiCl_4$ and water vapor as precursor and co-reactant, respectively resulted in a membrane with a pore size of 2 nm. A similar deposition of 50 cycles followed by a thermal treatment, produced NF membranes with a polyethylene glycol (PEG) molecular weight cut-off (MWCO) < 500 Da.

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Semi-flexible ultrafiltration inorganic membrane based on GO and ultra-long hydroxyapatite for wastewater purification

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Polymeric "organic" membranes dominated the market respect to their inorganic counterpart due to their lower production cost, operation flexibility, and ease of preparation and scaling up. However, polymeric membranes suffer from a short lifetime due to fouling problems and limited mechanical/thermal and chemical stability. On the other hand, ceramic membranes, a kind of inorganic membrane, can be easily backwashed and cleaned with various cleaning agents and sterilized at high temperatures, with no effect on their performance and/or lifetime overcoming some of the most diffused drawbacks typical of polymeric materials. However, ceramic membranes were rarely used in wastewater purification due to their high fabrication and operating cost, and low membrane performance. In this work, we developed new hydroxyapatite-based ultrafiltration (UF) membranes using ultra-long inorganic hydroxyapatite of one dimensional size (1D) as a base of ceramic materials combined with two dimensional (2D) graphene oxide (GO). Different ratios of GO were blended with ultra-long hydroxyapatite and the membranes were fabricated via a simple vacuum-assisted filtration technique. The properties and performance of produced membranes were evaluated through different chemo-physical characterization techniques such as SEM, contact angle, and pure water permeability. The developed membranes capable to provide semi-flexibility with acceptable mechanical properties in addition to the superior hydrophobicity with an average contact angle 146˚. The super hydrophobic character of the obtained membrane strongly recommended to resist organic fouling of such kind of these membranes. There is no notable change in the contact angle values of the developed membranes even with the blended ratios of GO, however there is a notable improvement in the pure water permeability with GO.

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Influence of silicon carbide content and temperature on mechanical and electrochemical characteristics of ceramic membranes based on kaolin and saponite

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Ceramic membranes have been used for a wide range of applications. The development of ceramic membranes is much slower than that of polymer membranes because of their high cost and lower processability. However, they have good stability and are more resistant to fouling. Additional antimicrobial and photocatalytic properties that can make membranes even more effective in preventing their contamination. Ceramic membranes are mainly made of Al_2O_3, SiO_2, TiO_2, ZrO_2 or their mixtures, the application of which is limited due to high cost. Therefore, the development of ceramic membrane compositions that are both inexpensive, reliable, and productive remains relevant. In this work, samples of ceramic membranes based on Ukrainian clay minerals - kaolin and saponite were obtained. The use of clay minerals aimed at reducing the cost of ceramic membranes. Calcium and ammonium carbonates were added to create the specified porosity (60-70%), and silicon carbide was added to regulate the mechanical properties. The temperature of final treatment of ceramic membranes was also regulated in the range of 800-1100 ºC. Twelve samples were obtained, which differed in the content of silicon carbide and the temperature of the final treatment. The samples were tested for bending strength according to the standard technique, and the electrical resistance and flow potential were determined. The results indicate a significant effect of silicon carbide content and temperature on the mechanical and electrical properties of the membranes. It was found that the mechanical properties are largely dependent on both the temperature of the final membrane treatment and the phase composition of the membranes. The electrical properties are mainly determined by the phase composition of the membranes. As further directions of research, it is planned to optimize the composition of ceramic membranes, test them for transport characteristics, and modify them to impart antimicrobial and photocatalytic properties.

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Fabrication of Polyelectrolyte Complex Hollow Fiber Membranes through Salt Dilution Induced Phase Inversion

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Currently, the fabrication of commercial hollow fiber membranes relies on the usage of harmful, reprotoxic organic solvents, which will potentially be banned in the EU shortly [1]. Therefore, research and industry focus on the search of suitable alternative sustainable solvents for membrane fabrication or even alternatives to the organic solvent-based phase separation process. A promising process is the fabrication of porous membrane structures via the all aqueous phase inversion of polyelectrolytes. However, hollow fiber membranes with sufficient mechanical stability have not yet been reported. We present the spinning of free-standing, defect-free and mechanically stable hollow fiber membranes using the salt induced phase separation of polyelectrolytes. An aqueous polymer solution containing polystyrene sulfonate (PSS), polydiallyldimethylammonium chloride (PDADMAC) and potassium bromide (KBr) is extruded with an aqueous lumen fluid through a spinneret into a purely aqueous coagulation bath at 0°C. Subsequently, the fibers receive a photo-induced crosslinking with the crosslinker disodium 4,4'-diazidostilbene-2,2'-disulfonate tetrahydrate (DAS) inducing the desired mechanical stability. The achieved asymmetric hollow fibers with an inner diameter of around 450 µm show a pure water permeance of 0.5 LMH/bar and a molecular weight cut-off of 200 Da. Furthermore, the all aqueous phase separation allows the incorporation of bioactive components like enzymes into the polymer solution. In filtration experiments, the enzymes alkaline phosphatase (ALP) immobilized in the fibers prove to be highly active. By showcasing the incorporation of alkaline phosphatase, we emphasize the huge potential of the fabrication procedure not only as green alternative to organic solvent-based approaches but also as a universal platform for the fabrication of bioactive membrane materials.

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Bio-Inspired Membranes with Directional Water Transport Characteristics

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Directional transport is exploited by several natural organisms as a strategy to retain water. Some species of plants achieved this property by developing a protective membrane called cuticle, whose main function is to regulate the water exchange with the environment. Cuticles consist of a polyester matrix mixed with polysaccharides that extend from the internal side and gradually fade in the transversal direction, and an external layer of hydrophobic waxes. In this work, we fabricated artificial dense membranes using a hydrophobic poly(styrene)-block-poly(butadiene)-block-poly(styrene) (SBS) copolymer and hydrophilic polyvinyl alcohol (PVA) with the aim of mimicking the polarity gradient of the cuticles' structure. The water-induced plasticization of PVA, combined with the asymmetric design, results in directional water transport when the membranes are exposed to a relative humidity gradient. The directionality can easily be tuned by varying the composition and the structure of these bio-inspired membranes, and this enables the development of a wide variety of new materials, whose properties may be useful for packaging applications.

Magnetic hybrid separation materials with controlled properties – covalently modified PVDF with lanthanides oxide particles

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Tunable and selective transport across membranes is considered to be a key limitation in membrane technology. Intervening the fluid matrix on the surface and inside membrane pores influences molecular transport across the membrane. Therefore, a new strategy is proposed where magnetic nanoparticles (lanthanide oxides) are anchored via a silane-based spacer onto the surface. The modification pathway is bio mimicked from the nature, i.e. cilia mechanism exists in many biological tissues. The entire modification has been accomplished via stable covalent bonding. PVDF surface has been activated with piranha reagent prior to the functionalization with silane molecules (3-isocyanatopropyltrimethoxysilane). In parallel, gadolinium oxide was modified with 3-Aminopropyltrimethoxysilane. Finally, the reaction between terminal groups (amine and isocyanate) of both components took place. The material possessed attached particles on the long enough chains to make them movable. The presented approach allows to tune roughness, hydrophobicity and adhesion of the generated samples. With the implementation of electron paramagnetic resonance, it was confirmed that the silanization process did not diminish the magnetic features of lanthanide oxide particles.

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NMR and MS characterization of apple juice and partial removal of sugar by dia-nanofiltration process

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Apple is one of the most consumed fruit worldwide as well as its juice [1]. However, while consumption of whole apples can improve vascular function, decrease systolic blood pressure and reduce cholesterol levels, clear apple juice has been associated with adverse effects, mainly related to its high fructose and low fiber content [2]. Therefore, the partial removal of sugar in apple juice without changing the rest of the natural composition and bio-functional properties is a remarkable technical challenge [3]. In the present work, apple juice was characterized using Nuclear Magnetic Resonance (NMR) and Mass spectrometry (MS) coupled with HPLC-UV. In order to reduce the sugar content and to preserve its phenolic composition, the juice was first clarified by ultrafiltration and then nanofiltered through a combination of diafiltration and batch concentration processes. Three different nanofiltration membranes with MWCO in the range of 200-600 Da were tested. For all selected membranes, the recovery rate of both sugars and phenolic compounds decreased in the retentate by increasing the diafiltration volume; however, the separation factor between phenolic compounds and sugars remained almost unchanged during the diafiltration process. Among the investigated membranes, a thin-film composite membrane with the lowest MWCO (200-300 Da) showed the highest retention of phenolic compounds in both diafiltration and concentration processes. More than 70% of phenolic compounds were recovered in the retentate stream of this membrane at a volume reduction factor of 4, while recoveries of glucose and fructose were 41 and 44%, respectively. Therefore, the combination of diafiltration and batch concentration with this membrane represented a good compromise to remove up to 60% of sugars from apple juice with minimal losses of phenolic compounds.

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Microfiltration of milk: Linking membrane fouling with the colloidal properties of milk

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Separation of milk proteins by microfiltration is a technology for creating ingredients with novel functionalities. Milk proteins consist of two major groups; Caseins which are present as supramolecular protein aggregates and whey proteins found in the serum phase. A high permeate flux is obtained by using diafiltration and a minimum of caseins transmitting with the whey protein-containing permeate is wanted. However, the potential of batch to batch variation in the composition and functionality of the retentates, regardless of similarity in composition, clearly points to the need for a better understanding of how the physical and chemical properties of the proteins present in the retentate, and their supramolecular structure, may affect the permeation behavior during membrane filtration. The composition of the diafiltration media has been shown to affect the permeability of whey proteins explained by a fouling layer formed during dead-end filtration [1], and the functionality of the casein-containing retentates [2]. It was therefore hypothesized that a better control of the formation of a fouling layer is linked to a control of the physical and chemical changes occurring to the milk concentrate during filtration, and this will be key to the production of consistent, high quality and functional casein/whey streams. This work aimed to understand, through an in-depth analysis of the protein and mineral composition of the streams, the changes occurring to the fouling layer and the corresponding permeations. Milk was processed with microfiltration membranes and diafiltered with two different diafiltration media (UF permeate and water). Experiments were conducted with a custom-built setup, using separate clean membranes at various volume fractions. Filtration parameters were measured, and in addition to the chemical composition of the permeates and the fouling layer. The result will aid in a deeper understanding of the differences in the fouling mechanism during cross-flow microfiltration of skim milk

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Sequential ultrafiltration for the fractionation of lignin and galactoglucomannan from acidic Norway Spruce steam explosion liquors

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In a biorefinery, harsh pretreatments are often required in order to overcome the recalcitrance of biomass and ultimately the utilization of the inherent components. However, these pretreatments usually hydrolyze and solubilize the hemicellulose which decreases the overall yield of the process. In order to overcome these loses, the pretreatment can be performed at a lower pretreatment severity with the aid of enzymes, such as esterases, that target lignin-carbohydrate complexes (LCC) for the delignification. However, it has been shown that even at low severities, oligomeric and polymeric hemicellulose are solubilized, yielding solutions with concentrations as high as 6 wt%. The recovery of hemicellulose from diluted process solutions can be costly using conventional methods such as, evaporation, precipitation and adsorption. A cost-effective alternative would be the use of membrane filtration for the fractionation and concentration as has been demonstrated in previous studies [1]. In the present work, sequential ultrafiltration was performed using three regenerated cellulose membranes with the molecular weight cut-offs of 10, 5 and 1 kDa. The feed for the ultrafiltration was produced by steam explosion of Norway spruce at 210°C for 5 minutes using pre-soaking with dilute acids to alter the severity. The liquors were characterized for content using the NREL protocol together with HPLC and the structure of the hemicellulose and lignin was determined using HSQC 2D NMR and phosphorous NMR. The results have shown that the presented scheme can produce a hemicellulose product with a total yield of 70%, purity of 80% at a maximum capacity of 140 L/m^2h and volume reduction of 90 %. Structural data using HSQC 2D NMR also indicated that the major component concentrated was galactoglucomannan. Minor lignin aromatic and LCC signals were also detected which indicated that these impurities were probably linked to the hemicellulose through LCC linkages.

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LIBERATE: Nanofiltration for the recovery of lignin and depolymerization products

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Lignin, a complex aromatic polymer, is the second most abundant biopolymer on Earth and is mainly used to produce energy by incineration. Lignin depolymerization into value-added aromatic products under mild conditions would greatly benefit both the value-added fine chemical industry and green/renewable chemistry sector. Degradation of Kraft lignin into vanillin and phenolic compounds is one of the studied processes in LIBERATE but the subsequent isolation of the vanillin from the largely unreacted electrolyte represents a significant challenge. The available processes are mainly ion exchange resin extraction, liquid-liquid extraction and membrane separation being membrane separation the most interesting and advantageous process due to its continuous, easily scalable, modular and low-cost process. In the present study, commercially available ultrafiltration and nanofiltration membranes from NX Filtration were tested for the separation and recovery of vanillin and unreacted lignin. Cross-flow filtration experiments were performed using ultrafiltration membranes and two direct nanofiltration membranes with cut-offs of 800 and 400 Da. The retention capacity was calculated by analyzing the lignin, vanillin and acetovanillone concentration in both feed and permeate streams by UV-Vis spectroscopy and high-performance liquid chromatography. In addition, the molecular weight of lignin was determined by gel permeation chromatography in both feed and permeate streams. This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 820735.

The Performance Comparison and Troubleshooting of PVDF Transfer Membranes Using Different Chemiluminescent Substrates

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Western blotting is a technique which consists of SDS-PAGE gel electrophoresis to separate proteins, electrotransfer of the proteins from gel to hydrophobic membranes and finally selective detection of the proteins. It is a powerful method to investigate the presence, relative abundance, relative molecular mass, and interactions of proteins [1]. Even though Western blot is very sensitive to variations in experimental design, methodology, and technique, this method is conventional in diagnostic and therapeutic applications as well as academic researches. Even insignificant differences in the reagents and parameters applied in each experiment may have a surprisingly strong influence on the results [2]. Thus, troubleshooting knowledge is always a required skill in performing this technique. Different types of membranes could be used in the electrotransfer step such as PVDF, Nitrocellulose or Nylon. The choice of the membrane type depends on the desired accuracy, testing condition and sample type. This study focuses on comparison of the performance of the GVS PVDF transfer membranes (0.45 and 0.22 μm) with a competitor by applying different chemiluminescent substrates to detect the Beta Actin proteins in a HeLa whole cell lysate. In addition, the causes of unaccepted results in a western blot test are discussed and the solutions are presented. The results showed that GVS membranes had a better or equal performance with respect to the competitor membranes. Aiming at detecting very small quantity of the targeted protein in a western blot, incubating the membranes in chemiluminescence Light Wave Max would be the best option comparing to the other lower grades of chemiluminescence substrates. Moreover, according to the troubleshooting system, high background could be the most problematic reason of unreliable results.

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Nanofiltration and ultrafiltration of sugar beet molasses used for the production of hydroxymethylfurfural.

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Today sugar beet molasses is an underutilized by-product in sugar mills. Sucrose in the beet molasses has the potential to be a raw material for a wide range of biorefinery processes, e.g. in the production of the platform chemical 5-Hydroxymethylfurfural (5-HMF) [1]. However, the 5-HMF formation process is affected by purity and concentration of the raw materials. In this study, two membrane processes, ultrafiltration (UF) and nanofiltration (NF), were studied for the purification of sucrose in sugar beet molasses to improve 5-HMF production. Two ceramic tubular membrane modules - 10 kDa UF membrane (Atech, Germany) and 200 Da NF membrane (IKTS Fraunhofer, Germany) - were evaluated for the concentration of diluted molasses in recirculating batch cross-flow filtration [2]. During the concentration studies of the molasses, normalised fluxes of 11-34 L/(m^2h·bar) (UF) and 14-27 L/(m^2h·bar) (NF) were achieved at 60°C with volume reductions of 65-85%. The retentions of different feed components varied significantly for the two membranes, and a strong correlation to molasses concentration was observed. The sucrose in the purified molasses was then hydrolysed using Saccharomyces Cerevisiae. It was found that the NF pre-treated molasses had a higher reaction rate compared to crude sugar beet molasses. In the subsequent 5-HMF production experiments, UF pre-treated molasses was superior for the conversion of fructose to 5-HMF. UF pre-treated molasses could reach close to 100% fructose conversion in comparison to only 49% fructose conversion for crude molasses, without impacting the 5-HMF selectivity (57% and 59% selectivity, respectively). It was concluded that NF and UF are both suitable to increase the overall process productivity of 5-HMF, one with regards to the hydrolysis process and the other for the dehydration reaction.

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Nanofiltration in Purification of Hemicellulosic Sugars in Biorefinery Side Streams

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Attempts towards climate change mitigation are driving the world to replace non-renewable fossil resources with renewable bio-based raw materials. This has created an increased demand for new product concepts and reaction routes, comprising especially biorefineries with a wide range of (bio)products. In 2020, more than 2000 plants utilizing bio-based raw materials were known to operate in the EU [1]. However, efficiency of the biorefineries could be further improved by enhancing the recovery of valuable compounds from the side streams and closing the material loops via process intensification. In this research, nanofiltration (NF) with an NF99HF membrane from Alfa Laval was studied as a potential technology in the purification and concentration of hemicellulosic sugars from side streams of lignocellulosic biorefineries. The aim of the research was to produce a sugar stream of high purity for further processing into value added products. The effect of operation conditions (i.e., feed flow rate and pressure) was studied to further optimize the recovery of sugars. The results show high separation efficiency between organic inhibitory compounds and sugars: as high as 97% and 89% rejections were achieved for glucose and xylose, whereas under 20% or even negative rejections were observed for formic acid, acetic acid and furfural. Diafiltration (DF) was studied in further purification of the sugar streams. However, a minor contribution of DF in increasing the purity of the NF retentate was observed in this case and with the selected membrane. Future studies should focus on hybrid separation methods to remove the inorganic compounds from the sugar streams to further increase the purity.

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Novel paths towards sustainable membrane manufacturing in industrial scale

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Within the last decades the transition towards eco-friendly technologies has spread over all industry branches. However, solvents such as dimethylacetamide (DMAC), N-methyl-2-pyrrolidone (NMP) or dimethylformamide (DMF) are still widely applied in non-solvent induced phase separation (NIPS) processes in both flat-sheet and hollow fiber membrane productions. Besides their outstanding ability to solve various membrane polymers such as poylsulfone (PSU), polyethersulfone (PESU) or polyvinylidene fluoride (PVDF) they exhibit low biodegradabilities, high toxicities and potential water hazards. Within this work an existing membrane manufacturing process based on solvents and additives with limited sustainability will be converted based on the aspects of green chemistry conventions. Therefore, compositions of dope solutions and precipitants will be adapted using only biodegradable and/or recyclable ingredients which reveal no potential for environmental or health endangerment. In that matter, Hansen solubility parameters will be utilized first to evaluate the compatibility between solvents and membrane materials while cloud-point measurements and flat-sheet membrane castings will be performed subsequently to obtain insights into the phase separation characteristics of dope solution recipes. In addition to the inherent features of green chemistry and solvability other soft factors such as economic viability, availability and batch comparability shall be considered as well to provide an implementable manufacturing process at the production plant. Simultaneously, exemplary membranes of the existing ultra- and microfiltration portfolio shall be replicated while preserving the high-quality standards of Sartorius Stedim Biotech products and its outstanding performances in terms of permeability, retention and stability.

Membrane processes for fractionation of bioactive compounds extracted from tomato leaves and used in vivo to induce plant defense

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The use of agricultural waste as a source of bioactive compounds for the development of functional products opens a pathway towards a sustainable and circular economy. In this context, tomato leaves are rich in carbohydrates that can be used as pesticides in agriculture, since they function as damage-associated molecular patterns that regulate plant defense signaling [1], so their recovery with a green technology is highly required. Membrane technology is an unexplored approach for biorefinery of tomato leaves, and the present work investigated the fractionation of major biomolecules (carbohydrates, biophenols and proteins) from this biomass by a combination of aqueous extraction and membrane processes [2]. The aqueous extract was processed by a two-stage membrane cascade using commercial membranes with different physicochemical properties. The fractionated extracts were sprayed on tomato leaves to induce an immune response in the plant prior to infection with Pseudomonas syringae, which can lead to a loss of crop yield and tomato quality. The first stage of the membrane system allowed the fractionation of proteins from carbohydrates and biophenols using 30 kDa regenerated cellulose membrane. Further fractionation of carbohydrates and biophenols was achieved in the second stage. In this case, the 5 kDa polyethersulfone (5 kDa PES) membrane showed the greatest difference between the rejection coefficients of carbohydrates and biophenols. The results of the in vivo tests showed a decrease in the P. syringae population when the tomato leaves were treated with the aqueous extract. In particular, the fractions obtained with the 5 kDa PES membrane caused a decrease in the population growth of P. syringae by at least one order of magnitude compared to the control.

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RAWMINA: Integrated Innovative Pilot System for Critical Raw Materials Recovery from Mines Wastes in a Circular Economy Context

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Critical Raw Materials (CRMs) are crucial to Europe's economy. They form a strong industrial base, producing a broad range of goods and applications used in everyday life and modern technologies. CRMs are needed for significant economic and strategical sectors, as for example manufacturing batteries, metals, electronic and medical devices, automotive, defence or renewable energy sectors. However, limited supply and difficult substitution to CRMs is a growing concern within the EU. RAWMINA aims to develop and to demonstrate the RAWMINA pilot system: an industrially scalable and flexible innovative pilot in continuous operation for Mine Waste (MW) valorization, achieving 95 % recovery rate and 95 % selectivity for CRMs (Co, Sb, Ge and W), and 80 - 90 % recovery rate and 95 % selectivity for Au, Ag and Fe-based high-value products, whilst reutilizing 90 % of water. RAWMINA will implement and standardize an innovative energy, water- and cost-effective pilot system able to treat up to 100-150 kg MW/day on an industrial demonstration (TRL7) including efficient and robust process control. To achieve these aims, RAWMINA's activities include MW sampling, MW dressing and characterization, upscaling of innovative technologies such as continuous bioleaching, iron removal with magnetic separation and CRMs selective recovery through a combination of nanofibrous composite materials and electrocoagulation processes. The novel nanofiber composite materials (NCMs) with selectivity towards Co, Sb, Ge and W will include tailored-made membranes made of polymeric nanofibers produced by electrospinning and centrifugal spinning processes. Different particles and MOFs have been synthetized and their adsorption capacity towards the target CRMs has been evaluated. The most promising particles and MOFs have been incorporated into polymeric nanofibers and the adsorption and desorption capacities of these materials has been evaluated. This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 958252.

Exfoliation of MoS_2 Nanosheets Using Stimuli Responsive Poly (N-isopropylacrylamide-co-allylamine) for Multipurpose Membranes

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The idea of concurrently exfoliating and functionalizing transitional metal dichalcogenides (TMDs) nanosheets for membrane applications is appealing. Water as an exfoliating solvent for two dimensional (2D) TMDs nanosheets synthesis is both ecologically acceptable and cost effective. Exfoliating TMDs nanosheets in aqueous solution with stimuli responsive stabilizers could have a synergistic effect on both exfoliation and smart behavior manifestation. Membrane applications benefit greatly obtained from porous membranes through smart substance permeation management. For hydraulic permeability applications, we reported stimuli-response gating membrane made of MoS_2-PNIPAm-PALA nanosheets. Vacuum filtering of an exfoliated aqueous dispersion was used to prepare MoS_2-PNIPAm-PALA laminar membranes. This production process, which removes complex chemical grafting procedures, solves the water dispensability problem of stimuli-responsive functional components. With a gating ratio more than two, the membranes have positive response coefficients. The responsiveness of the MoS_2-PNIPAm-PALA membrane is dependent on the synergistic management of the copolymer's molecular chain morphology conversion and the MoS_2 sheets' constant layer spacing in response to changes in environmental signals. The use of a solar simulator as a source of both heat and light is a promising feature for improving membrane separation and antifouling ability. When the copolymer chain shrinks, the MoS_2-PNIPAm-PALA's channels open, and when the copolymer chain expands, the smart channels close. Moreover, the MoS_2-PNIPAm-PALA laminar membrane also showed photocatalytic effect, which manifests the synergistic behavior of the membrane excellent stimuli-reversible performance. In addition to selective separation systems, this unique MoS_2-PNIPAm-PALA with a simple prepared approach could be used in drug-controlled release systems as well as intelligence-controlled permeation systems.

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INNOMEM PROJECT: OPEN INNOVATION TEST BED FOR NANO-ENABLED MEMBRANES

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INNOMEM aims at developing a sustainable OITB (Open Innovation Test Bed) to foster deployment and scale-up of innovative nano-enabled membranes and their derived products. INNOMEM is an H2020 project with a total budget of 16M€ funded by the EU with around 14.7M€. It is coordinated by TECNALIA Research & Innovation and involves a consortium of 32 partners from academia, research centres, SMEs and large companies. Within the scope of INNOMEM, different types of membrane materials (polymeric, ceramic, metallic and nanocomposite), surface modification, membrane morphology and geometry and applications will be covered, providing for the first time a Single Entry Point to provide the businesses in the sector with a one-stop-shop of the best available experts and technologies. European companies, mainly SMEs, will access through the SEP to develop, test and adopt, new high performance, multifunctional, safe and environmentally friendly nano-enabled membranes in a cost-effective and sustainable way while opening-up opportunities for demonstration of innovative nanomembranes in real life industrial problems and thus accelerating the market opening for these new products. INNOMEM gathers some of the most recognised Membrane departments (>20) in Europe and acknowledged facilitators of technology transfer, corporate finance, funding and coaching, making available (i) the most promising and breakthrough manufacturing pilots and (ii) advanced characterization techniques and modelling together with (iii) non-technical services through Test Bed: while relevant improvement metrics can be defined, the potential network of reachable stakeholders counts thousands of businesses on an international scale. The main KPIs for INNOMEM: •Technical: 20% Membrane productivity improvement, 30% faster verification, >40% CO2 emissions and energy consumption reduction. • Non-Technical: 10 Showcases, >15 Democases, >100 reachable SMEs and > 300 reachable investors. As a starting point, 14 Pilot Lines and their enabled technological services, coming from past investments at Regional (RIS3) at National and European levels, are deployed.

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Molecularly Imprinted Membranes in Advanced Separations

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The prompt industrial development, the necessity of recovering and recycling raw materials as well as the increased demand of high purification level of targeted compounds amplified the awareness and the activities devoted to the production of highly selective and efficient separation systems. In this scenario, the development of membrane science and the advent of the imprinting technology assumed a significant role in the production of molecularly imprinted membranes (MIMs). They are “smart membranes” that exhibit selective recognition properties towards specific compounds of interest called “templates” and are able of separating them from a mixture containing other substances or similar compounds, including their structural homologues [1, 2]. In comparison with traditional membranes MIMs exhibit improved selectivity but preserve their stability and permeability. Their ability of achieving high separation level of the target compounds through their selective facilitated or retarded permeation as well as via their selective adsorption, has rendered them promising for the development of advanced sustainable membrane separation processes [2]. Nowadays, MIMs are applied for the selective detection and separation of a wide variety of compounds, including bioactive compounds, pharmaceuticals, optical isomers, pesticides and so on. This work summarized three examples dealing with the preparation and application of MIMs in nutraceutical and pharmaceutical fields. In particular, the application of the alternative molecular imprinting for producing phase-inversion MIMs able of selectively separating the flavonoid quercetin, the vitamin B9 (folic acid) and the xanthine theophillyne used in the treatment of the chronic obstructive pulmonary disease are presented. All the prepared imprinted membranes were competitive with their corresponding non-imprinted ones and exhibited good selectivity towards template molecules with respect competing similar compounds.

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Removal of Pharmaceutical Contaminants from Aqueous Water: Diclofenac Case Study

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Upgrading wastewater treatment with new technologies will not solely solve the problem of pharmaceuticals in water. Most of these technologies are limited by their removal efficiencies, high capital investment and operation costs, and increased energy consumption. In this study, Flat sheet Supported Liquid membrane and laboratory-scale commercial Hollow fibre membrane coupled with ozonation were evaluated for the removal of pharmaceutical Diclofenac and its degradation products. A higher initial concentration of pharmaceuticals was used (30 mg/L) compared to the one normally found in the water effluent (0.01mg/L), to evaluate the effects of several operational parameters. Cyanex 923 (Cy923) was demonstrated to be an efficient extractant for Diclofenac through a facilitated transport mechanism from the aqueous feed phase to the aqueous stripping phase. Around 95 % of Diclofenac was extracted in 4 hours with 10% Cy923 (extractant). Ozone was bubbled in the stripping phase to oxidize the extracted pharmaceutical. Two-hybrid models were investigated and the highest mineralization was obtained when combining ozonation and supported liquid membrane in a separate unit (74% of TOC removal).Finally, transport modeling of this process using artificial neural network (ANN) was conducted for the removal of diclofenac from aqueous solution.

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CO_2 Removal from a Low Partial Pressure Environment

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Removing CO_2 from low partial pressure environments is challenging. Low driving force requires strategies focused both on process development and on the development of materials with greater affinity for CO_2. Coupling gas permeation and membrane contactors could enhance the overall CO_2 removal. In order to reduce the amount of membrane area, computer simulations of the coupling revealed that it is necessary to focus on the development of gas permeation membranes that are more permeable and more selective to CO_2, seeking to overcome the boundary delimited by Robeson plot. Facilitated transport membranes have great potential for this application. Combining the high permeability and good selectivity of a PEBAX polymer matrix with the availability of free amino groups due to the presence of an ionic liquid (IL) which anion is an amine, it is intended to increase both permeability and selectivity of the gas permeation membrane. Preliminary gas solubility results showed a 200% increment in CO_2 sorption using 30% (w/w) of TEPA[NO_3] as IL. TGA and FTIR results revealed that IL was incorporated at polymer matrix. These promising results indicate great potential for application of this material as a selective layer at thin film membranes (TFM). From now on, all efforts are focused on developing extremely thin selective layers in order to guarantee high permeability, covering all possible defects to guarantee the selectivity of the material.

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Three-Phase Nanocomposite Membranes Containing Guanidine-Based Mobile Carriers for CO_2/H_2 Separation

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H_2 is widely recognized as the most promising non-carbon-based fuel and clean energy carrier. Currently, hydrogen is mainly obtained from steam reforming of hydrocarbons (e.g., natural gas and coal) followed by the water-gas shift reaction. However, a large amount of CO_2 is produced when generating H_2. Pressure swing adsorption (PSA) and amine absorption are the main H_2 purification technologies in today's industry. Membrane separation can be a promising alternative to PSA and amine absorption due to its lower energy consumption, smaller footprint, lower capital and operating costs, higher energy efficiency, operation simplicity, and high modularity. In this work, a three-phase CO_2-selective membrane based on the facilitated transport mechanism has been developed. Polyvinlyamine (PVAm), the polymer phase, has a great positive effect on the facilitated transport of CO_2 in CO_2/H_2 separation. Incorporating Polyvinyl alcohol-grafted GO (PVA-GO) nanosheets in the polymeric matrix improves the selectivity of CO_2 over H_2 as they are impermeable to small molecules, but PVA-GO nanosheets provide additional CO_2 adsorption sites thanks to the high CO_2 affinity of PVA-GO, which benefits the competitive CO_2 sorption over H_2. The main drawback of the PVAm/PVA-GO membrane is the low mobility of the CO_2 carriers as the amino groups in PVAm are fixed to the backbones, showing lower CO_2 permeance compared with membranes containing mobile carriers [1]. Therefore, selected CO_2-philic molecules were tested, and the Guanidine-based molecules were found to be promising CO_2 mobile carriers owing to their strong basicity and easiness of structural modification to tune for desired properties. Several different guanidine-based molecules with different concentration ratios in the membrane matrix was evaluated with respect to their physical, chemical, and separation properties. The optimized mobile carriers in the three-phase membranes exhibited significantly improved separation performance.

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Extraction and Recovery of Rare Earth Elements by a Polymer Inclusion Membrane

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Rare earth elements (REEs) are a group of 17 elements from the periodic table, specifically the 15 lanthanides plus scandium and yttrium. Although the REEs are relatively abundant in the Earth's crust, they are not found as native element minerals in nature. Some REEs were found to be critical in between 2015-2025 based on the importance of clean energy, and supply risk. Therefore, it is indispensable and promising to find techniques to recover and separate REEs from urban mining to contribute a circular economy. This study focuses on the use of a polymer inclusion membrane (PIM) containing the phosphonium ionic liquid IL trihexyl(tetradecyl)phosphonium bis(2,4,4-trimethylpentyl)phosphinate (Cyphos IL104) for the extraction and separation of Lanthanum, Neodymium, Samarium, and Yttrium. These metals have been chosen to be representative of light REE (La, Nd), and heavy REE (Sm, Y). In previous works, CyphsoIL104 was successfully used for the extraction of some REEs. Extraction experiments were performed using the solid-liquid extraction procedure, using a piece of a PIM made of cellulose triacetate (CTA) and the IL, in contact with solutions with metals in NaCl at different pH. Both individual and metals mixture extraction were done. Results showed that the extraction of Y was quantitative at acidic pH, but for the other metals a pH=5 was necessary. Moreover, the elution was investigated, and a HNO3 solution was the most effective among the tested stripping agents. Taking into account the different behavior on both extraction and elution process, the separation of these metals can be achieved using a simple procedure.

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Low-fouling coated PES and PVDF membranes for seafood wastewater treatment

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Seafood industries are involved in converting fish or shellfish into the products which can be sold at stores or restaurants. Vietnam occupies a leadership role on the world stage in exporting and processing these foodstuffs. Each industrial step requires high quantity of water and produces wastewater that needs to be treated. Membrane technology associated with a biological process (such as anaerobic digestion) have created a successful system in the sector of wastewater treatment named membrane bioreactors (MBR) [1]. Despite of the many advantages, membrane fouling and biofouling are the main limitations to the widespread use of this technology. A promising approach to mitigate this problem is the surface modification of existing membranes with a polymerizable bicontinuous microemulsion (PBM) [2-3]. In this work, PBM solution was obtained by following the procedure described by Galiano et al. [3]. The formulation was cast (knife thickness 50 µm) on the surface of commercial ultrafiltration polyethersulfone (PES) and polyvinylidene fluoride (PVDF) membranes to allow the polymerization of the PBM on their surface. Fouling tests were carried out by filtrating humic acid (HA) with a concentration of 100 mg/L. The results showed a better permeability and a higher HA rejection for both the PBM surface-modified membranes compared to uncoated ones. After treatment with HA, PBM membranes were washed and subjected to pure water permeability tests to assess their permeability regain after fouling. Despite the uncoated commercial membranes, PBM ones exhibited a complete regain in permeability showing a lower fouling tendency thanks to their intrinsic properties (e.g. hydrophilic surface, smoother surface) and their great potentiality in application in MBR systems for the treatment of polluted wastewaters like the ones originating from the seafood industry.

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Modification of PVDF/PSF hollow fiber membranes

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Poly(vinylidene fluoride) (PVDF)/polysulfone (PSF) blend hollow fiber (HF) membranes have shown great application potential in water treatment (e.g., microfiltration and membrane distillation) due to their aligned microfibrous structure in HF membranes. Here, we present the hydrophilic and hydrophobic modification of PVDF/PSF HF membrane to broaden its applications. To increase the hydrophilicity, we propose a strategy in terms of in-situ grown inorganic layer (including TiO2 and Al2O3). With the adhesion of the inorganic layers on the PVDF/PSF membrane surface, the contact angle decreased from 80^{o} to 50^{o}. They improved the mechanical strength from 7.7 MPa to 9.2 MPa and decreased the membrane roughness from ~50 nm to ~10 nm. The water permeance of the composite membranes can be finely tuned from 50 to 500 Lm-2h-1bar-1. In addition, the resulting membranes simultaneously enhanced the separation performance and anti-fouling performance characterized by bovine serum albumin (BSA) rejection behavior and flux recovery rate (FRR), respectively. The best BSA rejection and FRR of the PVDF/PSF-inorganic composite membranes reached 95% and 72.2%, respectively. In terms of increasing hydrophobicity, we present the strategies based on chemical modification (1H,1H,2H,2H-Perfluorooctyltriethoxysilane grafting) and physical modification (Hyflon coating). The contact angle could be increased from 80 to 120o via physical modification and to 140o via chemical modification. In the separation of saline water (35 g/L salts and 0.1 mM sodium dodecyl sulfate), modified membranes showed higher water flux and rejection than the pristine membrane. Their water flux increased from ~4 kgm-2h-1 (40 hr) to ~6 kgm-2h-1 (120 hr, chemical modification) and ~5 kgm-2h-1 (120 hr, physical modification). The modified membrane showed greater than 99.99% rejection and outstanding stability even after operation for 120 hr.

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In-situ hydrophilization of hollow fiber membranes using electron beam grafting

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Membrane technology is an energy-efficient method that has a wide range of applications in various industries (e.g. purification of wastewater, seawater desalination, hemodialysis). A major problem in the application of membranes is the so-called fouling. Fouling is an undesired adsorption, deposition, or precipitation of substances (organic and inorganic substances, biomolecules, and microorganisms) on the surface or inside the membrane structure [1]. A large part of the current research on membranes for aqueous applications is directed at additional hydrophilization of the membrane material to prevent hydrophobic interactions between foulants and the membrane. Improved water wettability leads both to an increase in membrane performance and to a reduction in membrane fouling. To reach that goal, a method was developed which allows the in-situ immobilization of small molecules or hydrophilic polymers (e.g. pore forming agents like PVP) on a PES membrane directly after the hollow fiber spinning via nonsolvent-induced phase separation (NIPS) in a roll-to-roll process. To modify the membrane the method of electron beam-based grafting [2] has been used. Due to the electron beam irradiation, radicals are formed at the membrane polymer and the modification reagents, respectively. These radicals then recombine and form covalent bonds [3] . Electron beam-based grafting is an efficient modification method which forms stable bonds between modification reagents and membrane polymer through the entire cross section of the material. In this study, the immobilization of PVP molecules in the membrane structure has been validated by IR spectroscopy investigations of the hollow fibers. In comparison to an unmodified membrane the PVP/PES peak-area-ratio was up to 20 percent higher for the modified membranes.

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Effect of amine modification on CO_2 separation performance of commercial silica membranes

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The latest Intergovernmental Panel on Climate Change (IPCC) report warns of the "irreversible" impacts of global warming and ultimately stresses the urgency of taking action to cut greenhouse gases. The development and integration of efficient carbon capture, utilization and storage technologies (CCUS) is crucial to address the existing energy crisis while complying with the new environmental guidelines. Membrane separation systems have been considered as potential, low cost, green alternatives to conventional CO_2 capture technologies. Microporous silica membranes are among the most studied inorganic membranes for gas separation applications. Although they have been already commercialized for small–scale pervaporation applications, the main challenge – to reproducibly synthesize large area, defect free membranes – still remains. In this direction, the scope of this work is to develop CO_2 selective membranes starting from commercial silica membranes and implementing tailor-made modification protocols that involve, in situ chemical vapor deposition (CVD), and atomic layer deposition (ALD). The modification and surface functionalization procedures aim for precise pore size control and surface chemistry adjustment. More specifically, commercial silica membranes are modified using primary, secondary and tertiary amino silanes to simultaneously narrow the membrane pores and enhance silica's affinity for CO_2. The effectiveness of the proposed procedure is assessed by single gas permeation, relative permeability, and CO_2/N_2 separation tests before and after modification. In addition, Fourier transform infrared spectroscopy (FTIR) is employed to confirm the superficial attachment of amine groups. Finally, the effects of the main design operating parameters (temperature, pressure ratio, modification time, etc.) on the membrane's separation performance are also investigated. Ultimately, high performance membranes have been successfully developed, demonstrating sufficiently enhanced CO_2/N_2 separation properties, that are attributed to amine–assisted CO_2 adsorption and surface diffusion encountered at relatively low temperatures, and by imposing vacuum (P ~ 3·10-4 bar) on the permeate side.

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Novel antifouling surface grafted membranes for wastewater treatment

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The declining water flux over time due to fouling represents the main onstream issue of the existent membranes in wastewater treatment. Thus, preventing and mitigating membrane fouling is considered the key challenge to improving performance and extending the use of membranes in water treatment and recovery applications. Novel membranes with improved properties will lead to reduced energy consumption, reduced cleaning and consumption of chemicals, as well as the less frequent replacement of membranes. In the current investigation we developed methods for antifouling and anti-scaling functionalizing of commercial ultrafiltration (UF) and microfiltration (MF) membranes. The manufacturing technology aims to obtain membranes with improved water flux, stability, antifouling properties exceeding the performance of commercial membranes. The membranes are aimed to treat the intake water in fish farms and wastewater in general. We aim for easily scalable methods for large production, such as direct surface modification inside existent commercial membrane modules. The fabrication process consists of UV grafting of polymer chains from acrylate type of monomers (acrylic acid, methacrylic acid, hydroxyethyl methacrylate) onto the surface of microfiltration membranes (PVDF and PES). The successful membrane hydrophilization was characterized by FTIR, wettability (contact angle measurements) and water permeability. The membranes shows a decrease in the contact angle from 85 to 70 for PVDF and from 61 to 44 for PES indicating hydrophilization of the membrane which was confirmed by FTIR as well. The clean water flux was measured in cross-flow configuration at room temperature, various water flow rates and two applied pressures. The water flux was reduced due to the surface grafting but the membrane bulk porosity was little affected by grafting. We investigated various fabrication strategies to prevent and reduce the clogging of the membranes pores during grafting. The porosity of the membrane prior an post grafting was investigated using mercury intrusion porosimetry.

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Systematic development of a low fouling modification for micro and ultrafilter polyethersulfone membranes

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In the biopharmaceutical industry downstream processing is essential to obtain a biopharmaceutical target molecule or protein of sufficient purity. Hereby filtration is crucial in different process steps to achieve this goal. Commonly used membranes are therefore polyethersulfone (PES) based due to their high chemical stability and their inertness. However, a major disadvantage of the material is its hydrophobic character which increases protein fouling on the membrane surface during filtration and thus reduces membrane performance. Effective approaches to overcome this obstacle are based on the chemical modification of the membrane surface or the physical entanglement of other polymers within the PES main structure to introduce hydrophilic groups. For this purpose, several strategies, such as blending of PES with hydrophilic polymers or grafting/crosslinking of hydrophilic building blocks onto the membrane surface by radiation- or heat-induced polymerization may be performed. For the grafting approach, the type of monomer used, the concentration and the ratio of monomer to crosslinker play a critical role in the success of this kind of modification. The blending of the PES base material to entangle hydrophilic polymers within the membrane matrix is influenced by the size and chemical composition of the blending polymer. In this work, both modification strategies using different hydrophilic monomers, crosslinkers and polymers are investigated to efficiently reduce protein fouling on PES micro- and ultrafilter membranes. Furthermore, all approaches will be evaluated with respect to their sustainability, economic viability and their feasibility of upscaling. The most promising candidates in terms of capacity and unspecific protein binding will be tested for an implementation within existing flat sheet and hollow fiber membrane production process.

Waltzing around the stereochemistry of membrane crosslinkers for precise molecular sieving in organic solvents

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Crosslinking of polymeric membranes induces changes in both membrane stability and separation performance. Numerous membrane-crosslinking methods have been developed with the objective of obtaining improved membranes. However, none of these methods systemically investigated the stereochemical effects of the crosslinker in the pursuit of better stability and performance. Herein, we address this knowledge gap by presenting a systematic investigation of the stereochemistry of crosslinkers. The intrinsically microporous poly(ether-ether-ketone)-containing Tröger's base (iPEEK-TB) was synthesized and employed in the fabrication of organic solvent nanofiltration (OSN) membranes. Crosslinkers were carefully selected based on the stereochemical position of the two benzyl bromide functional groups, separated by distances of 4.3, 8.2, 8.5, and 12.4 Å and significant effects arising from crosslinking on membrane physical properties, morphology, and OSN performance were investigated. Crosslinked membranes showed excellent solvent resistance, mechanical flexibility, and thermal stability. As a function of crosslinking distance, the molecular weight cutoff (MWCO) values of the membranes varied in the range of 575–750 gmol^{-1}. The para isomer of the crosslinkers resulted in higher permeance relative to membranes crosslinked with their counterpart ortho isomers, and vice versa, the ortho substitution resulted in higher solute rejection values compared with para isomers. An increase of 50% and 12% in acetonitrile permeance relative to the annealed benchmark membrane was observed upon the treatment using iPEEK-TB with 4,4′-bis(bromomethyl)biphenyl(p-BBMBP) and 2,2′-bis(bromomethyl)-1,1′-biphenyl (o-BBMBP), respectively, whereas a permeance decrease of approximately 23% and 32% was noted upon treatment with α,α′-Dibromo-p-xylene (p-DBX) and α,α′-Dibromo-o-xylene (o-DBX), respectively. The corresponding MWCO changes were found to decrease for all crosslinked membranes within the range of 12%–40%. The crosslinked membranes demonstrated stable performance in polar aprotic solvents. This work demonstrates the importance of crosslinker selection for OSN membrane performance and solvent resistivity and opens new avenues for fine-tuning membrane stability and OSN performance

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Potential of PVDF-chitosan hybrids in water purification contaminated by pharmaceuticals – paracetamol

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Among many emerging water pollutants, drugs are the most crucial category, including antibiotics, analgesics, steroids, antidepressants, antimicrobials, analgesics, hormones, anti-inflammatory drugs, β-blockers. The persistence and potentially toxic effects of these drug residues make them a potential long-term threat to discharged water. So far, various treatment technologies have been studied to eliminate drug residues in wastewater, but the removal efficiency is not always satisfactory. Based on the high rejection rate of non-volatile compounds and low operation temperature, MD might be an option for the pharmaceutical wastewater treatment. In the work, PVDF-chitosan (CS) hybrid membranes, generated via covalent bonding modification, were used in air-gap membrane distillation (AGMD) to purify water containing paracetamol. The detailed studies of morphology, wettability, and physiochemistry characterization were performed. The material analyses were used to discuss differences in the transport and separation features of the membranes. The attachment of CS improved the membrane performance. The values of paracetamol rejection coefficient (Rpar) for hybrid PVDF-CS membranes were close to 100%. However, for the pristine PVDF, Rpar was equal to 90%. Moreover, the chitosan introduction, enhanced transport by 25%. Considering that modification was performed by stable covalent bonding, materials possessed very good stability and maintained their properties after many cycles of the AGMD process.

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FABRICATION OF FUNCTIONALIZED HOLLOW FIBER MEMBRANES BY E-BEAM IRRADIATION

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Often, hollow fiber nanofiltration membranes are created by off-line coating of a support with a (layer-by-layer) nanocoating to meet the specifications for nanofiltration applications. This means that the surface modification is carried out batch-wise in one or several post-treatment steps. This method is often accompanied with significant handling, which is a potential source for damages or defects. The EU-project INNOMEM develops and organizes a sustainable open-innovation-test bed (OITB) for innovative scale-up of nano-enabled membrane products. Potential clients can access the services of service providers through a single-entry-point (SEP) to develop, test and adopt, novel high-performance membranes to their specific needs. Within the project 14 pilot lines are upgraded/upscaled and 10 showcases carried out to validate the OITB-concept. After validation with the showcase, 2 periods of open calls will give external companies the opportunity to carry out a research question or small project. Within the scope of the INNOMEM project EMI upgraded the hollow fiber pilot line with an integrated, in-line, surface modification technology by means of electro-beam. We aim to develop a next generation membrane with surface modification, so the process will be more efficient, stable and less time-consuming. Treatment by E-beam can improve the stability of the membranes and potentially enhance the selectivity of the separation layer(s). The penetration depth of the e-beam allows for modifications within the porous structure or at the inner side of the hollow fibers instead of only at the visible surface. We will present the results obtained in the showcase in collaboration with NX Filtration, the showcase owner. The pilot line is equipped with a winder to allow fabrication of larger bundles to allow making full scale modules. With in-line dope preparation (mixing, filtering and degassing) and precise control of the area around the airgap/chimney we guarantee consistent quality of the fabrication process.

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Fine-tuning of carbon nanostructures/alginate nanofiltration performance: Towards electrically-conductive and self-cleaning properties

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Electrically-conductive membranes became the center of attention owing to their enhanced ion selectivity and self-cleaning properties. Carbon nanostructures (CNS) attain high electrical conductivity, and fast water transport. Herein, we adopt a water-based, simple method to entrap CNS within Alginate network to fabricate self-cleaning nanofiltration membranes. CNS are embedded into membranes to improve the swelling/shrinkage resistivity, and to achieve electrical-conductivity. The CaAlg PEG-formed pores are tuned by organic-inorganic network via silane crosslinking. Flux/rejection profiles of Na2SO4 are studied/optimized in reference to fabrication parameters. 90% Na2SO4 rejection (7 LMH) is achieved for silane-CaAlg200-10% CNS membranes. Membranes exhibit outstanding electrical conductivity (~2858 S m−1), which is attractive for fouling control. CaAlg/CNS membranes are tested to treat dye/saline water via two-stage filtration, namely, dye/salt separation and desalination. A successful dye/salt separation is achieved at the first stage with a rejection of 100%-RB and only 3.1% Na2SO4, and 54% Na2SO4 rejection in the second stage.

Modelling protein fractionation with Ultrafiltration

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Membrane processes has been used for fractionation of liquid streams with relatively low energy and resource consumption under mild processing conditions. There is increasing interest on protein fractionation with membrane processes due to its high throughput and scalability. Therefore, quantification of protein transport through an ultrafiltration (UF) membrane is essential for designing a fractionation process. Some studies demonstrated that solute transport of charged particles is influenced by solute to pore size ratio and electrostatic interactions. However, studies on charged particles transport through a membrane were mostly performed for nanofiltration. Studies on protein UF mostly focused on describing the flux on a membrane that is fully retentive to the protein or the studies were limited to lab scale experiments. The protein transport description is also complicated by the fact that proteins may have different properties (charge, hydrodynamic radius, degree of polymerization) at different pH and ionic strength. This study has been done with an open UF in a pilot scale configuration. Experiments started with a single protein in an electrolyte solution. Next to that a more complex whey protein isolates mixture has been measured. Solute(s) transport has been modelled by using Maxwell-Stefan equations, membrane partitioning, and Nernst-Planck equations. These equations are used to mathematically describe the solute transport in the system by including interactions between solutes and the membrane both physically and electrostatically. These modelling activities are an essential tool for characterizing the membrane and understanding the phenomena during membrane filtration. Therefore, an effective and sustainable fractionation processes for protein can be designed.

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Advanced methods for the analysis of mixed gas diffusion in polymeric membranes

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Growing concern about climate change has spurred the research on novel high-performance gas separation membranes for the production of clean and renewable energy (hydrogen, biomethane) and for the capture of greenhouse gases like CO_2. Their basic transport parameters, permeability, diffusivity and solubility, are usually determined by the well-known time-lag method in a fixed-volume instrument. This relies on the assumption that the transport in dense polymeric membranes is governed by the solution-diffusion mechanism and can be described by Fick's laws. While this is true for materials such as polydimethylsiloxane, we will show that this method may be unsatisfactory for describing the gas transport in complex high free volume polymers and polymers of intrinsic microporosity (PIMs), or the transport of gas mixtures. For the detailed analysis of these novel materials, more sophisticated test methods are needed. This paper will present a number of alternative methods, using a variable volume setup equipped with a mass-spectrometric residual gas analyser for the continuous online analysis of the permeate gas composition [1]. The time-lag calculated from the total amount of permeate as a function of time will be compared with other singular points, such as the inflection point after a step change in the gas composition (differential method), or the peak position and peak maximum after a short pulse (pulse method) [2]. The paper will discuss the strengths and limitations of these methods, showing furthermore how the fit of the entire permeation curve reveals deviations from standard Fickian behavior for materials like PIMs, and how it allows to shorten the analysis time for common rubbery materials with Fickian behavior. Most importantly, the continuous analysis of the permeate composition allows the analysis of the diffusion coefficient of the individual gas species in a mixture, data which are not accessible via the standard time-lag method.

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Easy tool for prediction of separation performance

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This paper proposes a tool for analyzing the performance of a membrane separator. It is based on mass balance and consists of 1D 1st order ordinary differential equations, which were resolved using Wolfram-Mathematica. This tool gives, e.g., the permeate concentration of the permeating species as a function of the related recovery for any membrane length or area, in addition to concentration and pressure profiles of any species, permeating driving force, etc. It provides an immediate comparison of the performance of membrane modules of different length opening, at the same time, to the desired choice in term of desired specification. The results provided various scenarios depending on the membrane properties (selectivity and permeance), operating conditions (feed/permeate pressures ratio) and feed flow rate and compositions. For instance, a membrane with selectivity 20, for a target recovery of 70%, increasing the pressure ratio from 5 to 30, gives concentration values of the permeating species from 0.75 to less than 0.9. Instead, with a target recovery of 70%, pressure ratio 10 and selectivity 40, we can obtain concentration values of the permeating species greater than 0.9. Therefore, with a less selective membrane, to obtain the desired specification, we have to increase the compression load, while with more selective membranes we can operate at lower pressure ratio; a higher pressure ratio requires a greater energy and, hence, economic outlay. Furthermore, we can establish a lower limit of pressure ratio where the system does not allow desired performance and an upper limit where we obtain a maximum value of permeate concentration. In this way we can identify an operating condition, e.g., membrane properties and pressure ratio, for meeting the desired system performance.

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Osmotic Membrane Distillation Crystallization of $NaHCO_3$

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Osmotic membrane distillation crystallization (OMDC) is an innovative technique in which two liquids are brought into contact through a non-selective hydrophobic microporous membrane. Because the concentration is not the same on both sides, it induces water activity difference and leads to evaporation of water from the feed to the osmotic side. OMDC has advantages over conventional distillation and crystallization processes. This technique shows a very high specific contact area promoting higher mass transfer with more compact equipment than in conventional crystallization or distillation. The main advantage of OMDC is lower energy consumption OMDC of sodium bicarbonate ($NaHCO_3$) was studied, proposing the use of osmotic membrane distillation crystallization. Crystallization takes place due to the saturation of the feed solution after water evaporation on the feed side, permeating through the membrane pores to the osmotic side. The process operational parameters, i.e., feed, and osmotic velocities, feed concentration, and temperature were studied to determine the optimal operating conditions. Regarding the feed and osmotic velocities, values of 0.038 and 0.0101 m/s, respectively, showed the highest transmembrane flux, i.e., 4.4×10^{-8} $m^3/(m^2 \cdot s)$. Moreover, the study of the temperature variation illustrated that higher temperatures have a positive effect on the size and purity of the obtained crystals. The purity of the crystals obtained varied from 96.4 to 100% In addition, the flux changed from 2×10^{-8} to 7×10^{-8} $m^3/(m^2 \cdot s)$ with an increase in temperature from 15 to 40 °C. However, due to heat exchange between the feed and the osmotic solutions, the energy loss in osmotic membrane distillation crystallization is higher at higher temperatures. The process presented here is registered under the patent WO 2022/117800 A1

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Effect of water content on the properties of the polymer electrolyte membranes

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The polymer electrolyte membrane for fuel cells transports hydronium ions through the water channel formed inside the membrane [1]. Therefore, the hydronium ion transport efficiency varies depending on how effectively the water channel forms a wider and more connected morphology without clogged channels, ultimately determining the fuel cell's overall performance [2]. Accordingly, various studies have been conducted to analyze the formation of water channels in the polymer electrolyte membrane. For this purpose, analysis techniques such as SAXS and TEM are widely used [3]. However, in these analysis methods, although it is possible to analyze the approximate channel size and distribution of hydrophilic functional groups, it is difficult to elucidate the shape of the actual water channel accurately. In this study, the molecular dynamics simulation technique was used to analyze these water channels and finally observe the morphology of the water channel inside the membranes and describe how it can change the polymer electrolyte membrane performances. For this purpose, a hydrocarbon-based polymer electrolyte membrane was selected, which is known to have relatively poor performance under the low-humidity conditions compared to the perfluorinated polymer electrolyte membranes. Their water channel morphologies were analyzed under various water content conditions, and finally, the morphology trend was compared to the hydronium ion transport performances.

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Molecular dynamics simulation of alkaline ionomer under excessive water condition for water electrolysis

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Although extracting hydrogen from natural gas is inexpensive, it generates carbon dioxide and causes environmental problems such as the greenhouse effect. For this reason, many countries are conducting a lot of research to produce 'green hydrogen' that does not emit carbon dioxide during the hydrogen production process [1]. The water electrolysis system is a technology that makes hydrogen by using water electrolysis. It is attracting attention as a next-generation hydrogen energy production method due to the advantage of no greenhouse gas emissions from the production stage. Among the water electrolysis methods, alkaline water electrolysis uses an alkaline electrolyte as the electrolyte. When the voltage and current are applied, OH- ions are transferred, an oxidation reaction occurs at the anode to generate oxygen, and hydrogen is generated through a reduction reaction at the cathode. Since the water electrolysis system operates under a condition in which water is continuously supplied, it is essential to maintain the structural stability of the ionomer under extreme water conditions and minimize the degradation of gas permeation and ion conduction performance [2]. This study investigated the alkaline ionomer's structural stability by increasing the alkaline ionomer's water uptake to 0%, 50%, 100%, 200%, and 400% using molecular dynamics simulation and analyzed the change in gas permeation performance and gas permeation behavior. Finally, it intended to present the direction of designing the molecular structure of the ionomer by observing the difference in the ion conductivity performance of the alkali ionomer.

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Advanced control of Reverse Osmosis with a data-driven fouling model

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Reverse osmosis (RO) has been widely applied and recognized as the leading technology of desalination. However, its performance is challenged by membrane fouling which increases the cost of operation due to expensive pretreatments, higher operating pressures due to flux decline and frequent chemical cleanings which can damage the membranes and hasten membrane replacement. A good operational strategy is key to avoid this. Nowadays, operators manually change the recovery setting, in order to prevent too much fouling from building up. This is often done only a few times a year during the seasonal transitions. In this study, an advanced control strategy is proposed, to automatically optimize the recovery setting based on the conditions of the incoming feed. The advanced control strategy consist of model predictive control (MPC) in which an objective function is minimized to find an optimal setting for the recovery by predicting the fouling risk of the system in real-time. The objective function is constructed in a way that fouling is minimized and recovery is maximized. The MPC uses a process model in an online manner to predict fouling (expressed as membrane resistance) under the prevailing process conditions. Since fouling is a complex phenomenon that covers a wide range of mechanisms and respective foulings, a data-driven recurrent neural networks (RNN) model was chosen to build the process model. To illustrate the operation of the controller, several step responses in the feed conditions are simulated and the response of the controller is studied. For example, a drop in temperature leads to a higher membrane resistance. The process model predicts this behavior, leading to the MPC controller applying a lower recovery to prevent the membrane resistance from becoming too high. In this way, a better operation of the RO installation can be applied and the negative effects of fouling are minimized.

Multi-ion physical modeling of nanofiltration/low-pressure reverse osmosis with and without ozonation pretreatment

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Since the last decades, an increase of numerous substances including organic matter and emerging micropollutants (MPs) which may affect human and animal health has been observed in aquatic environments 1. Conventional treatments of this increasing organic matter, for drinking water production, require increasing doses of coagulation and flocculation chemicals and generate increasing sludge production. Their limited effectiveness may increase the residual organic matter in the treated water and the risk of formation of disinfection by-products. Nanofiltration (NF) or low-pressure reverse osmosis (LPRO), integrated in more sustainable treatment lines, are proven to improve the treated water quality and contribute to retain MPs, while limiting sludge production and chemical consumption. Nevertheless, organic fouling is one of the main issues related with membrane processes. The potential benefits of pre-ozonation upstream membrane processes are investigated in the context of various drinking water production treatment lines. Thus, firstly, the transport properties of NF membranes are studied by means of the SEDE (steric, electric, and dielectric exclusion) homogeneous model. Within this 1D-model, the separation of solutes is considered as resulting from transport effects (described using extended Nernst-Planck equations) and interfacial phenomena, including steric hindrance, the Donnan effect, and dielectric exclusion in terms of both Born dielectric effect and image force contribution 2. Secondly, a pre-ozonation upstream of a NF membrane was investigated, as part of the development of a new treatment line of water loaded with natural organic matter, to potentially reduce the organic fouling and consequently to increase the permeate flow rate and/or to limit the concentrate volume 3 and to potentially improve the removal efficiency of MPs of interest, targeting modifying their ionic properties, without degrading the organic matter or MPs into by-products likely to degrade the quality of the filtered water. Meanwhile, based on the results obtained in the pre-ozonation study, the SEDE model is evaluated to verify the description ability for the complex water matrix containing micropollutants. It is noticed that the pre-ozonation has a minor impact on studied MPs rejection by the NF membrane. However, it is found that a relatively low specific ozone dose could effectively reduce the hydrophobicity of organic matter according to the fluorescence results which could potentially be a useful indicator for studying the impact of pre-ozonation on fouling mitigation. With respect to modeling, it is shown that the SEDE model can provide trends, however with significant differences between the MPs rejections simulated by the model and the rejection obtained experimentally. Therefore, improvement of the model should be considered for predictive purposes. Moreover, the same methodology will be conducted with the LPRO membrane to compare with the results previously mentioned of NF membrane regarding the objectives.

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Food & Beverage Concentration Process Design and Optimization Using Aquaporin Forward Osmosis Membranes

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Forward osmosis (FO), a form of an osmotic membrane, has gained popularity over the past few years [1]. FO membranes have demonstrated already huge potential in water/wastewater treatment however the food and beverage sector can also benefit from the advantages this technology can offer compared to the conventional methods for up-concentrating food streams like juice, coffee, beer, etc. [2]. This is because FO is highly effective and precise in separating water from other components and preserves the essential nutrients and aromas in streams because of operation in room conditions. In this study, various simulations are carried out in order to develop and expand a model for food and beverage concentration applications with Aquaporin membranes, focusing on apple juice concentration as a case study to screen the effects of process variables such as feed and draw solutions concentration as well as the membranes configuration and operating mode. Promising results in terms of water flux, recovery rate, and final feed concentration were acquired which were in good agreement with the experimental results. Furthermore, the operation of a pilot that combines FO and DS recovery via evaporation was simulated. Results confirm the feasibility of sustainably maintaining the FO process by regenerating the draw solution from 20% w/w to 60% w/w using a vacuum heat pump-based evaporator with a nominal capacity of 750 l/day. The development of a predictive model to calculate water flux is the final part of this study, within which the value of expected water flux in FO process versus a range of concentration of different feed and draw solutions is estimated. This tool assists engineers at Aquaporin in having a clear estimation of the performance of the FO membranes facing different FSs before starting any experimental campaigns which save time and resources for the company.

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Modelling of CO_2 Adsorption in PIM-1 Membranes using a Multiscale approach combining Monte Carlo technique, Molecular Dynamics and Reactive Vacancy Solution Theory

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Polymer of Intrinsic Microporosity (PIM-1) membranes have been studied for their peculiar morphological properties of having microporous clusters within a dense structure. The upper-bound performance of PIMs is attributed to high gas solubility, allowing an increased permeability without reducing the selectivity [1]. One of the most crucial phenomena caused by CO_2 is the swelling of the polymeric chains caused by the strong polymer-gas interactions. The influence of swelling by CO_2 in PIM-1 membranes has not been satisfactorily modelled in the open literature, which represents a gap of knowledge indicating that some aspects about it are still unclear [2]. To fill this gap, this work aims at modelling the adsorption of light gases in PIM-1 membranes by a multiscale approach, which consists of two steps: 1) Description of the polymer-gas system at a molecular level by a combined approach based on Monte Carlo technique and molecular dynamics; 2) Evaluation of adsorption macroscopic parameters from the molecular-level analysis using the Reactive Vacancy Solution Theory (RVST) coupled with the Wilson activity model for the adsorbate. As for the former step, the adsorption isotherms are obtained for CH_4, CO_2, CO using the Configurational-Bias Monte Carlo (CBMC) approach on polymeric boxes built by molecular dynamics, considering both single-gas and binary-mixture systems. As for the latter, the evaluation of the adsorption parameters is carried out in Matlab® environment through a minimisation procedure consisting in a non-linear constrained multivariate regression, from which a number of information pieces are obtained, like the extent of the polymer-species and species-species interactions, the interaction energies, saturation loadings, the heat of adsorption and others. The proposed multiscale approach represents a novelty in the open literature and allows us to describe and design more precisely adsorption-based separation and/or capture systems in the presence of swelling.

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Ion diffusion in membranes: a multiscale modelling starting from molecular scale

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Multiscale models are highly essential to interpret various complex phenomena such as ions transport through membranes. Herein, we present a multiscale procedure describing the diffusion of Cl^- and Na^+ through a polysulfone-tetramethylammonium-based anionic exchange membrane demonstrating the predictive ability of this procedure which illuminates different aspects of the complex that cannot be obtained by single-scale modelling. The Cl^- and Na^+ transport is modelled with particular attention to the co-ion diffusion that represents a tricky issue in (Reverse) Electrodialysis when the concentration of ions in seawater or brines is used. Density Functional Theory calculations were merged with Molecular Dynamics (MD) simulations to present a highly accurate description of the diffusion as a function of the membrane ionic exchange capacity. The diffusion coefficients were calculated using a mean square displacement approach and two macroscopic analytical models in which the characterizing parameters were evaluated on a smaller scale, allowing to establish a direct link between the nano-metric scale (i.e., the MD simulation cell) and a larger one. To simulate real conditions (i.e., non-ideal membrane selectivity), counter-ion and co-ion concentrations inside the membrane were determined using a modified Donnan-Manning model. Finally, for membranes with ideal selectivity, the Cl^- diffusion coefficients resulted in a favourable agreement with literature values, while, for membranes in real conditions, the computed values are in qualitative agreement with the experimental trends [1]. Furthermore, membrane-based ion separation requires monovalent cations selectivity. In order to provide fundamental information on membrane selectivity, the ion confinement effect in nanopores is also presented in this research. To achieve this purpose, carbon nanotubes with internal diameter fine-tuned to the first ion hydration shell [2] were chosen to perform in-silico experiments of single-cation permeation through the nanotubes by MD to unveil the correlation between ion rejection and CNT inlet sizes.

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Characterisation of fouling behaviour during liposome sterile filtration using CFD modelling

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Purification of biotechnology products is increasingly vital for developing nanoparticle-based therapeutics [1]. Liposome, consisting of a phospholipid bilayer with hydrophilic head and lipophilic tail, is flexible to encapsulate targeted drugs within its spherical structure. A sterilizing-grade filter is introduced to remove bioburden before the final Fill and Finish step [2]. However, liposomes present unique challenges not encountered with conventional therapeutics such as monoclonal antibodies [3-6]. The objective of this study is to elucidate the fouling behaviour via computational fluid dynamics (CFD) and improve liposome transmission performance through optimized operating conditions. Liposome filtration is conducted in normal flow filtration using a stainless-steel filter holder. The size of liposomes used is about 147 nm, and the pore size of the dual-layer membrane is 650 nm nominal for asymmetric upstream and 200 nm for symmetric downstream layers, respectively. To reduce the consumption of costly liposomes, we apply COMSOL to simulate fouling processes and predict liposome transmission performance. Based on Navier-Stocks and continuity equations, the profiles of hydrodynamic and local concentration are simulated time-dependently with constant pressure or constant flux. 3D-imaging technology is applied to visualize the position of aggregated liposomes. It was found that for this dual-layer membrane, liposome fouling in the upstream layer is close to the upstream surface rather than the downstream surface, which is unexpected based on the liposome size. Concentration of liposomes at the upstream surface was greater than that at the downstream surface in the downstream symmetric layer as well. Based on preliminary experimental results and model stimulation, this study provides insight into multiple fouling phenomena relating to liposome sterile filtration which enhances fundamental understanding, in turn enabling greater process efficiency for valuable biological products.

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Effect of ion pairing on ion transfer through sub-nanometer CNTs

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Narrow carbon CNTs are attractive mimics of biological water channels with high transport rates and significant water-ion selectivity. While there is an overall consensus regarding water transport in narrow CNTs, the physical mechanisms behind ion rejection still pose many questions. Experimental results and simulations suggest ion selectivity and transport are largely controlled and well explained by specific adsorption of OH- ions. CNT affinity towards these ions greatly exceeds that of salt ions such as Cl-, which effectively leads to a composition-dependent negative charge of the CNT. However, *ab initio* simulations indicate that transfer of these species as free ions show no preference for OH-. To explain the observations, we hypothesize that uptake of OH- ions in the form of ion pairs may attenuate the repulsion between the anion and CNT electron cloud and let ion-specific effects come into play. To test this hypothesis, we performed ab initio simulations of relevant ion pair form water to CNT, in which we varied the ions, cation-anion separation distance within CNT, and chirality of the nanotube. The preliminary results indicate that ion pairing significantly reduces the cost of ion transfer, and leads a strong preference to OH- over Cl-. With increase of ion-ion distance, transfer energy drastically increases up to limit of free ion transfer. Obtained results well rationalize the experimental observations. The study is ongoing and currently focuses on extending computation to more ions for better understanding of ion-specific effects. The updated results will be reported at the meeting.

Establishing structure-property linkages for wicking time predictions in porous polymeric membranes by data processing workflows

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The demand for rapid and easy-to-use medical diagnostic devices, referred to as Point-of-Care-Testing (POCT), has increased over the last years. Especially for lateral flow assays (LFAs) the advantages of POCT-devices are evident due to their user-friendliness and cost-efficiency. The working principle of LFAs is based on the capillary driven liquid transport of a fluid through the polymeric highly porous and open-pored membranes, commonly known as wicking, to a reaction zone. Although LFAs such as the currently widespread COVID-19 rapid tests, are produced and used on a large scale, the interaction mechanisms of the liquid transport and the microstructural characteristics in the fluid conducting porous membrane are still not fully understood. To improve the test design, it is necessary to link the microstructural properties at the pore-scale (µm-scale) with the macroscopic wetting behavior (cm-scale). To achieve reliable correlations, a data base with a large number of microstructures is created using a powerful generation algorithm implemented in the Pace3D [1] simulation framework. Analysis algorithms and single-phase flow simulations are applied to each structure for feature extraction [2]. By coupling the obtained microstructural properties such as porosity, ligament radius, pore radius and permeability with a macroscopic wicking model [2] and the well known Kozeny-Carman equation, a structure-property linkage between structural characteristics and the wicking time prediction is established. The processing workflow, including the generation and analysis of a total of about 400 porous membrane microstructures, is realized with the workflow editor KadiStudio, while the data handling in general is orchestrated by the research data infrastructure Kadi4Mat (https://kadi.iam-cms.kit.edu/) [3].

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Ion Transport Mechanisms in Polymers and Membranes

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Membranes are a mature field and the performance of current membranes is often good. Nevertheless, we always seek for still better performance and novel, currently unavailable, types of membranes, which requires new research and unorthodox approaches. We are working on a few such projects, such as development of biomimetic membranes based on aquaporins (water channel membrane proteins used by all living organisms), novel nanofiltration membranes that have a uniquely high permeability to salts, and membranes with improved proton conductivity for fuel cells. Over the past few decades, the commercial success of the membrane desalination has stood in stark contrast with a much slower progress in understanding and modelling of ion transport. The growing modelling "literacy" and constantly improving toolbox, especially, MD and ab initio simulations, have increased interest in modelling as a valuable tool in membrane research. Unfortunately, "engineering" mean-field models, preferred in membrane practice and development, are still unable to predict performance without extensive experimentation and model fitting. We believe that, rather than inability to estimate parameters, the difficulty reflects gaps in understanding ion transport in polymers and membranes. We propose to use focus on DR spectroscopy on the experimental side, as elaborated below, and employ simulations (MD, DFT, and properly amended "classical" models) to progress towards these goals. Answering above questions, properly amending current models, and developing methodology for evaluating relevant parameters may supply the critical missing link towards truly predictive modelling of membrane performance for many applications. This would not only be an important contribution to engineering using membranes, but could also help develop new separation materials and processes for water and energy sectors.

Noncovalent interactions between SARS-CoV-2 spike and membrane polymer surfaces

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The recent pandemic triggered numerous societal efforts to control and limit the spread of SARS-CoV-2. How the virion interacts with inanimate surfaces, such as polymeric membrane surfaces, is a critical challenge discussed in this research providing useful information to design rational materials. The long-range interactions between spike protein and surfaces, before a possible virion adsorption, are correlated to the membrane surface's affinity towards SARS-CoV-2. Herein, the interaction potentials for a single protein and the global potentials, referred to the interactions of multiple proteins with the surface, are calculated for the closed and open states. Molecular Dynamics simulations are performed to model the surface of three polymeric materials used in the preparation of membranes: Polypropylene (PP), Polyethylene Terephthalate (PET), and Polylactic Acid (PLA), subsequently employed in Molecular Mechanics calculations to obtain the interaction potentials. The single-protein potential energies demonstrate for the considered polymeric materials a similar trend regarding the protein-surface distance, highlighting a greater affinity towards the spikes of PP and PLA compared to PET. Worth noting that for the closed and open structures, the single-protein potentials decrease in the following order PP ~ PLA > PET and PLA > PP > PET, respectively [1]. Further, a similar pattern was found for the global interaction potentials, consequently, PLA and PP interact with the virion to a greater extent compared to PET, although the differences among the target surfaces are small. The global potentials exhibit that the protein-surface long-range interactions are weaker than covalent or ionic binding energy. However, the values close to the surfaces are sufficient to trigger effective adsorption of the spikes, and therefore a viron adhesion. The interaction potentials are advantageous for a comparative study at a predictive screening of polymeric membranes for SARS-CoV-2 purification and also could be applied for new spreading variants like Omicron (work in progress).

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The Determination of Equation of State (EoS) Parameters Using Machine Learning Methods for Membrane Gas Separation

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The generality and versatility of Equation of State (EoS) models in predicting gas solubility in polymers makes them an indispensable tool in membrane processes design. However, access to reliable experimental data is required for fitting the empirical parameters associated with these models, which may not always be available, as it is often the case for high-performance polymers, e.g. polyimides. With the advent of deep learning, Artificial Neural Networks (ANN) can be employed to capture the complex dependencies of the EoS parameters on the chemical structure in a statistically superior manner than other conventional means. An ANN is a data-driven model that is made of interconnected processing elements (i.e., neurons) that form a network capable of describing nonlinear functional relationships [1]. The process of developing an ANN model for such applications can be divided into three major steps: (1) building the dataset, ensuring that it is sufficient and fully representative for the proposed application (2) training the model by providing it with input data and the desired output data for comparison. The model's parameters and hyper-parameters are then tuned accordingly (3) validating the model to test its predictive capabilities. In this contribution, we propose a proof-of-concept for the estimation of the characteristic parameters for the Sanchez-Lacombe EoS using ANN methods. The application is restricted to gases and families of polymers, with particular focus to those that are relevant to membrane gas separation processes. This investigation paves the way to the extension of this technique to other prominent EoS such as the different variants of SAFT.

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Data-driven screening and discovery of polymer membranes for gas separation: from the molecular structure to the industrial performance

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Membrane science and the design of membrane separations rely heavily on materials chemistry. Whenever a new separation has to be designed, the first step is to select the best candidate membrane material among a rather large number of possible polymers. The polymer chemistry plays a vital role in membrane separation performance, processability in solvents, and ability to selectively capture gases like CO_2. For this reason, in recent years a number of papers have proposed various types of Machine Learning (ML) based approaches to enable a fast screening of existing polymeric membranes performance for targeted applications, e.g. the CO_2/N_2 separation for carbon capture from flue gas, or to guide the discovery and synthesis of new materials. With this review we aim to provide a rational approach to guide the interested reader in finding the most suitable ML approach to predict the membrane performance based on limited information, or even from knowledge of its molecular structure only. In particular, we classify the articles reviewed by type of membrane property that is the output of the simulation: solubility, diffusivity, permeability, ideal and multicomponent selectivity, swelling and miscibility in common solvents. Those are the more relevant parameters as far as the practical application of membranes is concerned. Then, we further critically categorize the ML methods applied to obtain each type of membrane property according to its modelling strategy. For instance, in the case of works aimed at evaluating the gas solubility and permeability in polymers, we identify three families of approaches, that differ for the amount of input information required and, consequently, for the ability to estimate properties outside the training interval. We provide a detailed overview of how methodological advancements successively led to improved performance and computational efficiency, comparing and contrasting the results of different methods applied to the same datasets.

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Machine Learning Models to Predict Organic Solute Rejection in Reverse Osmosis and Nanofiltration Using Molecular Fingerprints

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Reverse osmosis (RO) and nanofiltration (NF) are experiencing growth in emerging applications including drug purification and industrial wastewater treatment. These applications require rejecting myriad emerging compounds which have not been subjected to hazard identification and can cause negative impacts on humans and ecosystems. Thus, there is a need to understand the rejection behavior of a wide range of organic compounds with polyamide membranes. Machine learning (ML) models are promising to predict rejection of emerging compounds with collected experimental data, but previous ML models for rejection have been built on molecular properties (e.g., molecular weight, dipole moment, and octanol/water partitioning coefficient) which must be computed or measured, an expensive and labor-intensive procedure when considering the enormous number of organics that exist in feed waters. Furthermore, the property features include no structural information and atom connectivity, preventing mechanistic interpretations of rejection. In this study, molecular fingerprints (MF), which transform molecule sub-structural features into binary vectors consisting of only 0 and 1, are used to represent organic compound structures and extract molecular-level knowledge for predicting rejection. First, we assess the validity of ML models, which are trained with fingerprints from 1906 rejection samples (228 unique compounds). The models showed favorable prediction performances (above 0.8 of R^2) compared to previous rejection models that used molecular properties. We then use Shapley Additive Explanations to rank input features and molecular fragments generated by MFs, allowing for mechanistic interpretation of rejection values. Lastly, Tanimoto similarity is computed from the fingerprints to group compounds and further investigate the interplay between membranes and organics. The results reveal the presence of specific interactions such as hydrogen bonding and pi-pi stacking that affect membrane rejection. Overall, our analysis elucidates the underlying effects of molecular structures on rejection and provides design principles to improve organic solute rejection.

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On the changes of boundary flux values adopting different control valves

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The development of membrane processes as a technology for environmental treatment applications and in particular for the purification of wastewater streams has significantly increased in the last decades. Fouling on membranes appears to be one of the main technical limits of this technology. This phenomenon causes the unavoidable deposition of particles on the membrane surface, building a resistive growing layer to permeability. Sensible fouling of the membrane leads to a significant reduction of the performances, a decrease of the operating life and, consequently, the increase of the operational costs due to the replacement or cleaning of the exhausted membrane modules. A possible approach to tackle the problem is adopting advanced control system strategies based on the theory of the boundary flux [1]. A critical aspect of this approach is the boundary flux determination and capability to predict the value as a function of operating conditions and time. In this framework, boundary flux values may change according to hydrodynamic conditions and more precisely on process dynamics, directly affected by control valves. In this work, different control valves characterized by different regulation speeds were used to check if boundary flux values of the membrane system keep constant or not. The constancy of the process behavior is one of the requirements for a stable control. It was checked that using different control valves with different regulation speeds and sizes affect the process dynamics and at the end the boundary flux values, and thus must be considered to allow proper design of the control systems for membrane processes. Insights to this problem will be provided.

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Evaluation of NaCl pre-cleaning of PVDF based MF membrane fouled by skim milk

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Recently hydrophilized PVDF spiral membranes were introduced in the dairy industry for the separation of casein micelles and soluble proteins of skim milk (33 g.L-1 proteins). PVDF has a high resistance toward chlorine disinfection known to induce PES membrane long term degradation. However, the crystalline PVDF polymer has an upper pH limit ≈ 11 for cleaning while PES can be cleaned up to pH 12.0-12.5. Regardless the polymer, the fouling is mainly due to proteins that are the cleaning target. The efficiency of NaCl cleaning was reported for UF PES membranes fouled by 10 g.L-1BSA (evaluation from flux measurements) [1] as well as an enhanced efficiency of alkaline cleaning for a 0.1 µm MF PES membrane fouled by O/W emulsions after a NaCl pre-cleaning [2]. In this study, a PVDF MF membrane (FR800 kD, Synder) was fouled by skim milk (0.5 bar, room temperature, 120 min), rinsed with deionised water and "cleaned" 80 min at 0.5 bar, 46°C with NaCl solutions ranging from 1.3 to 85.5 mmol.L-1. After DI water rinsing, neither flux increase nor protein removal (ATR-FTIR quantification) were obtained regardless of the NaCl concentration. Complementary experiments were achieved with 3 commercial formulated alkaline detergents classically used for UF PES membranes, but more diluted to reach a pH ranging from 11.0 to 12.0 (1 g.L-1 Ultrasil 10® and 2 mL.L-1 Ultrasil 110® , Ecolab; 2 g.L-1 Deptal 117L®, Hypred-Kersai). Finally, cascades of 1.3 or 41 mmol.L-1 NaCl + one alkaline formulated detergent were achieved. With respect to the residual protein amount, no significant difference was evidence when using the alkaline step with or without the NaCl pre-cleaning. However, the water flux recovery was sometimes poorer for the cascade procedure compared with the single alkaline step. Additional experiments with a PES MF membrane provide explanations with respect to the polymer

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Natural Phenolic Compounds as Membrane Modifiers to Decrease Biofouling Problems

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Membrane fouling is a critical issue as it greatly comprises the efficiency of membrane-based processes and water purification and plays an important role in reducing the actual performance of membranes. To ensure cost-effective and energy-efficient operations of membrane processes and wastewater treatment, it is paramount to develop strategies to prevent the fouling formation. Our aim is to identify novel antibacterial phenolic compounds and to develop novel modification routes to produce membranes with antibiofouling characteristics. The study combines computational modeling and screening, membrane modification and antimicrobial testing. Natural phenolic compounds from plant extracts and industrial side streams can be used to modify the polymeric membranes to disturb the formation of biofilm. To identify novel natural phenolic compounds, in silico shape and phase screenings from large commercial natural products library were performed. Based on virtual screening results several phenolic compounds were selected for experimental testing of antibacterial activity. Because quercetin has shown potential antibacterial properties, it was chosen to be used in an adsorptive modification of membrane samples. The filtration and surface properties of the modified membranes were tested by dead-end Amicon filtration and rejection measurements, and characterization of membrane samples was conducted by infrared spectroscopy and contact angle analysis. In addition, antibacterial activity of the modified membranes was tested with E. coli bacteria and Petrifilm count plates. The information from the experimental results can be utilized in advanced antibiofouling strategies to prevent biofilm formation in wastewater treatment applications as well as in other applications and locations, at which biofilms tend to form and cause problems. The results of this work can support the transition of the use of membrane processes in wastewater treatment to the next level and facilitate significantly the possibility to reuse water in bigger volumes than what is done today.

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Investigation of the effect of immobilization media materials in quorum quenching membrane bioreactors for biofouling control

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Biofouling is one of the most important effects that limits the use of membrane technology and increases costs [1]. The main reason for biofilm formation that causes biofouling is the result of communication between microorganisms (Quorum sensing, QS) [2]. Communication between microorganisms can be disrupted by the enzymatic degradation of signaling molecules (Quorum quenching, QQ) [2]. The aim of the study is to retardation the membrane fouling with the QQ mechanism and to increase the membrane lifetime. In this study, the production and characterization experiments of beads as an immobilization medium to be used in the QQ membrane bioreactors (QQ MBRs). Beads were produced with three different materials to present a QQ mechanism, which has proven to have positive effect on biofouling. Sodium alginate, polyvinyl alcohol and magnetic nanocomposite beads were produced and comparatively studied. To obtain optimum recipes for different materials, different combinations were tried and beads were produced in the expected size and structure in the study, since the material resistance is important for long-term operation. Cross sections of the beads were examined by confocal microscopy and Scanning Electron Microscopy (SEM). In addition, their mechanical strength was determined by various experiments. Considering the experimental results, the bead production for QQ MBR operation had been carried out with the optimum immobilization media material. Various ideas were also explored to experiment with producing beads with an innovative and sustainable material. Bead production will also be tried with the use of these innovative materials in the study. It is planned that the QQ beads produced will be used in membrane bioreactor tanks in further studies and provide a solution for biofouling on the membrane surface.

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Controlling mineral scaling using electric fields

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Mineral scaling of membranes in desalination and other water processes is a common and severe problem. Heterogenous scaling is formed when the concentration of sparingly soluble ions is increased beyond their solubility threshold near the membrane surface, leading to the formation of salt crystals. These crystals can significantly reduce membrane performance, increase energy consumption, and increase operational costs. Scaling mitigation using electric fields is a promising method;1,2 however, an understanding of the exact dynamics and scalant-membrane interactions is still lacking. It is hypothesized that electric fields mitigate heterogeneous scaling formation through the disruption of the ion stoichiometry required for nucleation, via counter-ion repulsion within the electric double layer (EDL) formed near a charged surface. In the current study, we investigated the dynamics of heterogeneous scaling formation in the presence of direct current (DC) electric fields, using confocal microscopy, which enables direct observation at a high spatial resolution of in-situ crystallization. Laser beam reflection from the membrane surface was used to probe scaling formation, since the scaled layer absorbs the reflected beams and appears as dark areas on the membrane's surface. This technique enables the observation of the scaling process - both spatially and temporally. The results couple the microscopic surface coverage of scaling and the macroscopic permeate flux decline. Process dynamics were compared with and without an applied electric field, with variations of factors known to contribute to scaling formation, such as flow velocity, operating pressure, and scaling solution concentration. Then, the tradeoff between these factors and scaling mitigating electric fields was determined. This research provides fundamental insights into the scalant-surface interactions and the framework to further develop mineral scaling control using electrical fields.

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Optimization of membrane cleaning for nanofiltration of kraft black liquor

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Membrane technology has proven to be a successful method to separate and purify compounds from several waste streams in the pulp and paper industry. One of these streams is kraft black liquor, from which lignin can be recovered by the use of nanofiltration. However, membrane technology is still not implemented in a large scale for lignin recovery, and one of the reasons for this is membrane fouling. Therefore, minimizing membrane fouling and optimizing membrane cleaning is essential to achieve long operation cycles and high flux recovery. The aim of this study is thus to optimize the cleaning protocol for NF090801 thin film composite nanofiltration membranes (Solsep BV) fouled with kraft black liquor and identify the main foulants. The influence of the cleaning parameters studied on the flux recovery were temperature, cleaning agent concentration and duration of the cleaning. The impact of the cleaning parameters was investigated with a Design of Experiments (DoE) approach using the software MODDE (Sartorius). Additionally, the membrane surface and the fouling layer were analysed with Fourier transform infrared spectroscopy (FT-IR), Brunauer-Emmet-Teller (BET) analysis and Quartz Crystal Microbalance with Dissipation (QCM-D). The results show that the main foulants on the membrane surface can be successfully removed by cleaning at 50 °C with 1wt% Ultrasil 110 (Ecolab) as cleaning agent for a duration of 60 minutes. Furthermore, during the study it was revealed that the cleaning temperature is key for the flux recovery, and a temperature between 50 and 60 ºC seems to be the most effective which is close to the maximum operation temperature of the membrane suggested by the manufacturer to avoid damaging the membrane surface. Overall, this study provides new insights to improve the cleaning efficiency of membranes.

Exploring the Membrane Fouling Effects and Cleaning Strategies in Osmotically Assisted Reverse Osmosis Processes

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Osmotically assisted reverse osmosis (OARO) has shown a great potential for low-cost and energy-efficient brine management. However, its performance can be significantly limited by membrane fouling. Here, we performed for the first time a comprehensive study on OARO membrane fouling (organic fouling, scaling and combined fouling), explored the associated fouling mechanisms, and evaluated fouling reversibility via simple physical cleaning strategies. Firstly, the results show that fouling on the membrane's draw side is negligible whereas fouling on the membrane's feed side is significant and caused by the formation of a cake/gel layer on the membrane's active layer. The greatest flux reduction was caused by the deposition of calcite and gypsum colloids in combination with calcium alginate aggregates. On the other hand, membrane scaling alone was not observed to cause a significant loss of water flux. Flux behavior in short-term operation was correlated to both the evolution of fouling and the change of internal concentration polarization. In long-term operation, membrane fouling constrained the OARO water flux to a singular, common upper limit, in terms of limiting flux, which was demonstrated to be independent of operating pressures and membrane properties. Generally, once the limiting flux was exceeded, the OARO process performance could not be improved by higher pressure operation or by utilizing more permeable and selective membranes. Instead, different cyclic cleaning strategies, including surface flushing and osmotic backwashing (OB), were shown to be more promising alternatives for improving performance. While both strategies were found to be highly effective when using pure water, a full flux recovery could not be achieved when a non-pure solution was used during OB as the foulants from the draw solution got entrapped within the substrate pores. The presented findings provided significant implications for OARO operation and fouling control.

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Toward sustainable and cost-effective production of drinking water from eutrophic and micro-polluted water using the PAC-UF membrane hybrid process

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So far, the hybrid process of powdered activated carbon adsorption and ultrafiltration (PAC-UF) is not considered the most suitable drinking water treatment process in case surface water is contaminated with both, microalgae and micropollutants, because irreversible fouling accrues atop of the membrane, which is cost effective to remove. Often, a number of conventional treatment methods are employed, including flocculation processes or adsorption on activated carbon, a material that is in many cases produced from fossil raw material. Replacing conventional technologies with the PAC-UF hybrid process, combined with the usage of PAC produced from drinking water sludge, offers a cost-effective and sustainable solution. Four sludges were collected from different drinking water treatment plants in Germany. The sludge samples, with different elemental carbon content, were activated at various conditions in a rotary kiln. The produced sorbents were tested for their micropollutant adsorption capacity using selected model compounds (i.e., caffeine, benzophenone, benzophenone-3, ibuprofen, diclophenac, trichlosan), and compared with conventionally available PAC. Benzophenone and benzophenone-3 were found to be removed using the produced sorbents to the same extent as with the commercial PAC. A good removal of caffeine was also measured, while the other micropollutants were hardly eleminated. A semi-pilot plant was constructed to examine the treatment of microalgae-conatminated water employing the PAC-UF hybid process. The motivation was to investigate whether irreversible membrane fouling can be reduced or avoided by a combination of PAC and coagulantion, combined with an improvement of the permeate quality. Based on previous expertise [1], a series of jar tests was performed using ferric chloride and polyaluminium hydroxide chloride to determine the optimal coagulant dose, pH, and G-values. Membrane filtration experiments were conducted, whereas the impact of different operational parameters (e.g., PAC concentration, PAC dosing rate, and feed flow rate) on the performance of the hybrid process was studied.

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Antimicrobial and Antibiofouling Electrically Conducting Laser-Induced Graphene Spacers in Reverse Osmosis Membrane Modules

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An ongoing and persistent challenge for water treatment membrane processes is biofouling. Reduced operation, maintenance and membrane module replacement costs can be achieved by novel ways to reduce biofilm growth on the membrane surface or on the polymeric feed spacer. Laser-induced graphene (LIG) is an electrically conductive carbon material shown to have antibiofouling properties, and is readily available via a low cost, environmentally friendly procedure. Here we demonstrate antimicrobial and antifouling effects of an electrically conductive LIG coated polypropylene (PP) feed spacer under a low electrical current. When implemented into a spiral wound membrane module reduced biofilm growth on both the membrane and the spacer components was seen. The antibacterial property of the LIG spacer was tested using Pseudomonas aeruginosa and the brackish water Rheinheimera sp. as model organisms. P. aeruginosa was completely inactivated in 10 h using a voltage of 12 V, while a dynamic accumulation assay employing Rheinheimera sp. significantly reduced ($p<0.05$) bacterial adhesion compared to an uncoated spacer. The spacer was incorporated into a spiral wound reverse osmosis membrane module, and electrical connections were engineered through the module housing to allow passage of electrical current through the spacer while operation under pressure. Reduced biofouling was observed on both the membrane and LIG spacers components using brackish water and 12 V. This study demonstrates the feasibility of electrically conductive feed spacer components in spiral wound RO membrane modules.

Quantifying the Biofouling Potential of Polymer Films: A Combined AFM and QCM-D Study

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Biofouling adversely affects the operation of submerged surfaces such as water filtration membranes. To further understand the mechanisms behind the biofouling of polymeric membranes, we use atomic force microscopy (AFM) colloidal probe technique and quartz crystal microbalance with dissipation monitoring (QCM-D) to measure the biofouling potential of a model biofoulant, i.e., bovine serum albumin (BSA), toward an array of polymer films: (cellulose acetate (CA), polyvinyl chloride (PVC), polyvinylidene fluoride (PVDF), and polysulfone (PS)), frequently used in membrane synthesis. AFM colloidal probes coated with BSA were employed to measure the adhesion of BSA molecules to the polymer films. In parallel, gold-coated QCM-D sensors were covered with the polymer films and used to measure nanosensitive changes in the BSA adsorbed masses. In addition, with the help of contact angle and zeta potential measurements, we apply the extended Derjaguin, Landau, Verwey, and Overbeek (XDLVO) analysis to the system in order to reveal the contributions of each component force (electrostatic, Lifshitz van der Waals, and Lewis acid-base forces) to the overall adhesion force that causes biofouling. The adhesion of BSA to all of the polymer films in deionized water was dominated by the Lewis acid-base force component, which contains both hydrogen bonding and hydrophobic interactions. A similar trend was observed for the microscale AFM adhesion forces, QCM-D rate and molecular scale mass of BSA adsorption, and the free energies (ΔG132) measured from the microscale contact angles. This trend was consistent also with the energy barriers approximated by the XDLVO analysis except for PVDF, which probably was due to its highest roughness (roughness ratio of 1.09 for PVDF) as compared to the other investigated polymer films. The polymer films were ranked in the order of PS> PVDF> PVC> CA, according to their biofouling potential.

Impact of enzyme stabilizers and formulation on the real efficiency of enzymaticdetergent useful to clean polymer membrane fouled in skim milk ultrafiltration

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PES/PVP membranes are widelyused for skim milk ultrafiltration, but membranes aresystematically fouled by proteins. An enzymatic cleaning can be desirable to remove them.However, enzymes used alone, are generally not efficient enough and formulations have to bedeveloped that willfurther be diluted in a buffer at the appropriate pH. The formulateddetergent can be decomposed in the enzyme and the matrix containing all added ingredients(except the buffer). The formulator can be pro-active in the matrix ingredient selection but itis more difficult to control the impacts associated with the choice of enzymes because onlystabilized enzymes are commercially available. They are mixed with stabilizing agents thatcould potentially have an impact on the final detergents' performances. To account for theimpact of these stabilizing agents, a prototype matrix has been formulated by our industrialpartner (Hypred®) in which one among 3 different stabilized subtilisins (protease) has beendissolved (same hydrolysis activity) [1]. Only part of the stabilizers was identified thanks tothe safety data sheets given by the enzyme providers. The selected UF membrane (HFK-131,Koch) has been fouled by skim milk in standardized conditions (50°C, 2 bar, 0.5 m. s-1, 180min). Then, the cleaning efficiency at 50°C (2 bar, 0.5 m. s-1, 60 min) of the prototypedetergents was evaluated by the mean of water flux recovery (WFR). Moreover, residualproteins were quantified on membrane by ex-situ ATR-FTIR evidencing a very high cleaningefficiency. Based on complementary ATR-FTIR characterization of membranes, thediscussion provides a comparison of the cleaning efficiency of stabilized enzymes + bufferwith that of the prototype detergents aiming also at giving a fundamental explanation of theWFR sometimes greater than 100%. Discussion of the respective role of stabilizers andmatrix ingredients provide keys to go ahead in the formulation optimization.

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Optimizing the technical parameters of AnMBR with commercial PES membrane

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Anaerobic membrane bioreactors (AnMBRs) are gaining popularity as a more economically viable alternative to aerobic processes. AnMBRs reduce the demand for chemical oxygen and convert organic matter into biogas, a resourceful form of energy. Moreover, AnMBRs can generate effluents free of solids and pathogens and rich in nutrients while occupying a small footprint. However, some drawbacks remain, such as membrane fouling and its effects. In this study, a commercial polyethersulfone (PES) membrane was used in AnMBR with a 0.5 m^3/day scale. Important operating parameters including hydrodynamic conditions, membrane flux, HRT, SRT, MLSS, temperature, and pH were investigated in order to find the ideal hydrodynamic conditions for preventing membrane fouling. The intensity and duration of gas flushing in submerged AnMBRs was increased as well as the flow rate of mixed liquids in sidestream AnMBRs. Membranes operated with a filtration flux lower than the critical flux in order to prevent membrane fouling. The operating parameters impacted the metabolic processes of microorganisms, particularly the release of soluble microbial products (SMP). Longer solid retention times hastened the complete breakdown of solid matter but at the expense of an increase in sludge and SMP concentration, both of which increased membrane fouling.

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Optimizing Flocculation of Digestate to Increase Circularity in the Nutrient Recovery from Manure

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The agricultural sector aims for valorization of pig manure into separate nutrient-rich fractions for precision fertilization while limiting nitrogen and (climate driven) CO_2 emissions. This often involves the production of biogas by co digestion of manure, followed by several mechanical and membrane separation technologies to produce nutrient rich fractions [1]. The mechanical separation, a process generally improved by the addition of clarifying agents, produces a liquid and a solid fraction. The solid fraction contains a large amount of phosphorus. The liquid fraction is subsequently cleaned from floating parts, after which a high quality liquid stream is obtained that is further treated by reverse osmosis to produce mineral concentrate and/or nitrogen or potassium rich fractions for precision fertilization [1]. Currently, the dosage of clarifying agent in the solid-liquid separation step is done rather intuitively. This often results in overdosage and thus accumulation of polymers in the system as well as in the recycle loops, inevitably ending up in the RO process step, thus resulting in increased fouling of the RO membranes and higher operating costs. In this study we therefore aim to gain a better understanding of the flocculation/coagulation mechanism in the pretreatment step and thus improve the solid-liquid separation efficiency to decrease the burden on the RO membranes. This is done by investigating various synthetic polymeric clarifying agents and the addition of iron salt as coagulant. The organic matter removal from the digestate is measured in terms of turbidity and compared to the corresponding dosage of polymer into the digestate. Results show that the removal efficiency and its stability depend on the characteristics of the clarifying agents such as molecular weight, structure and charge.

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Optimization of Coagulant Dosage and Pre-treatment Configuration to Minimize Ultrafiltration (UF) Fouling

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Coagulation/flocculation prior to ultrafiltration (UF) may be optimized for the reduction of fouling or removal of natural organic matter (NOM). However, the selection of optimal coagulant dose and pre-treatment configuration applied prior to UF remains a major knowledge gap. Three main coagulation mechanisms may be defined as: i) adsorption destabilization, ii) combined adsorption and sweep, and iii) sweep coagulation. Coagulation, which includes coagulant addition and rapid mixing, may be followed by flocculation to increase particle aggregation and floc size. While evidence suggests that both coagulation mechanism and pre-treatment configuration (coagulation only, coagulation + flocculation) impact floc properties, their combined impact on floc properties and subsequent UF performance has not been clearly defined. This study evaluated the impacts of coagulation mechanism and pre-treatment configuration on UF performance for two source waters using a bench-scale continuous-flow coagulation/flocculation-UF system. Jar tests were performed to identify optimal pH and alum dosages with respect to each coagulation mechanism in terms of NOM removal. During UF trials, coagulation (G = 350 /s, HRT = 2 min) and coagulation + flocculation (G = 20 /s, HRT = 12 min) were utilized to continuously produce feed water. Results to-date indicate that the combination of adsorption destabilization (pH 5.5, 2.5 mg/L alum) and coagulation + flocculation affects the properties of floc $\leq$1 μm in size, increasing hydraulically reversible/irreversible fouling resistance, when compared to no coagulant addition, while providing similar NOM reduction. Enhanced particle aggregation and floc growth for a combined coagulation mechanism and coagulation + flocculation is anticipated to result in optimal UF performance by simultaneously increasing cake layer permeability and NOM removal. Guidance regarding coagulant dose selection (favouring a specific coagulation mechanism) and pre-treatment configuration could be readily adopted by the water treatment industry. Quantification of floc properties provides evidence regarding the mechanisms which dictate optimal performance.

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Pre-Treatment Adsorption Methods of Reverse Osmosis for Removal of Surfactants and Organic Compounds from Electroplating Wastewater

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Reverse osmosis (RO) is one of the most suitable technologies for the treatment of electroplating rinse water, due to its superior rejection, aiming to recover heavy metals such as Cr (III), Cu and Ni that can be reused in the electrolyte baths, on the one hand, and pure water, on the other. However, the high load of surfactants, carboxylic acids and other organic compounds of the electroplating rinse water may damage, reduce longevity and cause fouling to the RO membranes. This leads to partial effective pore size reduction and blockage that in turn reduces permeate flux and increases operational energy demands. Therefore, the industry is in need for efficient selective pretreatment methods for the removal of surfactants and total organic carbon (TOC) content, without affecting heavy metal concentrations. In this study, we investigate three ion exchange resins and powdered activated carbon (PAC) as possible pre-treatment methods for surfactants and TOC reduction. The adsorption kinetics were investigated using real Cr (III) electroplating rinsing bath solution and fitted to three adsorption models (Freundlich, Langmuir and Redlich-Peterson). To determine the reduced fouling tendency, the reduction of chromium, surfactants and TOC content was monitored for different wastewater concentrations and pre-treatment conditions. Initial measurements with the use of activated carbon showed that above 90% of anionic surfactants were removed from the rinsing bath solution, while the chromium concentration remained constant, as desired.

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Better Performing Ultrafiltration Membranes: from Fundamental Research to Large-Scale Membrane Modules

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There is a growing need for novel water purification concepts based on membrane filtration. Emerging pollutants containing viruses may over time cause severe health problems. Membrane can provide an excellent barrier for these contaminants, in order to yield purified water at high throughput. For an efficient purification process, the type of membrane used is key. Polymeric capillary membrane filtration is a well-known concept in the world of ultrafiltration (UF) and microfiltration (MF) membranes. One of the major advantages of capillary membrane filtration is the fact that, due to the configuration, little to no pre-treatment is necessary. Furthermore, with hollow fibres, it is possible to perform a backwash and thereby easily reducing the fouling behaviour of the membrane. This work presents the newly developed capillary hollow-fibre ultrafiltration membranes by Pentair X-Flow. Our researchers and product developers have created a stronger membrane material that enables the fibre's inner and outer diameters to be modified, which results in an extension to 75 m^2 of active membrane surface area. By not increasing the element size, the new X-Flow XF75 Membrane is applicable to projects entailing common membrane element size without the added complications to accommodate footprint differences. Excellent chemical resistance and the possibility for back-washing make these membranes very suitable for cleaning procedures. Within these characteristics, this newly developed membrane module is extremely applicable for the production of potable water.

Evaluation of the effectiveness of physicochemical pre-treatment of flat-sheet and hollow fiber membranes in anaerobic MBR for seafood processing wastewater treatment

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Currently, seafood processing industry is developing due to the increasing demand for food consumption. Therefore, the issue of wastewater in this industry needs more attention. This study evaluates the effective potential of physicochemical pretreatment in anaerobic MBR in the treatment of seafood processing wastewater. The wastewater parameters, measured at the Baseafood I Co. in Ba Ria Vung Tau City, such as as chemical oxygen demand (COD), total nitrogen (TN), total phosphorus (TP) and salinity were of 1000-2500 mg/l, 100-250 mg/l and 25 ± 5 mg/l and 5-10 g/l tnam, respectively. The treatment carried out by means of hollow fiber membranes significantly reduced total suspended solids (TSS) by more than 70% and resulted in a decrease in COD, TN and TP concentrations. This maximized the hydraulic retention time (HRT) and the treatment efficiency. With an HRT, in the anaerobic treatment tank, from 8 to 12 hours, the COD removal efficiency always remained stable above 90% and, at the same time, achieved a conversion efficiency of total nitrogen to ammonium ($N\_NH_4^+$) more than 90% with a phosphorus removal efficiency also over 40%. Besides that, the flux through the membrane remained stable with a suction and backwash time ratio of 9:1 during operation. This study proposed a flexible condition for the pretreatment of seafood processing wastewater using AnMBR.

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Development of a sustainable cellulosic membrane platform with green solvents

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Most porous polymeric membranes are made from polymer solutions (casting solutions) where membrane formation is based on phase separation. There are four main mechanisms for phase separation: vapor-induced phase separation (VIPS), non-solvent-induced phase separation (NIPS), evaporation-induced phase separation (EIPS) and thermal-induced phase separation (TIPS). Often, the precipitation casting process consists of a combination of two mechanisms VIPS and NIPS. This provides the possibility to vary the resulting membrane structures and thus the membrane properties in many ways. In addition to the specified process parameters, the resulting membrane properties are also dependent on the composition of the casting solution. To influence the thermodynamics and kinetics of membrane formation, various solvents and additives are part of the casting solution in addition to the membrane-forming polymer. Cellulosic materials show excellent properties like low fouling, high chemical resistance and cellulose is sustainable. Nevertheless, it’s difficult to handle membranes based on cellulose materials because of mechanical stability and gamma sterilization stability. In this work different green solvents like Cyrene or Methyl lactate are tested to develop a sustainable cellulose acetate microfiltration platform. One target is to create high performance membranes with tailored structures. Therefore, the membrane casting process (VIPS + NIPS) is developed on a laboratory scale casting line. Here the most important process parameters can be investigated and a pre-development for production scale is conducted efficiently. To deepen the understanding of membrane formation phase diagrams [1] are developed. The deepened understanding of the membrane formation process allows a more effectively process development.

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Utilization of Commercial Ultrafiltration Membranes to Recover Phenolic Compounds from Wet Olive Pomace

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The antioxidant properties of phenolic compounds have been widely demonstrated, and they motivate the application of these molecules in the cosmetic, pharmaceutic and food industry [1]. The olive fruit is one of the main natural sources of polyphenols. This is also applicable to the olive mill by-products, such as wet olive pomace, which consists of a semi-solid residue, rich in phenolic compounds [2]. Polyphenols from wet olive pomace can be extracted by an aqueous solid-liquid extraction (at 40ºC, for 45 minutes), but this extract inevitably contains other undesired organic compounds. Thus, ultrafiltration can be employed to remove the concomitant organic matter and obtain a permeate enriched in phenolic compounds. To that end, four polymeric commercial membranes, UP005, UH030, UH050 and UP150 (Microdyn Nadir) were tested, at three cross-flow velocities (1.5, 2.5 and 3.5 m/s) and a wide range of transmembrane pressures (0.75–5.5 bar). To clean the membranes, several strategies were evaluated, such as aqueous cleanings (at 20 and 35ºC) and the application of 1% (v/v) P3 Ultrasil 115 (at 20, 35 and 40ºC). The observed values of permeate flux at the steady-state varied between 15 and 150 $L\cdot h^{-1}\cdot m^{-2}$, depending on the membrane. Regarding the rejection of the chemical oxygen demand (COD), it varied with the membrane and the operational parameters, but a satisfactory rejection (>55%) of COD was achieved in most cases. To evaluate the individual rejection of each phenolic compound, a separative methodology was applied, based on liquid chromatography coupled to mass spectrometry. Among the five families of polyphenols that were identified, simple phenols (including tyrosol and hydroxytyrosol) were poorly rejected (0-40%). Flavonoids, secoiridoids and phenolic acids were also recovered in the permeate at a higher purity.

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REDWine: Chlorella’s Harvesting System Using Membrane Technology

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REDWine is a European project focused on the utilization of biogenic carbon dioxide (CO_2) coming from the wine fermentation process for microalgae biomass production and valorisation. A powerful synergy across bio-based industries results in REDWine’s innovative circular business model, which allows wine manufacturers to efficiently treat their liquid and gaseous effluents while profitably diversifying their revenues through the valorisation of Chlorella biomass into multiple high-value ingredients. REDWine will demonstrate a circular concept through the development of a simple biorefinery to be deployed in the winery which will yield sustainable and cost competitive ingredients for food formulations (protein and fatty acids), cosmetics (peptides, carotenoid rich oils and active polysaccharides), agriculture (carbohydrates as vine biostimulants) and wine production (proteins for wine clarification). Harvesting of algae from the cultivation media via centrifugation is an energy-intensive process that limits the sustainability of utilizing such technique. To minimise the harvesting process cost, pre-concentration using membrane technology will be validated as an additional step before applying centrifugation for complete harvesting of the algae. Membrane filtration can be operated continuously, does not require extra chemicals and has a low energy requirement. In REDWine, electrospun nanofiber membranes (ENM) together with conventional membranes are being developed and tested for Chlorella’s harvesting. The ENM can be synthetized using different polymers and these membranes show high porosity and superior permeability. The algae filtration capacity and energy requirements are investigated during the project by optimizing the operational parameters to reduce membrane fouling of the developed ENM and commercial membranes. The main objective of this project is the validation of demonstration-scale Chlorella production unit which includes a membrane harvesting system.

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Modification of Ultrafiltration Membranes to Enhance the Adsorption of Cations and Anions

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Safe and clean drinking water is an essential resource. Nevertheless, many sources of waters are often contaminated. Besides microbiological contaminations, pollution by chemicals such as heavy metals are relevant in many parts of the world. These chemicals are highly toxic and cause serious illnesses like cancer. Therefore, it is important to eliminate these contaminants from (drinking) waters to standards that ensure a safe supply. Many heavy metals are present in water as cations (e.g. copper, nickel and lead), but harmful oxyanions (e.g. arsenate and chromate) can be found also. Therefore, ion adsorbing porous membranes provide an interesting possibility to remove these ionic contaminants and microbes in a one-step procedure without adding chemicals to the water. Ion adsorbing materials usually use counter ionic functional groups to adsorb cations and anions. This means that anions are adsorbed using positively charged groups (e.g. quaternary amines) while cations are adsorbed using negatively charged groups (e.g. carbonyl groups). In this study, we present ion adsorbing ultrafiltration membranes that can be operated at low energy demanding pressures (<1 bar). Thus, polyacrylonitrile ultrafiltration membranes were chemically modified by the introduction of charged functional groups. The success of the modifications is demonstrated using chemical (e.g. zeta potential, IR spectra) and material characterization methods (e.g. pore size, water permeance). The modified membranes show high permeances (>1000 L/(m^2 h bar)). Additionally, the ion adsorbing properties of the membranes were analyzed using environmentally relevant cations and anions. Copper and cromate were applied in this study, as they are commonly found in source waters of drinking water supplies. The modified membranes were able to absorb the oppositely charged ions while the pristine ultrafiltration membranes did not reject the ions. Therefore, the purification of water from toxic ions using these membranes is possible.

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Dye removal with waste cotton textile and wood-based cellulose-rich ultrafiltration membranes: on the mechanism of dye removal

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The interest in the production and study of cellulose membranes originates from the biological nature of cellulose, its abundance, and potentially high applicability for water-based waste streams treatment. The utilization of nature-based materials allows upgrading the whole process to a higher level of environmental compatibility. However, the reports on the utilization of cellulose membranes are scarce, usually presenting petroleum-based polymeric membranes as filtration media. In our previous study, we showed the usability of plant cellulose-based membranes in removing residual phosphorus from the secondary clarifier wastewater effluent [1]. The current study aims to check the applicability of the produced membranes for textile wastewater treatment and study the mechanism of dye removal. Five cellulose-rich ultrafiltration membrane samples (MS1 – MS5) with different chemical compositions and performances were chosen for the experiments. All membranes showed similar adsorptive removal of the dye, both in neutral (7.4) and acidic (2.7) pH. Negligible adsorption in neutral pH might originate from the charge repulsion between the negatively charged surface of the membranes (−26 to −71 mV, depending on the sample) and the negatively charged functional groups of the dye. When the pH was adjusted to 2.7, all membrane samples showed a positive surface charge (1 to 10 mV, depending on the sample) and similar adsorptive removal values between 24.5 and 28.5%. MS3 and MS5 showed negligible dye removal in filtration experiments, which correlated well with their significantly higher permeabilities (450 and 380 LMHb, respectively). The results also revealed that based on the retention of non-charged polyethylene glycol (PEG) molecules, the retention of dye could not be predicted. Although the PEG 35 kDa retention is only 80 or 76% (for MS1 and MS2, respectively), the dye removal with these membranes is about 95%. All this implies the significance of electrostatic repulsion in dye removal with membranes.

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Remediation of water from per- and polyfluoroalkyl substances: ozonation vs electrochemical oxidation after coagulation-microfiltration

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Per- and polyfluoroalkyl substances (PFAS) are widely used as additives to either enhance the thermo-chemical stability of products or alter the properties of surfaces. However, PFAS are harmful to the environment and toxic to humans. Therefore, the EU included PFAS in the revised Drinking Water Directive in 2021 (0,5 μg/L for PFAS-total). Treatment of PFAS in wastewater is a challenge due to their persistence and low concentration in the complex wastewater matrix. Therefore, treatment trains require capturing or concentration followed by degradation. This study compares two options for destructing PFAS after coagulation-microfiltration: ozonation and electrochemical oxidation. Effluent after coagulation has been collected from a small-scale wastewater treatment plant in Norway (29 000 pe), filtered with a silicon carbide microfiltration membrane (Cembrane, Denmark), and oxidised in laboratory conditions applying ozone or boron-doped diamond (BDD) anode. We have studied the influence of PFAS concentration (C4-C12) together with two groups of process factors corresponding with microfiltration (flux and filtration time) and oxidation (dose/current density and flow rate). The results show the benefits of applying electrochemical oxidation after coagulation-microfiltration compared to ozonation. This includes treatment efficiency (84-99,88%) and estimation of operational costs. In addition, we have also observed a trend of PFAS degradation from C14 to C4 that gives an idea about possible oxidation mechanisms. Overall, the current density and oxidation time are directly proportional to the degradation rate of PFAS after coagulation-microfiltration.

1-(2’-Hydroxyethyl)pyrrolidin-2-one as Green Solvent for Poly(Ethersulfone) Ultrafiltration Membrane Fabrication

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Today poly(ethersulfone) (PESU) ultrafiltration (UF) membranes are manufactured by non-solvent induced phase separation (NIPS) in N,N-dimethyl acetamide and N-methyl-2-pyrrolidone as polar aprotic solvents which are rated as substances of very high concern. According to the principles of “Green Chemistry”, solvents as auxiliary substances should be made unnecessary wherever possible and innocuous when used [1]. In this contribution we introduce 1-(2’-Hydroxyethyl)pyrrolidin-2-one (HEP, CAS 3445-11-2) as novel, fully commercially available and innocuous polar protic solvent for PESU UF membrane preparation by NIPS processing. According to the globally harmonized system, HEP has no labelling and is REACH registered for quantities > 1000 to < 10000 tonnes per annum. Due to its high viscosity of 77.2 mPas (25 °C), 20 wt% solutions of PESU (Ultrason® E3010) in HEP have higher dynamic viscosity of 18 mPas (60°C) compared to 1.4 mPas for Agnique® AMD 3 L, 3.95 mPas for 2-pyrrolidone as comparable polar protic and 0.42 mPas for N-methyl-2-pyrrolidone as polar aprotic solvent. HEP allows the use of low molecular weight poly(vinylpyrrolidone) (PVP) or poly(ethyleneoxide) pore forming additives for the PESU UF membrane preparation. Flat sheet membranes were prepared from PESU solutions in HEP with PVP K12 (Kollidon® K12) by coagulation in water-HEP mixtures. Subsequent oxidative PVP removal procedure was omitted [2] and the post treatment was carried out soley as water wash. Depending on the PESU/PVP ratio membranes with hydraulic water permeances rangeing from 190 to 690 kg m^{-2} bar^{-1} h^{-1} and molecular weight cut-off values from 2.9 to 10 kDa were obtained. Scanning electron microscopy showed the membranes’ well established filtration layers. As summary, HEP proved to be a green solvent for the PESU UF membrane fabrication using low molecular weight PVP without oxidative post treatment. These membranes could have applications for surface water filtration and the precleaning step for seawater desalination.

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Ultrafiltration as pre-treatment to improve vinasse biomethanation potential

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Vinasse is a residue that remains after the fractional distillation of fermented juice to obtain ethanol. Its composition in terms of nutrients and organic matter, aligned with the high volume produced, motivated fertigation practices that still run into a limited economic radius. Therefore, aiming to extend the alternatives of resources recovery from vinasse, it was studied the biomethane production from vinasse and its fractions obtained after ultrafiltration (permeate and concentrate). The membrane application was a strategy to concentrate organic compounds in vinasse and alleviate the concentration of sulfate, sulfide, or thiosulfate that could potentially inhibit biomethane production. The ultrafiltration concentrate presented the highest degradation rate (μ_m: 2.83 ± 0.24 mL/g-VS.d.mL-sample) and longest lag-phase time (λ: 12.31 ± 0.83 d) compared with raw sugarcane vinasse (μ_m: 1.44 ± 0.11 mL/g-VS.d.mL-sample; λ: 6.57 ± 0.93 d) and ultrafiltration permeate (μ_m: 1.14 ± 0.04 mL/g-VS.d.mL-sample; λ: 2.92 ± 0.74 d). Furthermore, the biomethane recovery from ultrafiltration concentrate presented the lowest specific energy consumption (3.341 MJ/Nm^3-CH4), followed by raw sugarcane vinasse (3.515 MJ/Nm^3-CH4) and ultrafiltration permeate (3.643 MJ/Nm^3-CH4). The digested streams could still be applied to soils would for better use of their nutrients, however without an excessive load of organic matter. Overall, the study demonstrated an effective alternative to salinity relief in raw sugarcane vinasse by ultrafiltration membranes, and an alternative of higher vinasse valorization despite its single reuse in fertigation practices and treatment for disposal.

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A New Insight on Heavy Metal Removal by Absorptive Nanofiltration Membranes

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Adsorptive membranes combining both advantages of adsorption and membrane separation with high flow rate, low internal diffusion resistance and fast adsorption/desorption rates have attracted much attention for wastewater treatment in recent years [1,2]. Thin-film composite (TFC) nanofiltration (NF) membranes have a negatively charged active polyamide (PA) layer capable of adsorbing different cations, including heavy metal cations. The adsorptive capacity of the NF membranes can be further increased by incorporating appropriate nanoparticles into the PA layer. In this study, we investigated the dynamics of adsorption and rejection of heavy metals (Cu^{2+}, Pb^{2+}) by TFC and L-cysteine functionalized cellulose nanocrystals (CysCNCs)-based thin-film nanocomposite (TFN) membranes. We performed NF filtration experiments of different duration, including 4, 8, 12 and 16-hr. We monitored the water flux and concentration of heavy metals in permeate every hour. In addition, we determined the content of heavy metals absorbed by the membranes after the end of different duration experiments. The results demonstrated that the water flux reached the constant value after increasing during the first 4 hours of the experiments. The rejection of heavy metals also increased with time, but it took 8 hours to reach a constant value. Interestingly, heavy metal adsorbed content on the membranes was similar after 8, 12, and 16-hr tests. It suggests that it took between 4 to 8 hours for the membranes to reach equilibrium capacity for heavy metal adsorption. Despite reaching the equilibrium capacity for heavy metal adsorption, the water flux and heavy metal rejection remained at their highest values. This study suggests that adsorbed heavy metals from the feed solution improve the performance of both TFC and TFN membranes. The steady-state water flux and heavy metal rejection by TFN membranes were greater than those of TFC membranes.

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On the transmission of organic solutes in RO of salty and sea waters

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The presence of micropollutants in seawater will be an increasing problem to be faced during the desalination process. Sea water reverse osmosis (RO) membranes were primally designed for the inorganic salts' rejection, but the removal of (small) organic solutes appears as a new increasing challenge. Recently, small organics (MW $\leq$ 200 g.mol^{-1}) were evidenced to be only partially rejected by RO membranes. We have already achieved a preliminary RO studies using a low pressure RO membrane (TFC-HR, Koch) evidencing (i) the transmission of a positively charged vitamin and a negatively charged dye of close MW (254-293 g.mol^{-1}) in the 15-30 bar range and (ii) the impact of the physico-chemical environment (deionized (DI) water, 30 g.L^{-1} NaCl and seawater) on these transmissions [1]. However, the solute molecular weight limit, leading to a systematic full rejection is not clearly known and is probably associated to either hydrodynamic or physico-chemical conditions during RO. This study addresses this question by filtering a set of organic solutes at 0.5 mmol.L^{-1} each, a typical concentration range for nanofiltration but not for RO. The solutes (single, binary, ternary mixtures) were dissolved in DI water, 30 g.L^{-1} NaCl or seawater aiming at evidencing the impact of the salt content and their chemical nature on the organics' transmission. A set of azo-dyes, vitamin and antibiotics with MW ranging from 254 to 916 g.mol^{-1} and different properties (charges, hydrophobicity…) were selected. RO experiments were performed with the SW30 membrane (Filmtec) in the 30-40 bar applied pressure range in cross-flow conditions for which no concentration polarization was associated to the organics. Surprisingly, regardless of their MW and the presence or absence of salts, all the azo-dyes were transmitted through the membrane. This study provides a discussion of the physico-chemical parameters accounting for the partial transfer through the polyamide "dense" membrane.

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Characterization of membrane performance in whey nanofiltration

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Whey valorization is of fundamental importance to both the dairy industry and the environment. Among several possibilities, the recovery of whey's most valuable components, whey proteins, lactose and peptides, represents a real opportunity and requires several unit operations. In this context, nanofiltration is conventionally used to partially demineralize and concentrate lactose from a previous ultrafiltration step [1]. However, it is noteworthy to mention that the recovery of whey components is strictly dependent on its chemical composition [2]. In this work, two commercial thin film composite flat membranes manufactured by Suez, DK and DL (98% and 96% MgSO4 nominal rejection, respectively), were characterized in whey nanofiltration in a wide range of conditions. The characterization was performed in a radial flow cell placed in a pilot-scale plant operating in total recirculation mode. The main objective is to assess the effects of operative conditions on membrane performance by varying transmembrane pressure, feed concentration, pH and temperature. To this aim, mock solutions have been created to mimic the wide range of whey compositions, including acid and sweet whey. Sodium chloride, lactose and lactic acid were used; firstly the effects of individual solutes and then their combined effect in mixtures were studied. The data were preliminary analyzed in terms of hydraulic permeability, permeate flux and observed membrane rejection. Then an in-depth analysis of the mass transport phenomena, consisting in the evaluation of concentration polarization and osmotic pressure effects was performed as to calculate the intrinsic membrane rejection and the effective driving force. Given the increasing volume of acid whey being generated, improving the yield of lactic acid and lactose separation is a real need and can lead to several benefits. The results of this work provide a contribute to the ongoing research to improve whey valorization, and can be potentially exploited by the dairy industry.

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Novel Interfacially Polymerized Polyamide-Silicon Quantum Dots Composite Membranes for Nanofiltration Process

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The development, protection, recovery, and reuse of water resources are an important issue currently. Nanofiltration is an essential water treatment technology because of its cost saving, energy saving and environmental protection. In this study, the amino-functionalized silicon quantum dots (SiQDs) were fabricated by the hydrothermal synthesis method. Amino-functionalized SiQDs were incorporated into the polyamide layer through the interfacial polymerization to fabricate the polyamide-SiQDs composite membranes. The resulting composite membranes were applied to the nanofiltration process for salt and dye aqueous solutions. The results of attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy analysis and scanning electron microscopy (SEM) observation confirmed the polyamide-SiQDs layer was synthesized successfully on the polysulfone (PSf) support membrane. From the results of nanofiltration experiments, the polyamide-SiQDs composite membrane with the optimum fabrication condition (CHDASiQDs-tNBDC/PSf) showed a high dye (rose bengal) rejection of 99.4% with a low salt rejection. This result exhibited that the polyamide-SiQDs composite membrane had a potential for the nanofiltration separation of dye/salt mixture.

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Polymeric fluorine-free ionomers and their complexes with polyelectrolytes for thin-film composite nanofiltration membranes

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Selectivity in terms of ion and micropollutant rejection of nanofiltration (NF) membranes is a crucial factor for the performance of NF applications, such as wastewater treatment. Membranes based on a commercial fluorine-free ionomer may extended the available selecitivity spectrum as well as stability compared to common NF membranes [1]. Herein, the fabrication of a tunable and highly stable ionomer NF membrane by coating the negatively charged blockcopolymer Nexar on a support membrane with ultrafiltration characteristics is reported. The support membrane is prepared from L-PAN (93 % polyacrylonitrile, 7 % vinylacetate comonomer) via film casting and phase inversion. The nanoscale morphology of the Nexar film can be tuned by the solvent system of the casting solution [2]. In order to increase separation performance of the thin-film composite NF membranes beyond state-of-the-art (cf. [1]), different solvent systems are investigated. Low Nexar layer thickness (> 1 µm) as pre-requisite for high permeance of the membrane is controlled by dip-coating. The filtration performance of the new membranes in terms of solute rejection (Na2SO4, NaCl, CaCl2, sucrose) and permeance is competitive with established NF membranes. Subsequently, in analogy to recent other work with Nafion as part of the selective layer [3], a polyelectrolyte complex membrane is prepared by impregnating the fluorine-free Nexar layer with the polycation polyethyleneimine to further tune membrane separation performance.

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Effect of Relative Humidity on Nanofiltration Membrane Preparation via Phase Inversion

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Membrane structure, properties, and performance can be influenced by several factors during the membrane preparation process. Relative humidity (RH) can be an important and often academically overlooked attributing factor. In this study, one of the most common polyimides, Matrimid® 5218, was selected to prepare membranes using different ratios of N-Methyl-2-pyrrolidone (NMP) and tetrahydrofuran (THF) as solvent and co-solvent respectively. To observe the extension of relative humidity's effect, the membranes were synthesized by vapor induced phase separation (VIPS), followed by the non-solvent induced phase separation (NIPS) technique. Relative humidity and exposure time of membranes to it, were varied at different stages. The prepared membranes were characterized by water permeance, retention of two different sizes of dyes (rose bengal (RB, 1017 $gmol^{-1}$), and methyl orange (MO, 327 $gmol^{-1}$) in water using dead-end filtration and Scanning Electron Microscope (SEM). At high relative humidity and long exposure time, permeance increased substantially, while having less effect on retention of RB in water. At middle-range humidity, the effect on permeance was even more subtle. At lower humidity, membranes show less consistency. Changes in structure assessed by SEM images did not show a pronounced change in the morphology of the prepared membranes.

Downstream processing of corn steep waters for biosurfactants recovery by nanofiltration technology

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Corn steep waters (CSW) are generated during the steeping process of corn as a by-product. CSW are normally used not only as a low-cost source of nitrogen in biotechnological processes, but also as a direct source of cost-competitive biosurfactants [1]. In the case of CSW, the biosurfactants are obtained spontaneously by lactic acid bacteria as well by Bacillus sporulated strains [2]. In this work, the recovery of biosurfactants from CSW using membrane technology, in particular with nanofiltration processes, has been proposed. A lab-scale membrane MP025 module unit from NXFiltration BV (The Netherlands) with a hollow fiber nanofiltration (NF) membrane, named dNF-80 (MWCO of 800 Da), is used to treat 4 L of CSW diluted up to 50 g/L and without solids by a centrifugation step. The diluted and centrifuged CSW was pumped into the membrane cell, with an active area of 0.050 m2, while feed pressure was varied between 2.5 to 6.5 bar, with 10 min of stabilization between each pressure value. After each stabilization, a permeate sample was taken to measure the pH, surface tension, and organic acids concentration. The initial composition of CSW was as follows: pH=4.3, surface tension of 53.9 mN/m, 3.5 g/L of lactic acid and 5.4 g/L of phytic acid. After the nanofiltration process, results showed a permeate stream rich in lactic acid (maximum rejection about 47%) and a concentrate stream rich in biosurfactants (the surface tension in the permeate was about 71 mN/m, like water (72 mN/m)) and phytic acid (maximum rejection around 84%). The pH of permeate samples did not change with the transmembrane pressure. It should be noted that this is the first work in which this type of membrane was used for the recovery of biosurfactants. Therefore, NF is an innovative technology to purify CSW, obtaining a concentrate rich in biosurfactants.

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Predictive modeling of arsenic and manganese removal by reverse osmosis by multi-linear regression

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The risks to human health by exposure to manganese and arsenic are known. These components are found in surface and groundwater and, therefore, should be removed. Knowing this fact, it is necessary to evaluate the integration potential of reverse osmosis (RO) for a conventional water treatment plant, as well as the impact that the change in physical-chemical parameters would bring to the system. To perform a predictive modeling, the multiple linear regression tool was applied to promote a linear adjustment between the removal values of each element and the physical-chemical parameters: turbidity, pH, conductivity, apparent color, and concentrations of components of interest and iron. Both the adjustments for arsenic and manganese were statistically significant ($p < 0.05$), besides presenting relatively high coefficients of determination, whose values show the ability of the models to explain 79.5% and 86.3% of occurrences, respectively ($R^2As = 0.795$; $R^2Mn = 0.863$). The removal of As showed direct proportionality with the same component, manganese and iron concentrations, while only the Fe concentration was proportional to the removal of Mn. PH significantly influenced the removal of the first element, inversely proportionally, which can be explained by the more negative surface of the membrane analogously to the formation of anionic species of the component. Turbidity also negatively affected arsenic removal, an expected result due to the difficulty of moving through the membrane pores in the presence of more solids in suspension. Conductivity and apparent color were not relevant. Under any conditions, the inverse osmosis presented satisfactory results for the objective: among the 37 interactions, the lowest removal values were 75.0% for arsenic and 89.8% for manganese, and the arithmetic means obtained from the analyses were 91.1% and 98.0%, respectively.

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Interaction between brine discharge and catchment water of a seawater desalination plant: A case study of Mainis Algeria

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This study investigated the interaction between brine discharge and catchment water of a seawater desalination plant located in Mainis Algeria. The effect on the marine environment was assessed in relation to climatic variations such as speed and direction of wind and current. To achieve this, several dispersion scenarios were simulated of the release in the marine environment using the Cornell Mixing Expert System (CORMIX) code, a model employed for the analysis and prediction of contaminating or non-contaminating liquid discharges in aquatic environments. Results showed a considerable increase in salinity, which sometimes exceeded 42 g/L in the catchment water at the desalination plant. It was reasoned that there was an interaction between the discharge water and the catchment water due to insufficient dilution or deviation of the brine plume. To better exploit the results of the simulations and for a good visibility of the propagation of the brine discharge in the marine environment, three velocity cases were plotted (i.e., favorable, intermediate, and unfavorable). The planes of the plume in space considered the position of the water intake point. Alternatives and solutions to improve the performance of diffuser were proposed.

Fractionation of biologically active compounds extracted from onion skin bio-waste

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Biologically active compounds (BACs), such as polyphenols, have become an intense focus of research interest because of their perceived health-beneficial effects. They occur in a variety of fruits, vegetables, nuts, seeds, flowers, bark, and bio-waste. They have been reported to exhibit anti-carcinogenic, anti- atherogenic, anti-ulcer, anti-thrombotic, anti-inflammatory, immunomodulating, anti- microbial, vasodilatory, and analgesic effects [1]. Ultrasound-assisted extraction (UAE) of BACs possesses many advantages over conventional solid–liquid extractions in terms of higher yield, shorter extraction time and preserved biological activity of the extracted compounds Being also green and economically viable, the UAE technique has a great potential for application in the plant-based chemistry. Traditional approaches, including simple steam distillation and vacuum distillation, are adopted to BACs fractions. Generally, these methods require an increased temperature and high energy consumption. The first is inappropriate for heat-sensitive products. These methods may also result in a loss of compounds of low molecular weight, which can be removed together with the solvent during evaporation [2]. The aims of the present investigation are: (i) fractionation of BACs extracted from skin onion bio-waste using 60% ethanol and UAE by means of nanofiltration, and (ii) determination of anti-proliferative properties of the fractions after nanofiltrations.

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Mixed Polymeric Substrate RO Membranes with Nanographene-Surface Modification

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Nanographene oxide is considered one of the most promising key players in the industry of surface coating generally and specially in membrane surface modification. These modifications have been strongly emerging nowadays; to cope with the increase in demands for RO-membrane desalination units with polyamide severe deterioration and decline in performance due to higher chlorine remains to control biofouling. This problem reflects directly on increase in OPEX due to increase in RO cartridge cost and lifetime reduction. In this work mixed polymers, polysulfone and polyacrylonitrile were used as mixed polymeric substrate to enhance substrate properties and performance of polyamide layer. Modified RO/ or polyamide layer was reached using two routes: PVA layer deposition over blend membranes (P-layer) or thin film composite layer (T-layer). Over these layers, chemical grafted layer using methacrylic acid to form Poly-methacrylic acid (P-MAA) in presence of graphene oxide (G-layer). Characterization for surface modified membranes were fulfilled using BET area, SEM, contact angle, FTIR, and others for results discussion and interpretation. Successive layer by layer deposition were applied for surface modification using GO /or ZnO-glycerolate nanostructures together with tannic acid as hydrophilic additive. This shows a certain change in surface hydrophilicity which is highlighted in measurements for water contact angle together with the porosity denoted in BET area. Contact angles were found to give lowest angles for 3rd family of membranes nominated 3P to reach about 41.5°C which increases to reach 46°C for 1P to increase more when chemically grafted to reach almost the same value 49 °C and 51°C respectively. The highest salt rejection reached was about 88% for 3TG and decreases to 80% for 1TG with a corresponding permeate flux 25 and 30 LMH respectively. Optimized surface modification was reached through best permeate flux and salt rejection together with accepted chlorine resistance correlated to biofouling.

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Polyelectrolyte multilayer membranes for the separation of lactose and salts in dairy streams

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Separating lactose from milk and other dairy products is an important process in the dairy industry for many different reasons, like the production of low-lactose or lactose-free dairy products. After the separation from whey the lactose gets purified through crystallization and can be used, for example, in cheese production. Ultrafiltration is a common strategy to recover lactose from milk and dairy streams. In this process proteins and oil droplets are retained by the membrane while lactose is passing through [1]. However, dissolved minerals as calcium and phosphates will also end up in the permeate stream, impeding the lactose recovery. In this research we propose asymmetric polyelectrolyte multilayer (PEM) membranes as an addition to this process to separate lactose from these dissolved minerals. Earlier work on asymmetric PEM membranes has shown excellent retentions on small molecules, low salt retentions and high water permeability [2]. Initial experiments with membranes coated with a bottom layer of poly(4-aminostyrene) (PAS)/poly(4-styrene sulfonate) (PSS) and a thin separation layer on top of /poly(allylamine hydrochloric acid) (PAH)/poly(acryllic acid) (PAA) have shown promising results. With asymmetric coating we achieved low molecular weight cut offs while maintaining a decent permeability up to 8 $Lm^{-2}h^{-1}bar^{-1}$. Filtration of model solutions containing lactose and $CaCl_2$ show lactose retention up to 95% and calcium retentions as low as 30%. Such a membrane would thus allow simultaneous de-watering and desalination of lactose containing waste stream such as whey. Unfortunately, the separation is not yet as effective when multiple types of salts and lactose are mixed together in model solutions. We found that the pH of the feed is an important tuning parameter which allows to improve the separation in an easy way. Understanding the separation system is key to the further design of PEM based nanofiltration membranes for lactose/salt separations.

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Toward precision molecular sieving in organic solvents using metal–organic frameworks and conjugated microporous polymers

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In quest to achieve highly selective organic solvent nanofiltration membranes, we investigated the mixed matrix membranes (MMMs) containing metal–organic framework (MOF) fillers, owing to the regularity and ordered pore arrangement in the MOF structure [1]. The poor interfacial adhesion between the polymer matrix and the MOF filler was bridged by covalently grafting the MOF with poly(N-isopropylacrylamide) (PNIPAM) chains, which could then become entangled with the membranes' polymer matrix. This strategy was inspired by the natural phenomenon of strong interfacial adhesion between the calcified cementum of the tooth and the surrounding tissues provided by intermingled collagen fibers. We prepared a series of novel membranes comprising MOF nanoparticles with similar chemical and morphological properties but having various pore sizes (UiO-66–68-NH2). Morphological investigations and organic solvent nanofiltration (OSN) tests revealed that membranes with PNIPAM-grafted fillers do not suffer from the formation of pinholes at the filler–matrix interface that are detrimental to the filtration performance. The OSN test revealed the increase in the flux and molecular weight cut-off values with increasing MOF aperture size which confirm the key role that internal MOF pores play in the filtration. As for the disordered system, we studied the application of conjugated microporous polymer (CMP) as membrane materials for OSN. CMP membranes have an amorphous structure with rigid and permanent pore structure, as well as great chemical stability, which is suitable for OSN applications. In both ordered and disordered systems, the experimental nanofiltration results showed an excellent match with a predictive model of nanofiltration built around the premise of liquid transport through the highly ordered pores. This finding is particularly surprising for the CMP membranes as the overall structure is amorphous, but they still maintain the local ordering which facilitates the mass transfer.

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Graphene based membranes to massively reduce energy in the oil, gas and chemical industry

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Graphene, a two-dimensional single atomic carbon layer, is an attractive material for thin film and membrane applications. Graphene oxide (GO) membranes made by assembling small GO flakes have demonstrated great potential in gas and liquid separation. For upscale applications, GO membranes in hollow fibre geometry are of particular interest due to the high surface area per volume and easy-assembly features at module level. In our research group, ceramic hollow fibres (CHFs) have been developed for various membrane processes. They are an excellent substrate candidate for GO membranes due to its robust mechanical property and inert chemical nature which enables them to work under extreme conditions. However, our studies revealed that GO membranes on CHFs are unstable in dry-state, mainly due to drying-related shrinkage [1]. We reserved graphene oxide hollow fibre (GOHF) membranes in water after initial drying to avoid further shrinkage and the formation of defects. Though such a strategy worked well to preserve the microstructure of GO-membranes, it limits the GO-membranes only to water-related applications. In another approach, GOHF membranes were stabilized by using a porous poly(methyl methacrylate) sacrificial layer, which created a space between the HF substrate and the GO membrane that allows stress-free shrinkage. Defect-free GOHF membrane was successfully determined and the membrane was stable in a long-term gas tight stability test [1]. In this study, in order to employ the developed GO-membranes for solvent permeation, a post treatment of the stabilized GO hollow fibre membranes is being carried out which induces mild reduction that would lead to some controlled pore formations. These modifications enhances the performance of GOHF membranes in solvent nanofiltration while still maintaining the molecular sieve property of GO membranes. Solvents such as ethanol, toluene and benzene containing natural dyes with different MWCOs are being tested and will be reported at the conference.

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Interactive Organic Solvent Nanofiltration Database and Prediction Tools

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Niche membrane technologies, such as organic solvent nanofiltration (OSN), offer better energy and operation cost reduction than conventional separation methods. However, despite their many advantages, their industrial implementation is hindered by small, specialized datasets and the limited availability of predictive models. In this presentation, we show the advancement of the one-year-old OSN Database [1, 2], an open access online platform to quickly access literature, data, and predictive tools. Currently, the OSN Database contains more than 7000 datapoints measured in 55 solvents and solvent mixtures. The database contains data related to solute and solvent parameters, pressure, temperature, flowrate, concentration, process configuration and structural information for both the solvent and solute. The database has an extensive visualization diagram with 18 built-in and customizable filters. Each point in the dataset is directly linked to the original publication, which enables tracking their source. The website features chemical structure search options. The presentation will highlight the dataset's and database's most significant parameters and features.

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Organic liquid mixture separation using fluorinated covalent organic framework (COFs) supported ceramic hollow fibers

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Organic liquid separation is an essential industrial process for recycling valuable organic solvents from organic solvent mixtures. To achieve the goal, some traditional separation processes such as distillation, etc can be employed, but consume 10%-15% of the world's energy [1]. Alternatively, membrane processes can separate organic liquid mixtures more energy effectively [1]. Covalent organic frameworks (COFs), a crystalline porous polymer made of regularly extended and covalently connected network is a promising candidate for an exquisite selective layer due to its ordered pore structure and uniform pore channels which are optimal for the transportation of solvent molecules [2]. However, COFs have not been extensively applied in the field of membrane separation yet. The aim of the research is to develop a new organic liquid mixture separation membrane which consists of a fluorinated COF layer with high crystallinity and optimum pore size supported by ceramic hollow fibers (CHFs) which can provide excellent chemical stability and high pressure resistance. A new COF TpDABF was synthesized by the polymerization of 1,3,5- trimethylresorcinol (Tp) and 2,5-Diaminobenzotrifluoride (DABF), and the crystallinity was confirmed by XRD. A ultrathin film of TpDABF was fabricated onto the top of CHFs support through interfacial polymerization (IP) or in situ growth for a thin film composite (TFC) membrane. Compared with a conventional COF TpPa, this new COF showed improved hydrophobicity with the average water contact angle of 92° due to the presence of the hydrophobic side groups -CF3 in DABF, leading to a higher permeation of non-polar solvents. Meanwhile, the introduction of the side groups can also slightly reduce the pore size which should be smaller than the reported pore size of 1.8 nm for COF TpPa [3]. This kind of hydrophobic COF membrane showed a good performance in the separation of various organic liquid mixtures.

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Development and characterization of high molecular weight and high-osmotic pressure draw solute for forward osmosis applications

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Forward osmosis (FO) is an emerging technology for seawater and brackish desalination, wastewater treatment, and other applications such as food processing power generation, protein and pharmaceutical enrichment based on osmotic pressure as driving forces. FO utilizes the osmotic pressure difference of solutions across a semipermeable membrane to draw water from a dilute feed solution (FS) to a concentrated draw solution (DS). When using synthetic draw solutions, it is critical to recover the draw solute in a subsequent pressure-driven membrane process (e.g. reverse osmosis, nanofiltration) in which pure water is produced. In the study, we fabricated a novel draw solute by modifying water-soluble poly(styrene sulfonate) (PSS) using poly(3,4-ethylene dioxythiophene), a high electrical conductivity polymer. The performance of the aqueous solution of the new draw solute was compared with the aqueous solution of NaCl of the same electrical conductivity (50 mS) in FO experiments using commercial CTA-W (flat sheet) membranes. The experiments were performed with two types of feed solution: distilled water and synthetic brackish water (7 mS). The new draw solute's water fluxes were slightly higher than with NaCl for the same feed solution and ranged at 4 - 5 L/m^2.h. However, the reverse salt flux of the new draw solute was more than 30-times smaller than that of NaCl. More importantly, we demonstrated the regeneration of the new draw solute using commercial ultrafiltration (UF) membrane (PS35), with the solute rejection exceeding 96%.

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Preparation of Electrospun Forward Osmosis Membranes for Clean Water Production

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Clean water and clean energy production operations have devoted great attention due to the increasing concern on global warming, water scarcity, and depletion of natural resources. Solar power-based desalination systems are suggested as an alternative sustainable energy production technique to overcome these environmental problems [1]. Recently, researchers have proposed designing an innovative hybrid concentrated solar energy (CSP) system including a forward osmosis (FO)-based desalination process which recovers the waste heat for producing electricity. The main goal of this system is to create an energy-efficient prototype of the CSP coupled to FO desalination using both a current and an innovative power cycle while optimizing each constituent of the complete system [2]. FO systems benefit from the naturally generated water diffusion from a low osmotic (feed) solution to a high osmotic (draw) solution through a semi-permeable membrane. The main disadvantage of FO is the large contribution of internal concentration polarization, induced by inefficient membrane supports, which greatly decreases the driving force for water transport resulting in low transmembrane water fluxes [3]. In order to improve the structural properties of the membrane supports, a two-step membrane production method will be investigated. Firstly, a mechanically stable highly porous support will be prepared via the wire electrospinning technique. In the next step, the actual FO-membrane will be made by coating the open support with a thin selective layer using the layer-by-layer coating technology using polyelectrolytes as e.g. PDADMAC, PSS, or PAH. These layers which are stable even at the high salt concentrations provide the selectivity not only for water over the draw solution components but also for water over the NaCl present in the feed solution. In the end, the developed FO membrane will be merged with an CSP, power cycle with heat integration and draw recovery system to a complete drinking water production process.

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New applications of biomimetic hollow fiber forward osmosis membranes

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Forward osmosis has been considered as a break-through in membrane technology due to the reduced energy consumption of the FO process and the low fouling propensity of FO membranes. The trade-off between water flux and reverse salt flux, the suppression of the osmotic pressure difference between the feed and draw solutions by internal concentration polarization, and the underestimated contamination of FO membranes by potential foulants predominantly present in the feed have reduced the euphoria around FO. However, FO is useful for specific niche applications in the treatment of different water streams in the pharmaceutical and food sectors but also as a pre-treatment for desalination. Among those FO membranes commercially available today, the biomimetic hollow fiber membranes with incorporated aquaporins manufactured by Aquaporin A/S (Denmark) feature a high membrane surface area at a low foot-print and a superior performance characterized by a specific reverse salt flux of as low as 0.15 g/L. In this contribution, we report on the success and challenges involved in some new applications for these biomimetic HFFO membranes. The use of functionalized magnetic nanoparticles as a recyclable draw solute, the application of FO for the up-concentration of lactoferrin in whey, the recycling of chromium from electroplating wastewater, and the effect of different cleaning strategies for the re-use of HFFO membranes in the treatment of a fermentation broth for vaccine purification are examples, which underline the versatility of the FO process. The results of the corresponding FO processes will be supported by the classical characterization of the FO membrane performance by the measurement of water flux and reverse salt flux. Furthermore, we introduce the analysis of the surface zeta potential by the streaming potential method as a potential tool for the at-line monitoring of membrane fouling and the verification of the efficiency of different cleaning agents.

Membranes for pressure retarded osmosis as a new way for sustainable power generation from salinity gradients

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Nowadays, the current global energy demand is extremely high and it far exceeds the capacity for sustainable production. Moreover, now more than ever, it is crucial to become independent from fossil fuels as a way to decrease or even reverse the climate changes observed on the world panorama. Pressure retarded osmosis (PRO) is a promising technology for sustainable power generation from salinity-gradient energy, which is obtained when waters with different salinities are mixed. 0.75 kWh of energy is released when 1 m^3 of fresh water flows into the saline water [1]. The osmotic energy is converted into hydraulic energy by driving a hydro-turbine with the pressurized water. This waster then, permeates through a PRO membrane due to an osmotic gradient from the diluted “feed solution” into the more concentrated “draw solution”. Despite the potential of this technology, the development of PRO was hindered by the lack of a suitable membrane [2, 3]. In this work, the selective and support layer of commercially available thin-film composite membranes was modified with different methods in order to tune the characteristic PRO parameters: water permeability (A), salt permeability (B) and structural parameter (S). The aim of this work is to increase the A parameter, decrease the B and decrease the S thanks to the selective and support layer modifications. As of our knowledge, this is the first time when two sides of the same membrane are modified. The inspiration for this double side modification was the possibility to change the internal concentration polarization profile inside the membrane. Furthermore, the performance of pristine and modified PRO membranes was analyzed by a developed mathematical model designed to simulate the PRO process more closely in comparison to conventionally used Forward Osmosis (FO) or Reverse Osmosis (RO) models. The developed method helped predicting the performance of modified membranes.

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Development of a tool for understanding and design of forward osmosis processes

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Forward osmosis is a promising membrane process that has recently received a lot of attention from researchers. Most publications have focused on the draw solution, fouling, energy consumption and recovery system, and only a few on modelling/simulation and techno-economic analyses. In order to convince decision makers from industry of the yet little used technology, there is a high need for diverse pilot scale trials and demonstration projects [1]. The authors' research aims to develop an engineering tool for the design of forward osmosis processes in order to accelerate applied research and to create data for economically promising studies. In this particular presentation the authors present their progress in the research made so far, wherein they managed to numerically model the thermodynamic properties of known highly concentrated single electrolyte solution applying Bromley's equation over a concentration process. Furthermore, approaches are discussed on how to simulate substance systems of unknown composition. Via the preparation of concentrates and subsequently dilution series for the determination of the solvent activity, a correlation between the osmotic pressure and the recovery rate can be obtained. Different processes for the preparation of representative concentrates like vacuum rotation evaporation and laboratory scaled forward osmosis are argued based on the results that were measured. Moreover, approaches are presented, including the use of the osmotic pressure correlation in the simulation of a forward osmosis process. An outlook on future calculations is given, that comprises membrane and module parameters which influence the mass transfer process to further compare modules available on the market under variable process conditions and process solutions. The tool will thus be able to predict observable performance-determining parameters such as recovery rate and rejection of forward osmosis for single membrane modules but also for a whole membrane module cascade and thereby design the process.

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Thermo-responsive PAG Based Commercial Draw Solutions for Forward Osmosis

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Forward osmosis (FO) is a promising technology for the efficient separation of water from a feed solution at low operating costs. Using the osmotic pressure difference between the draw solution and the feed solution water is pulled from the feed side into the draw solution which is subsequently diluted. For desalination of seawater, the draw solution must be carefully selected to provide sufficient high osmotic pressures, low reverse solute flux, and efficient regeneration. Thermo-responsive polymers with a lower critical solution temperature (LCST), especially polyalkylene glycol (PAG) based on copolymers with hydrophilic polyethylene glycol and hydrophobic polypropylene glycol units, are interesting draw solutions because they can be regenerated using low-grade heat. Furthermore, by adjusting the ratio between the two units, the LCST and other properties can be adjusted. Several different PAG-based copolymers are commercially available, and in this study, some promising candidates are experimentally evaluated and compared. Three types of block copolymers Pluronics from BASF SE (Ludwigshafen, Germany), PE 6400, RPE 1740, and L-35, and two different types of random copolymers from NOF Corporation (Tokyo, Japan), UNILUBE 50MB-2 and POLYCERIN 55GI-2601, were selected and tested as a draw solution in a laboratory FO setup using two commercial FO membranes - Toray (Tokyo, Japan) and Aquaporin A/S (Kongens Lyngby, Denmark). In comparison to a conventional sodium chloride draw, the polymer draw solutions generated lower water fluxes but also lower reverse solute fluxes and lower specific reverse solute fluxes at similar osmotic pressures. Furthermore, all polymer solutions showed great regeneration ability at temperatures below 100 ºC and they are therefore interesting draw solutions for FO systems if low-grade heat is available.

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Recovery of Palladium Through Anion Exchange Membranes Using Electrodialysis

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The recovery of platinum group metals (PGMs) such as Palladium (Pd) has attracted much interest due to their common applications as jewelry, catalysts and drugs. However, high demand and scarce reserves have raised concerns about their sustainability in such applications. Therefore, it is critical to research the efficient separation of Pd from its solution. This study aims to investigate if membrane technology can be used to extract and retrieve dilute concentrations of Pd from hydrochloric acid-based solutions in a cost-efficient way using electrodialysis (ED). The dilute Pd complexes with chloride (~1200 mg/L) separation from industrial mixed-ion solution was studied using tailor-made anion exchange membranes (AEMs) involving quaternary ammonium bases. Salts of quaternary ammonium bases are effective extractants for PGMs [1]. The effects of the polymer concentration, ion exchange capacity and water uptake of the membranes were determined by the rate of the metal transport. It was shown that an effective recovery of Pd complexes up to 60% was achieved with different tailor-made AEMs after 3 hours of ED process. Pd transport through AEMs increased with increasing polymer concentration. However, further increment in polymer concentration caused a decreasing trend in the recovery process. Desorption of Pd to the concentrate compartment was the slowest for AEM possessing the highest IEC. Water transport was also induced more with a higher amount of fixed charged groups, which was not desired for the up-concentration process of PGM. After five months of exposure to acidic process solution (pH < 1), the resulting membranes displayed excellent acid stability behavior. We conclude that ED is a promising technology for Palladium recovery and enables large room for future research.

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Carbon capture from water bodies by Membrane Capacitive Deionization (MCDI)

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To meet the Paris Climate agreement, it is very important to develop carbon neutral technologies to limit the increase in the global average temperature and cut down the CO2 emissions. Membrane Capacitive Deionization (MCDI) is a promising technology to remove CO2 from natural process streams like oceans and rivers. The CO2 concentration in the oceans and rivers (as $CO_2 + H_2O \leftrightarrow H_2CO_3 \leftrightarrow H^+ + HCO_3\text{-} \leftrightarrow 2H^+ + CO_3^{2-}$; 50-100 mg/L) is significantly higher than in air (0.77 mg/L). The natural water bodies containing carbon in the form of bicarbonates form the feed solution for MCDI. Direct capture of CO2 from the atmosphere, e.g. by adsorption on amines, requires processing of large volumes of air and has high energy demands (240-400 kJ/mol(CO_2)). We show that by extracting CO2 from model mixture of brackish water using MCDI, energy requirements and the footprint of the capture system can be significantly decreased (40-70 kJ/mol(CO_2) and ~3x smaller, respectively). MCDI is an electrically-driven separation technology to remove low concentrations (< 1 wt.%) of ions. MCDI cells are composed of low-cost activated carbon electrodes and have the advantage of high energy efficiency, enabling capture at room temperature and atmospheric pressure. By applying current over the cell, ions are removed from the liquid feed and stored in the electrical double layer in the pores of the carbon electrodes. During this step, energy is temporarily stored in the electrodes. By reversing the current, ions are desorbed, creating a concentrated stream, and the stored energy is released. The feasibility of MCDI on a large scale depends on the efficiency, cost and scalability of the system. We present the key experimental results and preliminary techno-economic evaluation of MCDI for CO_2 capture from natural water streams.

Valorization of saltwork brines for the production of acid and base using electrodialysis with bipolar membranes

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Valorization of brines, usually considered as wastes, has become an alternative to extract critical and valuable raw materials, but also for energy recovery and generation of reactants. Within this context, the H2020 SEArcularMINE project aims to recover from saltwork brines Mg(II), Li(I) and some trace elements (B(III), Co(II) and Sr(II), among others), all of them listed as critical raw materials by the European Union [1]. Due to the fact that the technologies devoted to the recovery of the above mentioned elements require acid and bases, the SEArcularMINE project aims to produce them in-situ (HCl and NaOH) from exhausted brines (free of Mg(II) and Ca(II)) using Electrodialysis with Bipolar Membranes (EDBM), promoting in this way a fully circular scheme. The aim of this work is to evaluate the performance of a laboratory EDBM unit (equipped with membranes supplied by SUEZ-WTS) for the production of NaOH and HCl, mimicking conditions (in terms of solution composition and flows arrangements) expected within the treatment chain. Moreover, experiments were carried out with real pre-treated brines (Mg(II) and Ca(II) < 10 mg/L), and the transport of the above-mentioned trace elements was studied in detail. All the experiments were performed in a lab-scale unit (Electromat MkI ED STACK) with an active area of 0.028 m2 operated at 300 A/m^2. In order to compare experiments, several performance parameters were calculated, such as the specific energy consumption (SEC), current efficiency and acid and base purities. Experimental results showed the possibility of producing NaOH and HCl (>1 M), with SEC values ranging between 2.05 and 2.39 kWh/kg NaOH and current efficiencies higher than 70%.

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Coupling electrodialysis with membrane contactor: a key approach to recovering nutrients and volatile fatty acids from food wastes towards higher concentrations and selectivity

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About one-third of the food produced for human consumption is wasted worldwide. Besides, the rapid depletion of fossil fuels and mineral reserves, together with their adverse environmental effects during their recovery and processing, impact global economic and environmental sustainability. To tackle this worldwide issue, efficient technologies to sustainably recover nutrients (NPKs) and volatile fatty acids (VFAs) from bio-based streams such as food wastes from agricultural streams are essential for closing the loop of circular economy. Coupling electrodialysis (ED) with membrane contactor is a key approach to recovering NPKs and VFAs from food wastes. Electrodialysis separates species mainly based on their ionic charges, it produces relatively higher ionic concentrations, and lower water-content products, which are simultaneously cumbersome to achieve through Nanofiltration (NF) and Reverse Osmosis (RO). In this study, ED is applied to achieve at least 2-5-fold concentration of NPKs suitable for fertilizer applications as K^+, NH_4^+, NO_3^-, SO_4^{2-}, PO_4^{3-} respectively, corresponding to 90-98% recovery. Likewise, 70-90% VFAs have been recovered through ED, with acetate and butyrate concentrations reaching 22.1g/L and 6.1 g/L (i.e. 2.5-3.2-fold concentration), respectively. The membrane contactor is coupled with ED to boost the selective separation of VFAs from the electrodialysis concentrate, thus, separating more than 70% of the VFAs with over 98% of the NPK nutrients remaining intact as concentrate. Nevertheless, the operational parameters, feed conditions, and configurations greatly influence the process performance; therefore, these impactful factors are continuously tested to enhance the selectivity, recovery, energy consumption, and concentration factor of the NPKs, and VFAs.

The concentration, potential, and pH profiles along a diluate channel of a miniaturized electrodialysis unit

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Electrodialysis is an electro-membrane separation process traditionally used to remove small ions from processed water solutions. Desalination of the sea or brackish waters is a typical application of electrodialysis. Ion-exchange membranes constitute a significant part of the electrodialysis units because they provide selectivity toward the transport of the ionic components. DC voltage connected to a stack of membranes drives the movement of ions through the respective membranes, which produces two output water solutions. The solution characterized by a high ionic concentration is referred to as concentrate. The solution of low ionic concentration is denoted as diluate. This work focuses on the experimental analysis of electrodialysis in self-constructed milifluidic flow-through systems allowing to reconstruct 3D profiles of relevant quantities, such as salt concentration, pH, potential, temperature, etc. We performed an in-depth experimental study investigating the effect of the connected electric current load on the spatial evolution of the quantities above. We found that (i) the concentration of the desalted solution changes both in the lateral and vertical direction (concerning the channel), (ii) interestingly, only minor differences in the concentration were seen in the transverse direction, (iii) the concentration profile suggests an essential role of natural convection in the ionic transport form solution bulk to membrane surfaces, (iv) the pH changes along the channel indicate water-splitting reaction at the anion-exchange membrane [1], (v) the potential drop across the membranes of the diluate channel increases along the channel and corresponds to the level of desalination, (vi) minimal potential diffarences found along the channel or in the vertical direction, (vii) the potential drop ac across the diluate channel is monotonous. The obtained data underline the importance of natural convection [2] and spatial distribution of ionic current (and potential) on the overall ionic transfer in electrodialysis.

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Effect of ion exchange on the structure of heterogeneous ion-exchange membranes

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Ion selectivity of ion-exchange membranes originates from the electric field generated by membrane fixed charges and the internal membrane structure, providing efficient interaction between the fixed charged and mobile ions. The electric forces determine what type of ions can pass through such membranes. However, the electric forces are also responsible for membrane swelling and de-swelling when immersed in water solutions of various electrolytes. The degree of swelling is influenced by many factors such as ionic strength, pH, type of counterions, and many others. To understand the effect of the counterion type on swelling and shrinkage of heterogeneous ion-exchange membranes, we employed micro-computed tomography to reconstruct a spatial [1] and surface [2] membrane composition. We sequentially soaked heterogeneous cation-exchange membrane in solutions of LiCl, NaCl, KCl, $MgCl_2$, and $CaCl_2$. Our analysis showed the ion exchange is accompanied by profound changes in the spatial membrane composition. The results can be summarized in the following points: (i) the membrane reaches the most significant volume in LiCl solution and decreases in the following order of solutions: NaCl, $MgCl_2$, KCl, and $CaCl_2$, (ii) the swelling and shrinkage are reflected on the membrane surface at which the resin occupies the largest surface area for $CaCl_2$ solution, (iii) the swelling and shrinkage is history-dependent and to a large degree irreversible. Our study indicates that the change in the type of the counterions in the membranes may lead to the creation of large pores displaying poor ion selectivity. This effect may, in turn, result in the worsened performance of the membrane in processes such as electrodialysis or electrodeionization.

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Ionic transport properties of charged mosaic membrane prepared from fabric structure of poly (vinyl alcohol) based charged-fiber

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A charged-mosaic (CM) membrane is made up of parallel arrays of cation and anion exchange elements passing through the membrane. The membrane shows negative osmosis and is much more efficient in electrolyte transport than in non-electrolyte transport [1,2]. In this study, novel charged-mosaic (CM) membrane was prepared from fabric structure using two kinds of poly (vinyl alcohol)(PVA)-based fibers. The fibers were made from the anionic and cationic block-type PVA: poly (vinyl alcohol-b-sodium styrene sulfonic acid) and poly(vinyl alcohol-b-vinyl benzil trimethyl ammonium chloride), respectively. The fabrics were heat-pressed in order to make non-defects film and were crosslinked with glutaraldehyde (GA) to prepare fabric structured CM membranes. In the photograph of the prepared CM membrane (CMF-1) stained with methyl orange and methyl violet, yellow and violet parts indicated the positively- and negatively-charged domains of the CM membrane, and the charged mosaic structure of woven fabric was confirmed. In the volume flux experiments, non-charged PVA membrane, showed positive osmosis. On the other hand, the CM membrane (CMF-1) indicated a negative osmosis flow, which is a phenomenon peculiar to CM membranes [1,2]. The desalination performance of CMF-1 was performed by piezodialysis experiment using 500 ppm NaCl at 0.3 MPa of applied pressure to confirm the possibility of desalination using the membrane. In the piezodialysis experiments, the concentration at the high-pressure side decreased while that at the low-pressure side increased. This is the opposite phenomenon of piezodialysis using reverse osmosis membrane with water permselectivity. The piezodialysis test using CMF-1 indicates that the desalination was done using the membrane. Therefore, the charged mosaic membranes prepared from fabric structure has potential application to desalination of salt solutions with low concentrations.

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Relationship between anion permeability and membrane resistance in electrodialysis using monovalent selective anion exchange membrane

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Electrodialysis (ED) using ion exchange membranes (IEMs) is applied to separate and concentrate of ions. In the application of ED, it is important to elucidate the permeability of ions inside IEMs. In this study, to estimate ionic transport properties in monovalent ion selective anion exchange membranes (MS-AEMs), the membrane resistance (Rm), water content (H), ionic distribution (K), ion exchange capacity (IEC) and the ion concentration and ion mobility in the membrane were measured for two types of MS-AEMs (Neosepta®ACS-8T, Neosepta®AXP-D) (ASTOM, Corp., Japan) using various kinds of electrolyte solutions. In a mixed solution of iodide and chloride ions, iodide ions exhibited higher K than chloride ions even though iodide ions content in the solution was 50 %, suggesting that the affinity of iodide ions for the two IEMs was higher than chloride ions. K values of both iodide and chloride ions for AXP-D were higher than those for ACS-8T. AXP-D had lower water content than ACS-8T though the two membranes had almost the same IEC. Therefore, the ionic concentration in AXP-D, which is proportional to the value of IEC divided by the water content, was higher than that in ACS-8T. This will be one of the reasons why AXP-D had higher K value than ACS-8T. In the case of immersing the membranes into KI solution, Rm of both the membranes was higher than that in the case of immersing them into KCl solution even though iodide ions exhibited higher K than chloride ions. This indicates that the mobility of iodide ions in the two membranes will be much lower than that of chloride ions.

Evaluation of power generation characteristics in reverse electrodialysis power generation using novel profiled ion-exchange membranes

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As one of renewable energy, salinity gradient energy (SGE) generated by mixing two salt solutions with different concentrations has attracted [1]. Reverse electrodialysis (RED) can convert SGE to electric power by the selective permeability of the membranes and concentration differences between solutions as the driving force. In conventional REDs using flat ion-exchange membranes (IEMs), spacer nets exist to hold the flow paths between the membranes, which results in high resistance in the solution flow paths. Profiled (PF) IEMs have a concave-convex PF structure pattern on membranes surfaces, and the PF pattern keeps intermembrane flow paths instead of spacer nets. Therefore, except for the spacer nets by using PF IEMs, the power output will be expected to increase due to the reduction of resistance in the flow paths of the low-concentration solution in RED stacks [2]. Additionally, the use of PF IEMs will be expected to reduce pressure drop and improve net power density [2]. In this study, PF IEMs were prepared by using homogeneous flat IEMs: A-2/C-2 (ASTOM Corp., Japan) as base IEMs of PF processing for hot press method to form PF pattern with a nearly uniform membrane thickness. The power generation characteristics of RED stacks with PF IEMs were compared with RED stacks with flat IEMs (A-2/C-2). RED stack with PF IEMs had lower internal resistance than that with flat IEMs; hence, the stack with PF IEMs showed 145% higher the maximum power density (PDgross) compared to that with the flat IEMs. Further improvement of PDgross will be expected through optimization of the PF structure pattern by fluid analysis.

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Estimation of power generation of pilot-scale reverse electrodialysis (RED) based on performance of prototype RED stack

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Reverse electrodialysis (RED) is the emerging technology that can obtain electrical energy by mixing two different concentrated solutions such as seawater and river water. A RED stack consists of alternately stacked anion exchange membranes (AEMs) and cation exchange membranes (CEMs) with flow channels for solution supply. There have been many studies on RED performance using small size stacks; however, there are few studies about power generation performance using pilot-scale reverse electrodialysis stacks [1,2]. In order to investigate the feasibility of a commercial RED plant, we estimated the power output at a pilot-scale RED stack (6000 cell-pairs, the total membrane area, 10800 square meter) based on experimental data of a prototype RED stack (300 cell-pairs, the total membrane area, 90 square meter). The stacks had 200 µm of the spacer thickness as both the high- and low concentration flow channels, Pt-coated Ti as the electrodes, and 5 wt% Na_2SO_4 solution as the electrolyte solution to convert ion transport into electric current. Because RED performance depends strongly on the resistance of AEM and CEM pairs, we used the low resistance AEMs / CEMs (A-2/C-2, ASTOM Corp., Japan), and also the standard AEMs / CEMs (Neosepta® ASE / CSE, ASTOM Corp., Japan) as a control. 0.5 M and 0.017 M NaCl solutions were used as the model solutions of seawater and river water. Based on the power output of the prototype stack and the bench-scale RED stack [3] that used many kinds of IEMs, we estimated the power outputs of a pilot-scale RED stack using real seawater and fresh water.

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Donnan dialysis performance of monovalent ion selective hollow fiber cation exchange membranes prepared by plasma graft polymerization

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One method for recovering NH^{4+} from wastewater is the Donnan dialysis (DD) process using cation exchange membranes (CEMs) [1]. However, it requires large membrane area due to its slow dialysis rate [2, 3]. Hollow fiber (HF) membranes have the advantage of larger membrane surface area per unit module volume compared to flat membranes. In this study, monovalent ion selective permeable HF CEMs were prepared by forming positively-charged on the membrane surface, and DD performance in separation of NH^{4+} and Ca^{2+} ions was performed. Sulfonated polyethersulfone (SPES) was dissolved in N, N-dimethylacetamide to prepare a 20 wt% SPES polymer solution. Porous polyvinylidene fluoride (PVDF) HF membranes were dip-coated in this solution and allowed to dry naturally. The membrane was then irradiated with plasma to generate radicals, and then was immersed in a 20 wt% vinylbenzyl trimethyl ammonium chloride (VBTAC) monomer solution for a predetermined time to fabricate a monovalent ion selective HF-CEM by forming the positively-charged on the membrane surface. After that, a mini-module was fabricated using the prepared HF-CEM, and DD experiments were performed. FS (1.0 mM NH_4Cl and 1.0 mM $CaCl_2$ 500 mL) was circulated through the outside of the HF membrane, and DS (1.0 M NaCl 100 mL) was circulated through the inside of the HF membrane. The permeation flux Ji and the monovalent ion permselectivity coefficient PNH4Ca were calculated from the concentration change with time of each ion in FS. Optical microscope image of the membrane indicated that a cation-exchange layer (SPES) with thickness of 3.54~30.4 µm was formed on the PVDF support. DD experiments showed that PNH_4Ca of HF-CEM with positively-charged layer was found to be 2.09, while that of HF-CEM without the layer was found to be 1.30. P NH_4Ca was improved by a factor of about 1.7 by forming positively-charged on the membrane surface.

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Design of selective electro-driven membranes for ion resource recovery

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Selective ion transport is fundamental in many areas of science and technology for purifying ions of interest. In particular, tailoring high-precision separation materials would enable fit-for-purpose recovery of pure water, extract minerals and capture energy from a complex solution environment such as wastewater, seawater, salt lakes, and flow batteries [1]. Driven by the European directives of circular economy, the development of mefficient separation technologies are urgently demanded for ion resource recovery. Electro-driven membrane processes using ion exchange membranes show promise in promoting the selective transport of target ions under electrical field [2,3], but still faces the fundamental challenge to achieve high precision/selectivity via electrostatic interactions, among ions of similar electrochemical properties. Thus, this work uses several examples of in-house designed ion exchange membranes with superior properties promoting target ion transport. On one hand, homogenous ion exchange membranes were preprepared via a newly-developed dynamic layer-by-layer (dLBL) method utilizing the cation-π interaction between dopamine chemistry and electrolytes. The resulting membrane demonstrated thin selective layer with up to 6.5 bilayers of alternating charged functional moieties, producing much more homogenous morphology and enhanced stability in repeated separation experiments. The selectivity towards monovalent ions such as K^+ from a mixed solution was attributed to the combined mechanism of ion sieving, electrostatic and affinity interactions between the target ion and functional sites on the membrane surface. On the other hand, heterogenous ion exchange membranes were designed by incorporating 2D porous materials (e.g., MOF) into the membrane matrix. By fine tuning the fabrication conditions (e.g., types and dosage of charged monomers), a series of MOF-based membranes were obtained with high selectivity towards target monovalent ions such as Cl^- vs PO_4^{3-}, and Li^+ vs Mg^{2+} [4,5]. Finally, the main design principals of target ion selective membranes are summerized, coupled with in-depth analysis on ion transport mechanisms.

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Synthesis and fabrication of low-cost proton exchange membranes for artificial photosynthesis

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Proton exchange membranes (PEMs) are semipermeable membranes with a high selectivity towards transporting protons. An ideal proton exchange membrane only transports protons and blocks all other ions and compounds. The main application of such membranes is the use in a PEM fuel cell to generate electricity. For such applications, the commercial Nafion membranes are the most popular due to their high chemical resistance and high proton conductivity. This research looks into the synthesis of low-cost proton exchange membranes for artificial photosynthesis. This innovative process in which the membrane will be incorporated is being developed by a European multidisciplinary team in the context of the Hysolchem project (www.hysolchem.eu). The process happens in an electrochemical cell consisting of two chambers. In the oxidation chamber, organic pollutants in wastewater are oxidized into CO_2 at the anode, generating protons and electrons. These electrons are drawn to the reduction chamber where CO_2 and N_2 from the environment are converted into fuels (methanol, ethanol) and ammonia, respectively, via a photocathode. A PEM should separate the two compartments while only transporting the protons from the anode to the cathode chamber, since the reduction reaction needs the protons that are generated at the anode. The commercial Nafion membrane is not suitable for this application due to its high cost and relatively high methanol permeability. This research has the ambition to synthesize crosslinked sulfonated membranes from low-cost materials. A crosslinked sulfonated polyether ether ketone membrane and a polysulfon based membrane are synthesized and characterized (FTIR, IEC). Moreover, different performance tests are performed, such as the performance in the electrochemical cell, proton selectivity and methanol permeability. The membranes have a material cost at least 10 times lower than Nafion and a similar performance.

Effect of operational parameters in the production of acid and base using a electrodialysis with bipolar membranes at pilot plant scale

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One of the most crucial challenges is to ensure a sustainable and reliable access to water. In order to provide it, research is moving towards the development of innovative management solutions and alternative water resources, in agreement with Water Framework Directive, Circular Economy and European Union (EU) Green Deal packages. In this sense, the EU-funded WATER-MINING project aims to exhibit and validate innovative next-generation water resource solutions at the pre-commercial demonstration-scale in order to provide water management and recovery of valuable resources from alternative water resources [1]. Different layouts for urban wastewater treatment and seawater desalination are proposed. In particular, the framework of this work is Lampedusa (Italy) seawater reverse osmosis (SWRO) desalination plant. The aim of WATER-MINING project in this location is the production of fresh water and the recovery of salts ($Mg(OH)_2$, $Ca(OH)_2$, NaCl and Na_2SO_4)by the integration of different technologies. In order to promote a fully circular scheme, electrodialysis with bipolar membranes (EDBM) will be used to produce the chemicals needed in the process (NaOH and HCl) from exhausted brines under the application of electricity. A novel EDBM pilot plant scale has been installed and operated in Lampedusa SWRO facility. The EDBM plant has a commercial Fumatech stack of 6.4 m^2 effective area. The performance of EDBM for one single pass under different flowrates (2-8 $L \cdot min^{-1}$) for the acid, base and saline channels, and two current densities (200 and 400 $A \cdot m^{-2}$) have been analysed in terms of specific energy consumption (SEC) and current efficiency (CE). Results showed that by increasing the flow-rates, generation of HCl and NaOH slightly increased. For example, ΔH^+ shifted from 0.65 to 0.77 $mol \cdot min^{-1}$ when flow-rate increased from 2 to 7.5 $L \cdot min^{-1}$ at 200 $A \cdot m^{-2}$. Under these conditions, SEC decreased (from 1.45 to 1.18 $kWh \cdot kg^{-1}$) while CE increased (from 81.7 to 96.7%).

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Assessment of reverse electrodialysis membranes performance in the presence of divalent ions

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The ever-increasing demand for energy, mainly due to global population growth, coupled with the need to combat climate change is driving policies that encourage the development and implementation of novel and sustainable renewable energies [1]. Membrane-based technologies such as reverse electrodialysis (RED) hold great promise for harnessing the energy released from a saline gradient (SGE), also known as Blue Energy [2]. Similarly, the discharge points of Wastewater Treatment Plants (WWTP) effluent into the sea represent a niche of opportunities for SGE extraction. In addition, the procurement of SGE in coastal WWTPs can contribute to promoting the reclamation of wastewater effluents for further reuse for non-potable purposes such as agricultural irrigation, industrial uses and street cleaning in urban uses [3]. Whereas regeneration strategies are currently being constrained by the additional energy consumption of tertiary treatment. In this way, the "LIFE 3E Environment-Energy-Economy" project aims to address the water-energy nexus by integrating RED electrochemical technology for the production of SGE together with water reclamation. The ion exchange membranes that make up the RED systems are key components, as they determine the performance and overall efficiency of the process. Laboratory testing of Fumasep® FKS-75 and Fumasep® FAS-75 reinforced membranes has shown a reduction in gross power density (GPD) when using model water solutions versus non-reinforced membranes. A maximum GPD of 1.5W/m^2 has been achieved operating in continuous mode in a RED stack fed by pure NaCl water streams (0.02-0.5M). In fact, the presence of divalent ions in the synthetic wastewater–seawater streams (0.017-0.49M NaCl, 0.0024-0.06M $MgCl_2$, 0.0015-0.011M $CaCl_2$ and 0.0014-0.032M Na_2SO_4) impaired the performance of the ion exchange membranes reducing the GPD up to 1W/m^2. However, ongoing demonstration activities also proved these commercial membranes allow operation over long periods of time without fouling after simple pre-treatment of the water prior to the RED module.

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Control of Hydrodynamics by Spacers to Enhance Electroconvective Instabilities

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Electrodialysis (ED) is becoming more prevalent in desalination applications. Conventional ED processes are run at low currents due to the increasing resistance at high currents. Shortening or even overcoming this plateau would result in a major improvement in efficiency by attaining higher currents at respective lower voltage drops and in the overlimiting regime. Electroconvection is the predominant boundary layer transport mechanism in this regime. The development of these vortices therefore must be manipulated by device design.

In this work, we use controlled flow distribution to enhance ion transport and manipulate electroconvection. For this, spacer geometries were developed, 3D printed, and empirically evaluated in our newly designed ED cell. The altering flow behavior of the channel caused by different spacer designs was further investigated using three-dimensional fluid dynamics simulations. We demonstrate that spacer geometries can influence the channel's flow dynamics and enhance ion transport. To achieve greater currents in the ohmic range and to delay concentration polarization, spacers that redirect flow towards the membrane are used. Also, in the overlimiting regime, bulk mixing results in improved ion transport. In addition, we investigate the coupling between the buoyancy effects and electroconvective instabilities in a spacer-filled channel. We manipulate and measure areas where electroconvective vortices can arise to quantify the impact of spacers on local ion transport for the first time. Our experimental findings are in good agreement with the modeling data reported in the literature.

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Branched Poly(Aryl Piperidinium) Membranes for Anion-Exchange Membrane Fuel Cells

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Anion-exchange membrane fuel cells (AEMFCs) are a promising, next-generation fuel cell technology. AEMFCs require highly conductive and robust anion exchange membranes (AEMs), which are challenging to develop due to the tradeoff between conductivity and water uptake. Here we report a method to prepare high-molecular weight branched poly(aryl piperidinium) AEMs. We show that branching reduces water uptake, leading to improved dimensional stability. The optimized membrane, b-PTP-2.5, exhibits simultaneously high OH conductivity (> 145 mS/cm at 80 °C), high mechanical strength and dimensional stability, good processability, and excellent alkaline stability (>1500 h) in 1 M KOH at 80 °C. AEMFCs based on b-PTP-2.5 reached peak power densities of 2.3 W/cm^2 in H_2-O_2 and 1.3 W/cm^2 in H_2-air at 80 °C. The AEMFCs can run stably under a constant current of 0.2 A/cm^2 over 500 h, during which the b-PTP-2.5 membrane remains stable.

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Anion exchange membranes for the electrochemical reduction of CO_2

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The electrochemical reduction of residual CO_2 towards fuels and chemicals such as ethanol and ethylene is a promising route to lower our carbon footprint, especially when combined with renewable energy to drive the reactions. CO_2 reduction is mainly performed in membrane-containing flow cells consisting of electrodes on opposite sides of an ion exchange membrane in an alkaline electrolyte solution. The ion exchange membrane should facilitate the transport of ions between the anode and cathode while serving as a chemical barrier between the two compartments. When operating in an alkaline environment, anion exchange membranes are preferred because their positively charged exchange groups allow the transport of hydroxide ions from the cathode to the anode. Their main drawback lies in the poor chemical and mechanical stability at high pH and cross-over of negatively charged reaction intermediates. While extensive research has been performed on electrodes for CO_2 reduction, it remains unclear how the membrane affects the reduction and which membrane-type would be ideal. Therefore, we focused on commercially available membranes and analyzed their performance in terms of selectivity and efficiency for the CO_2 reduction reaction to use as a benchmark for further research. We aim to expand the current membrane technology by incorporating functionalized silica nanoparticles to improve hydroxide conductivity without sacrificing membrane selectivity and mechanical stability. By using wire-based electrospinning, a more homogeneous distribution between the organic polymer and silica nanoparticles is achieved, leading to better mechanical strength and conductivity compared to traditional casting methods.

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High-performance anion exchange membrane water electrolyzers with a current density of 7.68 A cm-2 and a durability of 1000 hours

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Low-cost anion exchange membrane (AEM) water electrolyzers (AEMWEs) are a new technology for the production of high-purity hydrogen; however, their current density and durability are far lower than those of proton exchange membrane water electrolyzers (PEMWEs). Here, we report poly(fluorenyl-co-aryl piperidinium) (PFAP)-based anhydrous cathode AEMWEs that exceed the state-of-the-art PEMWEs with respect to current density. In addition to a rational electrode design, PFAP-based AEMs with a high water diffusivity and ion conductivity are crucial for high performance AEMWEs. Using platinum-group-metal (PGM) catalysts, the present AEMWEs achieved a new record current density of 7.68 A cm-2 at 2.0 V with a 1 M KOH anode, which surpasses that of state-of-the-art PEMWEs (6 A cm-2 at 2.0 V). PGM-free AEMWEs displayed an excellent current density of 1.62 A cm-2 at 2.0 V. Importantly, PGM and PGM-free AEMWEs operated stably under a 0.5 A cm-2 current density at 60 oC for more than 1000 h. This work sheds light on current high-performance AEMWEs.

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Recovery of ammonium ion by Donnan dialysis using sulfonated polyether sulfone-based cation-exchange membranes

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Donnan dialysis (DD) is a technique for concentrating and separating useful or harmful ions in a large amount of dilute solution. DD process does not require electrical energy as compared with electrodialysis and reverse osmosis membrane processes. In comparison with forward osmosis membrane process, DD process can concentrate only ions from a diluted solution. Further, it has an advantage that the regeneration of resin is not required and continuous operation is possible as compared with an ion exchange resin process. In this study, we prepared cation-exchange membranes (CEMs) from sulfonated polyethersulfone (SPES) and evaluated fundamental properties and Donnan dialysis performance of the CEMs. Membrane charge density, membrane resistance, and dynamic transport number of the CEMs were measured by the same method as elsewhere [1]. Permeation experiments were carried out using a dialysis system consisting of a CEM, 400 mL of 400 ppm NH_4Cl solution as feed solution (FS), and 100 mL of mixed solution of 3 M NaCl and 0.1 M NH_4Cl as draw solution (DS) [2]. The ionic concentrations of FS and DS were measured by ion chromatography at predetermined time. The prepared CEMs showed almost the same membrane charge density and dynamic transport number as commercial CEM, Neosepta® CMX (Astom Corp., Japan). The concentration of NH_4^+ ions decreased with time, indicating that the ions was transported against their concentration gradient from FS to DS sides for maintaining electrically neutral conditions. In the experiments, about 85% of NH_4^+ ions in FS side were recovered at 22 h. The results indicate that Donnan dialysis has potential application to the concentrated separation of low-concentration NH_4^+ ions in industrial wastewater.

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Examination of operating conditions with high current efficiency in acid / alkali production using bipolar membrane electrodialysis

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Bipolar membrane electrodialysis (BPMED) [1] is consisting of cation exchange membrane (CEM), anion exchange membrane (AEM) and bipolar membrane (BPM), and one of the techniques to generate acid and alkali from salt water at the same time. In this study, we evaluated the relationship between the current efficiency and operating conditions of the acid/alkali production in BPMED process. BPMED was performed by ACILIZER EX3B (Astom Corp., Japan) using Neosepta® CSE, and, Neosepta® BPM. And for AEM of the BPMED, we used two types of AEMs, Neosepta® ACM as a proton blocking AEM, and Neosepta® ASE as a standard AEM (Astom Corp., Japan). The ED test was performed under a constant current of 4.4 A at 25 °C. The current efficiency in the process was calculated from the change in the amount of acid and alkali with operating time. In the case that the production concentration of acid and alkali were 0.5 mol/L, the current efficiency of alkali was 90% for both ASE and ACM. The efficiency of acid was 79% for ASE and 90% for ACM, indicating that ACM had 11% higher current efficiency of acid than ASE. The reason why ACM showed higher acid current efficiency than ASE will be as follows: In the case of using ASE, some part of protons generated by BPM passed through ASE to the salt chamber; hence, the ED system had low current efficiency for acid. On the other hand, ACM had lower proton permeability than ASE, the proton leakage during ED time was lower than that using ASE.

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H_2 Production by Water Electrolysis Using Cation Exchange Membrane: Insights on build a PEM electrolytic cell

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Low-temperature electrolysis by using polymer electrolyte membranes (PEM) is expected to play an important role in the hydrogen energy transition. In order to reach a high energy efficiency, not only the PEM itself, but also the cell design and the operation conditions need to be carefully optimized. In this work we present a study on some of the most important parameters to be considered for the design and operation of a PEM-based membrane electrolyzer. The core of the system is an iridium and platinum catalyst-coated Nafion membrane (Ir-Pt-Nafion™ 117 CCM), operated in the water electrolysis process at room temperature and atmospheric pressure [1]. The most important function of the CCM is proton conduction and catalytic conversion of water into H_2 and O_2. However, it was found that a higher amount of water is lost in the H_2 stream than the amount converted by the electrolytic process, due to the high water permeability of the PEM. This water should be recovered for the overall economy of the system, especially in those environments where the availability of water is limited, such as space applications. Some of the parameters investigated in this study are the cell clamping pressure, the PEM and catalyst type, the type and properties of the gas diffusion layer, etc. The process was evaluated under a range of different operation conditions, measuring the H_2 and O_2 production rates, electrical power, energy efficiency, Faradaic efficiency and polarization curves for all experiments. The performance of the cell will be discussed in terms of energetic and Faradaic efficiency as a function of the electrical current or current density and the other experimental variables.

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Role of tungsten trioxide in enhancing PEM performance for fuel cell at intermediate temperature

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Proton Exchange Membrane (PEM) represents a pivotal material and a key challenge in developing fuel cell science and hydrogen technology. Nafion is the most promising polymer, which will lead to commercializing it. However, it has a few disadvantages such as losing mechanical and dimensional stability in hydrate state at conventional temperature. Additionally, it has a high hydrogen cross over under same operating condition which negatively affects the cell performance. To overcome these limitations, hybrid membranes of nanosized tungsten trioxide (WO_3) and Nafion were fabricated. Different nanofiller content were applied in single cell under 80 °C and low humidity of 50% for H_2 and 30% for O_2 streams, respectively. 10 wt.% nanofiller showed a superior performance with respect to water uptake and mechanical stability compared to the recast Nafion as well as commercial membrane. The results promoted the study of the 10 wt.% membrane performance at different temperatures in single cell. Hybrid membrane containing 10 wt.% tungsten trioxide showed higher current as well as higher maximum power density at 95 °C compared to the commercial Nafion membrane.

A facile approach to fabricate ion exchange membranes for fuel cell and electrolysis applications

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An effective strategy to improve both the conductivity and chemical stability of ion exchange membranes was proposed by incorporating functionalized organic and/or inorganic materials into polymer backbones. The introduction of organic and/or inorganic materials provided the hybrid membranes with additional ion hopping sites and ion conducting channels. The hybrid membrane with high ion exchange capacity possessed the ion conductivity at high temperatures. Meanwhile, the hybrid composite membranes showed enhanced chemical stability due to the steric hindrance offered by organic and/or inorganic materials. The improved interfacial compatibility among the matrix polymer and organic and/or inorganic materials filler imparted the hybrid membranes enhanced mechanical properties, good dimensional and thermal stability. The prepared hybrid membrane with promising characteristics exhibited the potential for applications in fuel cells and electrolysis in considering their integrative properties.

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Beverage dealcoholization by stripping gas-liquid membrane contactors

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Over the last decades, the evolution of agronomic techniques in many wine-producing regions and the global warming has resulted on an increase in the average alcohol content of wines (+ 1.1° between 1992 and 2007). Due to health and social concerns about the risks associated with alcohol consumption, wine consumption has recently fallen sharply, particularly among young people. In order to attract a wide public and maintain consumption, wine producers are looking for processes that will reduce alcohol content of wine without altering its quality. Many gas/liquid separation processes have been currently performed on hollow fibers membrane contactors (HFMC) with hydrophobic membranes where the transmembrane transport is ensured by the difference in chemical potential between both phases. In the case of alcoholic beverages, HFMC allows driving vapors of main volatile compound (ethanol) of the liquid through the membrane to the stripping gas phase to be depleted or eventually condensed. Indeed, HFMC can be expected to be an interesting solution for beverages dealcoholization. To evaluate the feasibility and potential of the process, experiments were carried out on model solutions (hydro-alcoholic solutions) in a HFMC (LiquiCell ®) using dry or water-saturated air. Tests at different humidity content, gas flow rates, liquid temperature and ethanol concentration were carried out. This study demonstrated the effectiveness of the process, the alcohol content decreased up to 4° in 2 hours. Even if the use of humid air does not improve ethanol flux, it allows controlling water flux, which could be interesting in order to limit loss of aromatic compounds by steam drive. Temperature is a key parameter for the ethanol flux value. Further experiments need to be carried out on actual wines in order to evaluate the impact of other wine's compounds and assess the quality of the final product.

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Nitrogen recovery from effluents with high ammonium concentration using membrane contactor

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The recovery of nitrogen from effluents with a high ammonium concentration could be an excellent opportunity for biofertilizer production and promote the environment protection and a circular economy. In this way, anaerobic digestion is an extensive sludge treatment method that achieves organic matter reduction and bioenergy generation [1]. After anaerobic digestion, the sludge is dehydrated by centrifugation, and the liquid stream from this process is called sludge centrate. This wastewater has a high nitrogen concentration [2]. Membrane contactor is a promising technique that includes a hydrophobic membrane (which allows ammonia permeation in the vapour phase across the membrane) that acts as a selective barrier between the wastewater and the receiving agent. The ammonia reacts with the receiving agent, and it can be recovered as biofertilizer in the form of ammonium salt [3]. A membrane contactor via liquid-liquid phase with a polypropylene hollow fibre membrane was tested. The influence of type and concentration of the acid solution (H_2SO_4, H_3PO_4 and HNO_3 at a concentration of 0.05 and 0.1 M), the wastewater pH (9 and 10), the flow rates of the feed and the stripping acidic solution (equal 25 L/h and different 30 L/h and 15 L/h, respectively) and the contact time (test duration of 2 hours) on ammonia nitrogen removal and recovery percentages were evaluated. Results demonstrated that with a contact time of 67 min, removal and recovery ammonium percentages about 90% were obtained. In addition, both flow rates of stripping and feed solutions together with the acid concentration did not have a significant influence on the removal but on the recovery. Concerning used acid, H_2SO_4 and H_3PO_4 solutions achieved better results than HNO_3 solution. Finally, it has to be highlighted that pH was the most critical parameter. Higher pHs values led to higher ammonium removal and recovery percentages.

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Investigation of ceramic membrane contactors regarding their suitability for nutrient recovery from digestate

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The results to be presented here originate from a research project which focuses on the development of a highly efficient treatment system for the fermentation of organic residues with integrated nutrient fractionation. The process consists of many possible process stages for, which can be modularly combined according to customer requirements. The nutrients are to be recovered in the form of economically viable products (e.g. as liquid fertilizer). For the separation of nutrients and the production of these products, different process approaches are required depending on the substrates used [1]. At IKTS, special ceramic membrane contactors and filters are being developed and tested which are capable of removing ammonia and ammonium directly from the gas/vapor phase and the liquid phase under the sometimes very harsh conditions. The use of hydrophobic membrane contactors for so-called "membrane-assisted extraction" represents a promising solution to the recycling problem of ammoniacal nitrogen from waste, water or wastewater resources. Usually polymer-based hollow fiber membranes are used (made of PP, PVDF or PTFE) [2]. The receiving phase is mostly dilute sulfuric acid, which reacts with ammonia to form ammonium sulfate solution (ASL). ASL is an approved fertilizer. Inorganic, oxide membrane materials, such as Al_2O_3 or TiO_2, are considered to be intrinsically hydrophilic. IKTS has already demonstrated that macroporous inorganic membranes can be success-fully hydrophobized as single and multi-channel tubes as well as capillary bundles [3]. For ammonia extraction, different substrates and materials with different pore sizes were investigated. In addition to basic characterizations, such as determination of the wetting angle or the liquid entry pressure, initial investigations were carried out on the transfer of NH_4-N from NH_4OH to $(NH_4)2SO_4$ and determination of the transfer coefficient. Clear dependencies on material, pore size and process parameters were found and will be presented.

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NEW CONCEPTS FOR THE USE OF MEMBRANE CONTACTORS IN TREATMENT OF (WASTE) WATER AND THEIR IMPACT TO CLIMATE CHANGE

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Recent application developments for membrane contactors aim to reduce the levels of methane (CH_4) and ammonia (NH_3) in various water and wastewater streams. The goal is not only to separate the gasses from the primary stream as effectively as possible, but also to set the highest possible concentrations on the secondary side in order to enable further energetic or material use economically. The poster reports on various pilot and demonstration projects partially under the frame of EU nextGen Circular Water Solutions program. Methane is dissolved both in individual natural well waters and in the effluent of anaerobic wastewater treatment. A specially adapted mode of operation of the membrane contactors makes it possible to reduce both the concentration in the water from >20 mg/l to below 0.2 mg/l and about 90% of the gas to a concentration that enables it to be used for energy. Since the effect of methane on the climate is around 30 times greater than that of carbon dioxide (CO_2), incineration improves not only the energy benefit but also the climate balance. Ammonia is a raw material that is used in large quantities in our economic life. Because of the negative effects on aquatic ecosystems, the entry of ammonia (NH_3) dissolved in water as ammonium (NH_4^+) is regulated. In conventional activated sludge plants, the degradation to molecular nitrogen (N_2) requires increased effort and is associated with the release of nitrous oxide (N_2O). The climate impact of N_2O is estimated at around 300 times that of CO_2. A membrane contactor in a process known as TransMembraneChemiSorption (TMCS) can be used for recovery of the nitrogen as a concentrated ammonium salt solution, which can either be used directly as fertilizer or processed further, at significantly lower energy requirements and equipment costs compared to conventional chemical-physical methods.

PRERARATION OF MICROCAPSULES BASED ON POLYELECTROLYTE COMPLEXES BY MEMBRANE EMULSIFICATION

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Membrane emulsification is a simple, new, and convenient technology that allows to obtain of monodisperse emulsions by passing the dispersion phase to the dispersion medium through the pores of the microfiltration membrane of a certain pore size applying pressure [1]. Low shear stress during emulsion production in this method prevents the loss of therapeutic activity of the substance. Moreover, ability of obtaining particles with a narrow size distribution, which increases the system stability, is the main factor determining the usage of membrane emulsification in drug delivery systems design [2]. In these study microcapsules based on chitosan-carboxymethyl cellulose complex wew prepared by the membrane emulsification using polyethylene terephthalate track membranes. Papaverine hydrochloride was introduced in microcapsules in the process of their formation. The novelty of this research is determined by obtaining of polyelectrolyte-based microcapsules by alternating the introduction of polymer solutions into the dispersion medium by membrane emulsification, which allows obtaining of monodisperse microemulsions with drops of a certain size. Membrane emulsification was performed using track PET membranes with pore size of 0.1 µm. The size distribution and zeta potential of the surface of the obtained microcapsules were determined by the method of dynamic light scattering and the influence of temperature and pH on the particle size was investigated. The obtained microencapsulates were both thermo- and pH sensitive.

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The production of PCL nanoparticles by membrane emulsification/solvent diffusion and membrane nanoprecipitation: a comparative analysis

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The use of membrane technology for the production of polymer particles permits to redesign production processes with an enormous impact in terms of formulation quality (controlled size and uniform distribution), reduction of energy consumption, high efficiency, productivity and reproducibility. In the present work, two alternative methods based on membrane technology will be studied for the production of PCL nanoparticles: i) membrane emulsification/solvent diffusion and ii) membrane nanoprecipitation. The integration of membrane emulsification with solvent diffusion is widely applied for microparticles production while its use for nanoparticles production is still limited. On the other and, membrane nanoprecipitation has attracted high attention especially for the production of polymer nanoparticles. The performance of the two process for the production of PCL nanoparticles will be compared by using two different greener solvents for polymer solubilization. In particular, Benzyl Alcohol will be used in membrane emulsification while Ethyl Lactate for membrane nanoprecipitation. The two processes will be optimized in order to obtain uniform nanoparticles with maximum productivity (in terms of mass of particles produced over time) and mild operative conditions (reduced mechanical stress). The environmental impact of the two processes will be assessed on the basis of the metrics established by the Green Aspiration Level (GAL). The analysis permits to establish the best green process choice for the development of PCL nanoparticles. The method can be extended to other type of nanoparticles and used to design sustainable manufacturing processes in chemical industry.

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O/W Emulsions Stabilized with Lesser mealworm (A. diaperinus) Protein Concentrate: Studies of Droplet Coalescence with Microfluidics

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The need of finding new protein sources to cover their expected increase in demand has attracted attention to insect proteins, that are already present in the diet of more than 10% of the word population. Our study is centered in the technofunctional properties of lesser mealworm (A. diaperinus) protein concentrate, LMPC, and more specifically in its emulsifying properties. The goal is to ascertain the possibilitiy of replacing dairy proteins to stabilize oil-in-water (O/W) emulsions in food applications. As a part of this study, droplet coalescence in O/W emulsions stabilized with whey protein and LMPC under controlled conditions using a custom-made microchip has been studied. The experimental conditions for the two proteins are: two types of oil (hexadecane and sunflower oil), two pH values of the continuous phase (7 and 3), three different oil fractions (5, 7 and 14%), and two different protein adsorption times. The methodology to calculate the coalescence frequency is based on analysis of images obtained at the start and at the end of a coalescence channel and accounts for the formation of droplet dimers (two droplets coalescing to one), trimers (three droplets coalescing to one), and so forth. The results obtained clearly show that frequency of droplet coalescence is very similar for whey protein and LMPC at the same experimental conditions, pointing out that replacing this dairy protein in the formulation of the food emulsions is feasible. Longer adsorption times, high protein concentrations, and neutral pH are clearly correlated with lower coalescence for both proteins, while acidic pH causes and increase of droplet coalescence also for both proteins. The results so far indicate that LMPC is a sutiable replacer for whey protein to stabilize O/W emulsions.

Membrane ionotropic gelation as scalable method for nanoparticles production

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Ionotropic gelation (IG) is a production process for polymeric nano/microparticles that involves electrostatic interaction between two ionic species of opposite charge. The method, usually employed for the synthesis of natural water-soluble polymeric nanoparticles, is extremily attractive for its relatively simple procedure and the absence of organic solvents. Although IG is well studied at laboratory scale its application at large scale is currently under investigation. In the present work, for the first time, membrane dispersion technology in combination with IG was studied as suitable scalabe method for polymeric nanoparticles production. Chitosan (CS) and tripolyphosphate (TPP) were used as model materials. The two phases, one containing CS and the other containing TPP were put in contact by using a tubular SPG hydrophobic membrane with a pore diameter of 1 µm. TPP permeated through the membrane pores into the lumen side along which the CS solution flowed in continuous single-pass or recirculated operation mode. The process is strictly governed by the experimental variables such as pH or ionic strength and the physico-chemical composition of the polyelectrolyte and the influence of various parameters (i.e. CS/TPP mass ratio, CS and TPP concentration) on the characteristics of the prepared nanoparticles (mean size, PDI and zeta potential) were invetigated. The use of membrane technology permitted to carry out the IG in continuous mode under mild conditions with relatively low energy consumption and it is expected to promote the transfer of the process from laboratory scale to mass production. The designed membrane process permits to produce uniform chitosan nanoparticles from 170 to 300 nm with high stability over time.

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Maximize bioactive molecules encapsulation by Pulsed back-and-forward Membrane Emulsification

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In the present work, pulsed back-and-forward membrane emulsification (ME) method in combination with the cross-linking reaction was investigated to maximize the encapsulation efficiency of a bioactive molecule, tannic acid (TA), used as a model. TA a naturally occurring plant polyphenol, displays unique medicinal abilities as antioxidant, antibiotic, anticancer agent and it is also widely used in food applications. More recently, TA and its derivates have been increasingly investigated for their interaction with different material other species to obtain tannin-loaded systems with improved performances. The major advantage of the use of this membrane emulsification method is the low shear stress applied corresponding to the lowest energy input maintaining high uniform size distribution with high productivity [1]. Chitosan (CS) was used as carrier material while tripolyphosphate (TPP) as green chemical cross-linker. CS droplets were formed by preparing a water-in-oil emulsion in which an aqueous solution of chitosan and tannic acid was used as dispersed phase, while isooctane containing Span 80 as emulsifier was the continuous phase. The membrane system consisted of a tubular SPG membrane with a pore diameter of 1 µm. The emulsification process and the cross-linking reaction conditions were optimized in order to obtain droplets with a high degree of size uniformity, tuned nanosize and highest encapsulation efficiency. The results showed that pulsed back-and-forward ME has high potential for developing TA-loaded CS nanoparticles at the nanometric scale, with high degree of uniformity, and encapsulation efficiency of nearly 100%.

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A numerical demystification of the pre-mix membrane emulsification process: a comprehensive dynamic flow architecture impregnated distributed mass balance-based multi-phase modeling

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Pre-mix membrane emulsification (PME) is experiencing growing interest in the pharmaceutical and cosmetic communities for its impressively low shear approach of fragmenting emulsion drops into smaller ones by a dual-action of porous-network configuration and multi-phase flow distribution.

Herein, its relatively elusive residence time evolution of droplet size distribution (*DSD*) is numerically demystified by this dual contribution-based constructed “up-to-date” distributed mass balance-based multi-phase model, that this calculates utilizing developed two different pairs (mechanistic and ‘physics-taught’ Neuro-Network (NN)-based) of fragmentation functions (frequency and daughter size distribution).

The NN-based rate functions were formulated from segmented multi-phase CFD-knitted sufficient 3D videos of droplet life-stages (inside pore and near membrane surface) hatched by parametric design-matrix (a ‘design of experiment’ written discretized combination of (1) membrane anatomy, (2) velocity, (3) TMP, (4) relative viscosity, (6) volume fractions, and (5) interfacial tension ranges). These were eventually optimized to acceptable prediction-accuracy ($\text{RMSE}_{(\text{train, test})} = [> 0.87, > 0.92]$ and $\text{MSE}_{(\text{train, test})} = [< 0.29, < 0.32]$). The mechanistic counterpart was created in terms of the spatial evolution of distributed velocity, shear, and pressure for the same matrix's single-phase flow replication. All computation was done using Python and OpenFOAM software.

By taking pore’s vicinity and internal conditions into consideration via these rate-schemes, the model’s simulation was able to appropriately anticipate the sharp decline of mean sizes and the shift of DSD under different formulation and operational settings for SPG and NPs-type membranes (literature-harvested data). This model performed well for up to four consecutive passes, achieving a nearly monodisperse state and having final d50-values in the 0.5 to 1.1 range, with a maximum inaccuracy of 7.2 vol.%. Despite some discrepancies in specific cases, the findings seemed usually adequate when the PDI and mean size estimate were emphasized. Future perspective aims to broaden model's application to its sub-group processes.

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Graphical abstract

PVDF-chitosan hybrid membranes for membrane emulsification with olive oil

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Membrane Emulsification (ME) is the process of creating an emulsion by passing a pure dispersed phase or pre-emulsion through a microporous membrane. In the membrane emulsification process, premix (water-olive oil) with two types of pre-emulsion (with and without the emulsifier, i.e. Polysorbate 80) were used. PVDF membranes with different physicochemical properties were applied. Hydrophobic and hydrophilic PVDF samples were covalently modified with chitosan via a silane-based modifier. The study aimed to investigate the impact of the material modification on the physicochemical features (e.g. contact angle, surface free energy, surface charge, adhesion) and finally on the quality of the produced emulsion. In addition, the aim was to characterize the obtained emulsions and verify the effect of adding an emulsifier on their stability. Membrane emulsification was performed with an inert carrier gas (nitrogen), using a membrane emulsification measuring system. Analytical methods such as DLS and ELS were used to characterize the properties, e.g. average hydrodynamic diameter and zeta potential, of emulsions obtained in the membrane emulsification process. Optical microscopy was used to visualize the emulsions. The value of the applied pressure has an impact on the efficiency of the membrane emulsification process, regardless of the type of emulsion used as a premix, an increase in the pressure value was equivalent to an increase in the flux value. The membrane type played a significant role in the efficiency of the ME process only when the emulation was generated in the presence of Polysorbate 80. The addition of an emulsifier significantly influenced the properties of the obtained emulsions, their durability, and their stability. However, the type of membrane impacted the appearance of the emulsion.

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Hydrodynamics (Reynold’s number) are critical to limiting surface scaling in membrane distillation (and membrane distillation-crystallisation)

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In this study, the role of hydrodynamics in determining the onset of membrane scaling in membrane distillation is established. Scaling remains a critical challenge restricting the industrial implementation of membrane distillation/crystallisation. While the role of hydrodynamics that mediate crystal back transport from the membrane to the bulk solution has been briefly discussed [1], there is only limited theoretical description for how hydrodynamic conditions influence the probability for scale formation which precedes crystal growth [2]. Surface scaling has been observed in laminar and turbulent flows, thus its role in scale formation is unclear. In this study, we use in-situ diagnostic tools to determine the onset of nucleation, complemented with the application of Nyvlt’s solution to transform hydrodynamic data (Reynold’s number (Re), flux etc) for the characterisation of the metastable zone width (MSZW). This method can then establish how Re determines the kinetics of nucleation for scaling based on classical nucleation theory. For a fixed ΔT, an increase in Re reduced the induction time for bulk nucleation. This was explained by an increase in concentrating rate (LnR’) imposed by the higher Re, which reduced the time to induction, and increased the level of supersaturation at induction. At low Re, smooth cubic crystals were formed, whereas evidence of ‘rough growth’ was determined at higher Re, emphasising how the difference in supersaturation imposed by Re, informs on bulk crystal morphology. Surface scaling was most significant at an intermediate Re of 1750 (Re 1100-2300 tested). This was explained through boundary layer characteristics which limited solute back transport. For these conditions, surface scale comprised very different morphological properties, indicating the growth mechanisms underpinning bulk and surface crystals to be distinct. The work provides a unified framework for establishing hydrodynamic conditions to limit scaling and enhance control over morphological properties of bulk crystals for crystallisation applications.

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Energetic penalty for water recovery from mining brine by direct contact membrane distillation

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Several potential sources of water and soil pollution are arising from industrial process wastewater, waste dumps and brines streams from energy and water intensive industries such as mining facilities. In the past brine was simply discharged in the management strategy, but the environmental impact combined with water stress have recently imposed the adoption of new approaches. This change consists in the passage from disposal to utilization and resource recovery [1]. In this logic, the intelWATT Case Studies 2 [2] aims to valorise a brine stream from mining activities by harvesting salinity gradient power (SGP) by reverse electrodialysis (RED) and recovering deionized water by membrane distillation (MD) in an integrated system exploiting renewable energy sources (solar energy) [3]. The present work examines the performance of direct contact membrane distillation (DCMD) for water recovery and re-concentration of RED outgoing mixed solutions, when using synthetic solutions simulating mixtures of brine from mining facilities (used as high salinity solution in the RED process) and well water (used as low salinity solution). We compared the transmembrane flux, salts rejection, gained output ratio (GOR) and specific energy consumption (SEC), under the effect of different membrane material, feed compositions and flow rates. Transmembrane flux increases going from pure brine to pure well water as feed, due to the increased solvent activity that positively affects the vapor pressure gradient across the membrane. One of the main advantages of the integrated RED/MD system proposed, is the possibility to tailor the MD feed solution salinity (and specific thermal energy consumption) by controlling the mixing ratio of the RED outgoing solutions partially mixed through the RED module. The direct relation between energetic penalties obtained in the several process conditions of DCMD clearly shows the necessity of introduction energy recovery system to make MD process energetically affordable for water recovery.

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Laboratory-scale evaluation of petroleum industry wastewater dewatering through membrane distillation.

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The incumbent water stress scenario imposes wastewater valorisation to freshwater, promoting technology development for its effective treatment. Petroleum industry wastewater is quite problematic due to relevant levels of toxic contaminants both organic and inorganic [1]. Membrane distillation has been evaluated for its dewatering, considering two main objectives: - recovery of freshwater; - reduction of liquid waste to disposal. Membrane distillation is based on vapour pressure difference between opposite sides of hydrophobic porous membranes [2]. The latter ones act as contactors, allowing distillate vapour flows from the compartment containing warm feed. Distillate vapour can be condensed into liquid freshwater while concentrate remains as liquid waste. Membrane distillation is not based on osmotic pressure thus it can face high levels of dissolved solids. Resistance to fouling and mild operating conditions are further benefits. Petroleum industry wastewater was sampled from an Italian site. Average levels of its main physical-chemical characteristics were: pH of 12.7, electrical specific conductivity of 10180 uS/cm, chemical oxygen demand level of 711000 mg/L, boron concentration of 358 mg/L. It was fed to a laboratory-scale membrane distillation system based on hollow-fiber polymeric porous membranes at 40 - 50 °C and 1 bar. Air was used as sweep gas to collect distillate vapour that was finally condensed through a gas-liquid heat exchanger. Abatements of more than 99 % of electrical conductivity, chemical oxygen demand and boron concentration, respectively, were noticed in distillate with respect to feed. Furthermore, distillate was almost pH neutral. Obtained results point to effectiveness of membrane distillation for petroleum industry wastewater dewatering, offering also novel opportunities for freshwater recovery.

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Evaluation of membranes performance for the treatment of high salinity sources with membrane distillation

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Membrane distillation (MD) is a thermal desalination technique with promising application in the management of high salinity sources. It has been demonstrated that the tolerance of MD membranes to salinity is much greater than those used in other membrane operations such as reverse osmosis [1]. Features of the membranes determine their performance, and therefore their separation capacity [2]. In this study, the performance of newly developed MD membranes is compared with that of currently commercial ones made of different materials and with mean pore sizes between 0.1 and 1.5 µm. To carry out the experimental campaign, an MD system manufactured by the German company SolarSpring GmbH is used. This unit is fully equipped to work in different operational modes: direct contact (DCMD), air gap (AGMD), permeate gap (PGMD) and vacuum assisted air gap membrane distillation (V-AGMD). Membranes with areas of 77 and 375 cm^2 are installed in two different plate and frame modules. The influence on the performance of feed inlet temperature, feed circulation velocity, and vacuum level is assessed. Finally, membranes hydrophobicity is tested in each of the aforementioned operational modes using different aqueous sodium chloride feed solutions with concentrations between 35 and 292 g/L. Performances of each membrane in each operational mode are compared by means of the permeate flux, the salt rejection factor, and the thermal efficiency of the membrane, with particular emphasis on evaluating the ability of the newest ones for brine concentration. The use of vacuum has shown to improve significantly the flux at high salinities. However, not all membranes perform well in these conditions, since the mean pore size and the material, which directly influence the liquid entry pressure, become a stronger limitation with vacuum.

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Recovery and control of lactose crystals using membrane crystallization

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Whey is the main by-product in the dairy industry, as ~8-9 L of whey is produced for producing 1 kg of cheese [1]. Whey contains high amount of lactose (~4% w/v) [2], which highlights the potential of the recoverable lactose in the global dairy industry and the importance of resource recovery in this field [3]. In this research, the direct contact membrane crystallization (MCr) process was investigated for recovering the lactose crystals from aqueous solution and the potential to improve the control of the crystallization process with respect to conventional processes. The effect of operating parameters, i.e. feed temperatures (30-60 °C) and flowrates (200-600 $mL.min^{-1}$), was investigated on the permeate flux and crystals characteristics. The average fluxes for feed temperatures of 30, 40, 50, and 60 ^{o}C (under constant flow rate of 400 ml/min) were measured to 0.41±0.02, 0.74±0.08, 1.14±0.11, and 1.51±0.3 $kg.m^{-2}.h^{-1}$, respectively. In terms of the crystal recovery, two main morphologies were observed, including the tomahawk and rectangular shapes. Increasing the feed temperature leads to more rectangular crystals and the overall contribution of the tomahawk crystals decreased. However, at each investigated temperature, more tomahawk crystals appeared with increasing recovery factor (RF). The crystal recovery (CR) was also evaluated for each experiment. CR increased with increasing both the temperatures (16, 23, 37, and 44% at feed temperatures of 30, 40, 50, and 60 °C, respectively) and the flowrate (33, 44, and 50% at flowrates of 200, 400, and 600 $mL.min^{-1}$, respectively). Overall, the obtained results could introduce a more affordable unit operation for recoverying the high-quality lactose crystals from dairy industry, thereby strengthning sustainability and circular economy of food industry.

Acknowledgements

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Membrane distillation for produced water treatment

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Produced water represents the largest volume waste stream in oil and gas production operations on most offshore platforms [1]. It is a mixture of dissolved and particulate organic and inorganic chemicals in water. The major compounds of produced water include: dissolved and dispersed oil compounds, dissolved minerals, production chemical compounds, production solids, dissolved gases. Proper treatment and management of produced water are necessary to avoid possible negative environmental impacts associated with its discharge. Moreover, the treatment of produced water can be beneficial in bridging the gap between demand and availability of freshwater in water scarcity regions where generally oil and gas reservoirs are located. Various physical, chemical and biological methods and their combinations have been used to treat the produced water [2]. However, conventional treatment methods have certain limitations including the usage of toxic chemicals, high cost of treatment, large footprint, long retention times and the creation of secondary pollution. Additionally, new stringent environmental regulations put emphasis on more effective treatments, underlying the importance of adopting more efficient treatment options. Due to these reasons, recently membrane based treatments have been tried widely for produced water treatment. Membrane distillation (MD) is an innovative membrane based process with the capability to treat the highly concentrated solutions and can be suitable to be used for produced water. In the present study, the performance of direct contact membrane distillation (DCMD)/membrane crystallization (MCr) system for freshwater and minerals recovery from produced water was analyzed.

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Photothermal nanocomposite membranes for solar membrane distillation processes

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Membrane distillation (MD) processes have shown increasing interest as one of the most promising desalination techniques due to advantages such as low pressure requirements, tolerance to high salt concentrations, zero liquid discharge, and low temperatures compared to other distillation processes [1]. However, despite the advantages in MD, challenges related to hydrophobic membrane pore wettability and temperature polarization (TP) still need to be resolved. Therefore, the use of photothermal materials is one of the most innovative options to mitigate TP [2]. These materials are capable of localize heating on the membrane surface, allowing the harvesting of energy from solar light [3]. Thus, the effects of incorporation of photothermal nanoparticles (NPs) such as commercial CuO and SnO_2 NPs into hydrophobic PVDF membranes were investigated. Using the non-solvent induced phase inversion method (NIPS), loading of 0.5, 1.0 and 2.5 % CuO and SnO_2 NPs, respectively were incorporated into membranes. Chemical, morphological, hydrophobic, and photothermal properties were analyzed by different characterization techniques and Photothermal Tests. The SEM, EDX and FTIR analyzes confirm the incorporation of commercial CuO and SnO_2 NPs in the membranes, with good dispersion on the surface of the membranes without affecting their porosity, or PVDF chemical composition. The AFM showed microporous modified membranes with lower roughness than the unmodified related with the good dispersion of the NPs. Contact Angle exhibited a slight decrease in the hydrophobicity of modified membranes by both NPs respect to bare membranes. Also, the Photothermal Tests revealed a great increase in the surface temperature membranes under solar light simulated irradiation in relation with the loading of NPs in the membranes. Increases of the temperature of until 4.2 ± 0.3 °C were reached in the modified membranes, exhibited like as an excellent opportunity to alleviate TP problems in MD under sunlight.

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Direct Contact Membrane Distillation applied to the simultaneous treatment of two saline streams

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Desalination by reverse osmosis (RO) allows to obtain two streams: one concentrated, rich in salt (brine), and the other very diluted (fresh water). The RO brine cannot be directly discharged into the marine environment, without altering the ecosystem. Therefore, its handling and disposal are the main challenges to face. In this work, Membrane Distillation (MD) in Direct Contact configuration (DCMD) was applied for the simultaneous treatment of solutions with NaCl concentrations of around 70 g/L at the permeate side (representing the RO brine) and around 18 g/L at the feed side, in order to obtain, at both sides, streams with concentration of seawater (around 35 g/L), so as to be discharged back to the sea. For this purpose, the experimental tests were carried out using a lab set up in which the module was equipped with a flat commercial membrane made in polypropylene (membrane pore size: 0.2 µm, thickness: 91 µm, membrane area: around 40 cm^2). During the experiments, the effect on the permeate flux and feed/permeate conductivity of the feed temperature (40-50-60°C), permeate temperature and feed flow rate, while the permeate flow rate was fixed at 42 L/h, was investigated.

Enhanced Nutrient Recovery from Mariculture Wastewater via Air Gap Membrane Distillation Process

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Global seafood productions from marine aquaculture activities have been increasing rapidly since the last decade. As a consequence, majority of the global marine ecosystems are currently in risks due to the mariculture wastewater pollution from the leftover feeds and feces of farmed fishes. Mariculture wastewater often contains high concentrations of organic nutrients which could lead to an intensified case of eutrophication to marine body water and sediments. On the other hand, air gap membrane distillation (AGMD) process is regarded as a promising technology with high purification efficiency and exceptional performance for nutrients recovery. Previous studies have investigated the potential of AGMD for treating high-salinity wastewater from other sources. However, there are still less concerns about its enhanced nutrient recovery ability and optimum operation parameter particularly for mariculture wastewater. Hence, this work aims to investigate the performance of AGMD to purify mariculture wastewater alongside its enhanced nutrient recovery potentials for further use. In addition, optimum operation parameters were also determined through variating the feed temperature (40-70ºC) and membrane spacer width (0.6 – 1.1 mm). The result showed an exceptionally high permeate flux of 8.93 kg/m^{2}h and enhanced nutrient recovery rate of 83.7% which were achieved under 70ºC feed temperature, 0.6 mm spacer width, and feed water pH of 7. This result highlighted the potentials of AGMD as an alternative to solve the mariculture wastewater pollution issues.

Improving structural homogeneity, hydraulic permeability, and mechanical performance of sustainable skinny asymmetric monophasic cellulose acetate/silica membranes: spinodal decompositions mix

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Polymeric membranes have emerged as a critical component in solving vital energy and environmental problems and are intensively explored for gas separations, water purifications, and fuel cell and battery applications. Recently biomedical applications such as tissue engineering, artificial organs, and biosensing give them a prominent place. Notwithstanding, polymeric membranes reproducibility and accuracy are still an experimental challenge. In this work we propose an optimized protocol to synthesize reproducible, accurate, sustainable monophasic hybrid skinned asymmetric cellulose acetate (CA)/silica (SiO2) ultrafiltration (UF) membranes. Eight different membrane compositions were studied, divided in two series – series1 and series 2. In each series, the silica amount increased from 0 %wt up to 30 %wt (with increments of 10 %wt) while the solvent composition was kept constant within each series (formamide/acetone ratio equals 0.57v/v in series1 and 0.73 v/v in series2). Membranes' morphology was analyzed by scanning electron microscopy (SEM), chemical composition by Fourier Transform Infrared Spectroscopy, in attenuated total reflection mode (ATR-FTIR). Mechanical tensile properties were determined using tensile tests and retest trial was performed to assess mechanical properties variability over different batches. UF characterization was carried out after membrane compaction by measuring pure water flux and membrane hydraulic permeability. Two different membrane network was confirmed by FTIR-ATR – in the first series a homogeneous organic-inorganic network formed while in the second series silica micro-clusters emerge. Within each series (with constant formamide/acetone ratio) Lp and Young modulus decrease with silica contend. Changing the solvent system, Lp increases while Young modulus decrease with the formamide/acetone ratio.

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Multipass hollow fiber membrane modules for membrane distillation

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Membrane distillation (MD) is a promising thermal-energy-driven process for desalination and wastewater treatment; however high thermal energy consumption remains one of the main obstacles in its widespread adoption. The current study presents multipass hollow fiber membrane modules to improve thermal efficiency of MD process. Fundamentally three module designs are considered: conventional one shell and one tube (fiber) pass (A), 1 shell and multiple tube passes (B/C) and equal but multiple shell and tube passes (D). The performance of the proposed designs is analyzed as a function of length of each pass, number of passes and operating conditions by using Aspen Plus simulator. The results demonstrate that the traditional design A yields the highest flux whereas the multipass design D shows the highest energy efficiency. It was observed that under specific operating conditions, single shell and multiple tube pass designs also offer the opportunity of heat recovery within the module. The pressure drop in multipass modules was marginally (1.5%) higher than the conventional single pass modules.

Membrane distillation for phenolic compounds removal from surface water

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Micropollutants, such as phenolic compounds, are widely spread in surface water, especially in developing countries, where sewage and wastewater treatment are limited. These compounds cannot be properly removed using conventional water treatment systems and may occur in drinking water. Due to their toxicity even at low concentrations, the phenolic compounds are among the priority pollutants of the US Environmental Protection Agency (EPA). The removal of these compounds to achieve safe water quality makes fundamental the use of advanced technologies, such as membrane distillation (MD). Comparing MD with classic distillation and other membrane processes, it has advantages such as: (i) low operating temperature, which allows its association to alternative energy sources such as geothermal, solar and industrial residual heat; (ii) operation at ambient pressure, which increases the system's security and decreases equipment costs, and (iii) membranes are less expensive. Therefore, a series of studies were carried out to evaluate the removal efficiency of 15 phenolic compounds from real surface water by MD process using PTFE membrane, supplied by Sterlitech Corp. It was analyzed the operational parameters, such as temperature (40 – 60 °C), feed concentration (3 – 10 μg L-1) and permeate recovery (30 – 70%) [1], fouling effect (types and retention mechanisms) [2], and system expansion (effective filtration area of 0.0042 – 0.3 m2). The results achieved showed that compound's volatility contribute more to the removal efficiency than the hydrophobicity. The pollutants were removed with >90% efficiency at the highest temperature (60 °C). High recovery did not affect the pollutants' removal. The fouling layer favored the phenolic compounds concentration in the feed solution without compromise permeate quality. MD reduced the surface water environmental and human health risk, demonstrating the great potential in producing safe drinking water.

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Ceramic membranes to achieve zero liquid discharge through Percrystallization

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Availability of efficient methods to fractionate a solution into its solid and liquid components is the key to recycling and reuse of industrial effluents. Percrystallization is a novel membrane process where a thin film of warm permeate present at the membrane surface is evaporated through vacuum to crystalize the soluble compounds. Thus, the process separates a solution into crystalline solute and solvent in a single-step and can partly be operated with sustainable sources of energy such as solar and geothermal heat. Since the process is at early stage of exploration, there is no sufficient knowledge about the suitable membrane features for the percrystallization which hampers its further development and potential progress towards commercialization. This work performs a systematic investigation to identify and develop suitable membranes for percrystallization applications. The study has been carried out by using silicon carbide (Si-C) and polymeric membranes with different porosities and thicknesses. The effect of pore size and surface hydrophobicity on the process operation was investigated by coating the surface of Si-C membranes with methylated silica particles of different sizes. Inherent hydrophobicity of the polymeric membranes was also modified to test their suitability for the percrystallization applications. All the membranes were tested for separation of NaCl crystals from solution by using different concentrations (3.5, 10 and 17.5 wt%) of NaCl at operating temperatures of 50°C, 55°C and 60°C. It was observed that the membranes with the rate of permeation comparable with the rate of evaporation under the applied vacuum are crucial to operate the process. Thus, only less porous and thick Si-C membranes with liquid entry pressure approaching to 1 bar were suitable for percrystallization. The developed membranes yielded water and NaCl flux in the range of 9.43±2 and 1.2 kg/m2.h, respectively. The process outperformed state-of-the-art membrane processes in terms of productivity and simplicity.

Stability-time performance study of Methanol conversion to Dimethyl ether on a α-Al_2O_3 catalytic membrane

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Dimethyl ether, the simplest of the ethers, is a non-toxic and non-carcinogenic molecule considered one of the most promising alternative and clean fuels of the future. The catalytic conversion of methanol to DME over a commercial α-Al_2O_3 membrane was studied with the aim of knowing its stability over time. In literature the most acid catalysts used are ɣ-Al_2O_3 and zeolite pellets; e.g. over H-ZSM-5, conversion values of 80% are achieved [1]. Traditionally Al_2O_3 is the most investigated solid acid catalyst, due to its low cost, good thermal and mechanical stability, high selectivity to DME even at a high temperature. Nevertheless, the catalytic activity of Al_2O_3 is strongly inhibited by the presence of water, produced during the reaction [2]. For this reason, in this study, the dehydration of methanol to DME was carried out in a membrane reactor, using a tubular catalytic α-Al_2O_3 membrane, operated as a contactor in through flow configuration, so to provide greater stability of calalyst toward H_2O thanks to the continuous removal of reaction products (DME and water) from catalytic sites. The reaction was carried out at 250 and 290°C, feed pressure of 120 kPa and WHSV of 1h-1. The methanol conversion, at 250°C was 20%. After that, although decreasing at the beginning probably due to the adsorption of water on the catalytic sites, became stable over time. After a stand-by period of 30 days in which the membrane was kept under nitrogen flow, the conversion returned to a value of about 23% and then behave following the same trend as before. At 290°C, the conversion of the membrane was slightly lower, but quite constant over time. In addition, the α-Al_2O_3 membrane reactor showed a DME selectivity of 100%.

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Reusable nanocomposite-filters for arsenite and arsenate dual real effluents remediation in an up-scaled membrane reactor

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Water contamination represents one of the largest global concerns leading to significant threats to human health and ecosystem biodiversity. The primary water contaminants include organic dyes, pharmaceuticals, inorganic compounds, and heavy metals. The former deserves special attention due to their higher mobility in water bodies and adverse effects on both humans and natural life health. In particular, contamination by arsenic (As) oxyanions has become a severe environmental and public health issue. In this line, it is estimated that more than 150 million people in over 70 countries have been exposed to As-contaminated water with levels above the legal limits. To adress this contamination issue, a membrane reactor (MR), combining adsorption and membrane separation processes, represents a new hybrid and promising technology for arsenic-contaminated water treatment. This work reports on nanocomposite filters (NCFs) based on poly (vinylidene fluoride-hexafluoropropylene), PVDF-HFP, containing yttrium carbonate (Y2(CO3)3) and magnetite (Fe3O4) to adsorb neutral and anionic species of As(III) and As(V), in an up-scaled membrane reactor. The nano-sorbents and the NCFs were thoroughly characterized concerning morphological and physical-chemical properties. Flow rate and pH of the media are the main parameters affecting the adsorption efficiency: lower flow rates and acidic pH are the most favorable conditions for dual As(III) and As(V) adsorption. Y@Fe3O4/PVDF-HFP adsorption follows a pseudo-second-order kinetics described by Langmuir isotherm model, reaching maximum absorption capacities of 101.9 and 212.8 mg/g for As(III) and As(V), respectively. In addition, the NCFs proved to be effective for the removal of As(III) and As(V) in treated effluents and easily reactivated and reused without significant efficiency losses. Thus, Y@Fe3O4/PVDF-HFP nanocomposite filters and the designed up-scaled membrane reactor represent a straightforward, efficient, reusable, and low-cost alternative for a continuous treatment process for As(III) and As(V) remediation in real condition effluents sources.

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Vinasse treatment by anaerobic membrane bioreactor in soudano-sahelian climate: effect of SRT on biogas production and membrane fouling.

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The membrane bioreactor is a suitable technology for the treatment of industrial effluents. The Soudano-Sahelian climatic conditions are favourable for anaerobic digestion of effluents loaded with organic matter such as those from sugarcane industries like vinasse. It would be interesting to optimise the operating conditions in order to improve the quality of the treatment. The tests were carried out on an experimental anaeroby MBR configuration with an ultrafiltration membrane of 0.45 m² and a reactor volume of 20 liters. The pilot was fed with a vinasse from sugarcane industry located in the south-western part of Burkina Faso. The hydraulic retention time of 1.5 days was fixed while two solid retention time (SRT) of 40 days and infinity days were set. The COD load varied from 17.3 to 23.9 g COD/L/d during 221 days of operation with a 90 days acclimatization period and temperature ranging from 25 to 41°C. The results obtained showed that the infinity SRT was more interesting with a very active microbial population and high biogas production compare to the 40-days SRT. Fouling was better controlled during the campaign with the 40-days SRT. The anaerobic MBR provided over 77% color removal from the influent and COD removal in the range of 71 to 96% but was not very efficient for ions removal. The biogas produced was estimated at 0.41 L biogas/g COD removed. Thus, the Soudano-Sahelian climatic conditions have provided a good acclimatization of the methanogenic microorganisms.

Anaerobic membrane bioreactor for vinasse recycling and by-products recovery: water, biomethane, and fertilizer

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Aiming for a higher valorization, an anaerobic membrane bioreactor was assessed for its potential for water, energy, and fertilizer recovery from vinasse. The systems operated for approximately 200 days and give in-depth insights into the advantages and limitations while dealing with vinasse. It would be possible to recover 527 m³/h of permeate to be used in cane washing procedures or as feedstock for fertilizer obtainment (~100% recovery rate), the latter with the advantages of better reuse of its nutrients and lower environmental impacts compared with cases where the permeate is used in fertigation practices. Their demand, aligned with economic aspects, should be considered when deciding on the best reuse alternative. The water demand for sugarcane washing should be attained by the permeate recovered, which represents a potential to reduce the water uptake from primary sources by 30%. The remaining fraction could be used as feedstock for organo-mineral fertilizers. That would allow better use of its nutrients, besides being an environmentally correct way to dispose of this residue. In countries like Brazil, highly dependent on fertilizer imports and a large producer of ethanol from sugarcane, the use of vinasse as a feedstock for an organo-mineral fertilizer can be an interesting alternative. In 2021, Brazil produced less than 13% of its fertilizer demand and imported more than 86% of its consumption, a value 19% higher if compared with 2020. The AnMBR presented an operational cost of 0.515 US$/m³ and allowed to recover 6.1 Nm³-CH_4/m³-vinasse, which represents an energy production potential of 26.1 kW/m³-vinasse. The energy recovery can increase the electricity independence of biorefineries. Overall, the systems would allow for the higher valorization of vinasse, with better use of wastes and contributing to less dependence on raw materials.

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Reuse4Hos: Hospital Wastewater Treatment and Reuse by Solar-Powered Membrane Bioreactor

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In East-African cities like Kampala, the capital of Uganda only about 7 % of the wastewater is treated in the municipal treatment plant [1]. Most of the wastewater is discharged untreated into the environment or inadequately treated in the central municipal wastewater treatment plants. Reusing this water for irrigation and other purposes have introduced pharmaceutical residues in the food chain. Hospitals are known to be a major source of pharmaceutical residues with wastewater typically co-treated in municipal wastewater treatment plants [2]. The aim of 'Reuse4Hos' project is to develop and pilot a robust and cost-effective technology that enables decentralised treatment of hospital wastewater with a high share of renewable energy. For this purpose, membrane bioreactor (MBR) based on 25 m^2 polyethersulfone (PES) membrane module is operated autonomously by solar photovoltaic (PV) energy and supercapacitor. The permeate collected from the MBR is further treated with granular activated carbon (GAC) to remove the pharmaceutical trace substances that are not degraded in the MBR. The plant exhibited enhanced degradation of nitrogen (N) and carbon (C) load due to an additional denitrification stage in the design. The plant is designed with simple and robust technologies that is cost-effective in the long-term whereby most of the components have been procured locally in Africa. The MBR piloted in the Lubaga Hospital in Kampala is designed to produce up to 10 m^3 of treated water per day that can be reused for irrigation, toilet flush etc. The demonstration of this solution within the framework of the German Federal Environmental Foundation (DBU) funded pilot project at Lubaga Hospital will serve as a model for wastewater treatment and reuse in hospitals in Uganda and other countries in East Africa.

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Unraveling pH effects on UF membrane fouling by extracellular polymeric substances: Adsorption and conformation analyzed with localized surface plasmon resonance

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One of the main contributors to elevation in the hydraulic resistance of ultrafiltration membranes in membrane bioreactors (MBRs) are extracellular polymeric substances (EPS) adsorbed to the pores' surface. While pH elevation is assumed to remove EPS from membranes surface, small changes in pH can change the adsorbed EPS conformation and orientation, resulting in a decrease in membrane permeability. To test this new phenomenon, we analyzed adsorbed EPS layers with Localized Surface Plasmon Resonance (LSPR) sensing and quartz crystal microbalance with dissipation monitoring (QCM-D). While LSPR analysis is sensitive to the surface interactions with a decay length of ~20–30 nm, QCM-D provides the mass and viscoelastic properties of the hydrated adsorbed fouling layer in a length scale of ~200-300 nm. We designed for the first time a membrane mimetic hybrid QCM-D – LSPR sensor, indicating both "dry" mass and mechanical load ("wet" mass) of EPS adsorbed, by light- and acoustic-based sensing of the LSPR and the QCM-D, respectively. Processes of EPS attachment, detachment, and conformational changes were differentiated, for different surfaces studied, including polyethersulfone (PES), polystyrene (PS) and silica (Si). These analyses showed clear indication for pH effects on the EPS adsorbed layer's conformation and orientation. EPS layer's viscoelastic properties and hydrated thickness, as calculated using QCM-D analysis, corroborate with the shift in EPS areal concentration, ΓS, and the associated EPS conformation, provided by the LSPR analysis. As pH elevates, the reversible processes of (i) elevation in EPS layer's thickness (QCM-D) and (ii) decrease in the EPS areal density, ΓS (LSPR), provide a clear indication for reversible changes in EPS conformation, which decrease the effective UF pore diameter. This decrease in pore diameter together with the increase in surface hydrophobicity elevate UF membrane hydraulic resistance.

The potential of vapor-phase membrane separation of organic components from biomass treatment products

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The processing of biomass to produce energy carriers (bioalcohols) and valuable chemical reagents is one of the most popular promising technologies. Most of the processes of biotransformation of organic substances lead to the production of dilute aqueous organic solutions with the content of target products in the range of about 1-10 wt.%. Ensuring the profitability of biological processes largely depends on the costs of the recovery stage and concentration of target products. This is especially true for modern methods of biomass processing (second-generation bioalcohols, lignin processing to produce bioalcohols via the acid route). This task can be solved by membrane processes, such as pervaporation, membrane distillation, vapor-phase membrane separation (VPMS) which have been widely studied in recent decades. The use of real fermentation media pervaporation is difficult due to membrane fouling by the biological medium and sorption of low-volatile organic components, which can significantly affect transport and separation characteristics. VPMS is devoid of these disadvantages, since the membrane contacts only with volatile components. VPMS is studied using laboratory and commercial hydrophobic and/or hydrophilic water-selective polymer membranes. VPMS seems to be the most suitable for the concentration of bioalcohols in modern processes of lignin bio-processing along the acid route lead to mixtures containing aliphatic alcohols together with acids. Mixtures containing acetic acid and water do not form an azeotrope; however, the separation of acetic acid from water is associated with certain difficulties. Separation of aqueous solutions of acetic acid, bioalcohols and triple mixtures based on them VPMS is studied in the temperature range of 50-80 °C. The content of acetic acid in aqueous solutions ranged from 1 to 28 wt.%. VPMS was modeled with an assessment of the potential of its application for the release of biogenic energy carriers.

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Pervaporative separation of methanol – dimethyl carbonate system with PDMS based mixed matrix membrane with pristine and functionalized MIL-53(Al)

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Dimethyl carbonate (DMC) is considered as an alternative, green solvent characterized by low toxicity and good biodegradable properties. During production of DMC, an excess of methanol is used, and DMC creates the binary azeotrope with unreacted methanol. There are various methods which can be used for the separation of dimethyl carbonate/methanol azeotropic mixture. Membrane separation techniques, especially pervaporation, appear to be a good alternative to traditional separation methods. Hydrophobic, heterogeneous PDMS based membranes with MIL-53(Al) and analogues of MIL-53(Al), i.e. NH2-MIL-53(Al) and NHOCOCF3-MIL-53(Al), were fabricated for enhanced removal of DMC from azeotropic DMC/MeOH mixture. Hydrophobic NHOCOCF3-MIL-53(Al) was obtained during the post-synthesis modification of NH2-MIL-53(Al) with trifluoroacetic anhydrate (TFA). The influence of the presence of water in the separated mixture on the efficiency of pervaporation was also investigated. The physicochemical properties of prepared MOFs and membranes were determined by applying analytical methods. FTIR-ATR analysis of NHOCOCF3-MIL-53(Al) powder confirmed the successful modification. The characteristic peaks of NH2-MIL-53(Al) and TFA were detected. Obtained MOF particles are highly crystalline, and NH2-MIL-53(Al) structure is well retained after modification. SEM analysis proves the formation of the dense membrane. TGA analysis showed that membranes are thermally stable up to 250°C. Pervaporation results were analyzed implementing separation factor and thickness normalized Pervaporation Separation Index (PSI). Pervaporation experiments showed that the optimum amount of particles in the PDMS matrix is equal to 5 wt%. The best separation efficiency was found for the PDMS/ NHOCOCOCF3-MIL-53(Al) 5 wt% All PDMS based membranes demonstrated EFwater higher than 1. Up 3 wt% of water in DMC/MeOH mixture, fluxes of DMC and MeOH decreased. Results indicated that lowest content of water in permeate was found for the PDMS/NHOCOCF3-MIL-53(Al) 5 wt% membranes. EFwater decrease with increasing water content in feed up to 3 wt% of water in DMC/MeOH mixture.

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On the use of pervaporation for the recovery of pharmaceutical solvents

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Today pharma industries recover certain solvents by using conventional techniques, such as distillation/rectification. Its main problem is the high-energy cost, especially in a European context of high natural gas prices. Pervaporation (PV) is a membrane process used to separate liquid mixtures [1]. In the case at hand, it is about dehydrating a ternary process mixture whose components are methyl-tert-butyl-ether (MTBE), isopropanol (IPA) and water. Using a commercial hydrophilic membrane, it is intended to go from a water content of 5% to less than 0.1%. A survey of commercially available membranes was made, and the PERVAP 4155-80 model, manufactured by DeltaMem AG (Switzerland), was selected. The PV tests were performed at two operating temperatures (55°C and 70°C), while the permeate zone was kept under vacuum. Taking as a starting point a waste stream from a pharmaceutical facility, the composition of the mixture to be separated was as follows: 85% MTBE, 10% IPA, 5% water. From the experimental PV results, an important finding was that the membrane was almost completely selective towards water, that is, the content of MTBE and IPA in the permeate was negligible. This point is very important, because this separation technique would avoid the loss of valuable organic solvents with the permeate stream and, on the other hand, the costs associated with treating the permeate stream as wastewater are reduced. Regarding the membrane performance, the water permeation flux increases with the water content in the feed mixture. This is because the water flux is proportional to the driving force given by the difference in water activity between the two sides of the membrane. Membrane is much more permeable at 70°C than at 55°C, allowing a moisture content of less than 1% in the solvent mixture to be reached more quickly.

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Synthesis and Characterization of Membranes Based on Graphen Oxide and Carbon Nanotubes for H2 Separation from Gas Mixtures

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This research explored the effects of combining two promising carbon-based materials, graphene oxide (GO) and single-walled carbon nanotubes (SWCNT), with various inorganic additives. Fabricated GO-SWCNT composite membranes were tested for effective H_2 separation from permanent gases. The GO-SWCNT composites benefit from unlimited permeability-selectivity performance [1], while polymer-based commercial organic membranes usually suffer from the trade-off effect. However, the better separation performance of inorganic membranes is usually offset by their problematic scaling-up, fragility and complicated preparation. To mitigate unwanted issues, such as fragility and lack of reproducibility, the influence of the SWCNT as support and modification of GO skin layer by adding selected polyvalent metal ions on the overall separation properties was investigated. A GO modification was performed by adding selected polyvalent metal ions into the GO solution before filtration to evoke recently reported interactions of ions with the GO structure [2]. Doped GO sheets become connected via intermolecular bonds that make the material more ordered and stable [3]. Prepared composite membranes were further characterized using analytical methods (SEM, XRD, XPS, FTIR-ATR, Raman, BET) to reveal the effect of the additives on the structure of materials and transport properties. The gas permeability and selectivity of the membranes were tested for single and binary gas mixtures using a permeameter connected to a GC. Combining bivalent metal ions with a graphene oxide provided enormous H_2 permeability > 30.000 Barrer with excellent H_2/CO_2 and H_2/N_2 selectivity (above 4.7 or 2.8, respectively), both above the corresponding 2008 Robeson upper bounds. Obtained results thus significantly exceed the separation characteristics of the vast majority of published membrane materials. Achieved results demonstrate the uniqueness of the prepared composite materials and the possibility of tailoring their permeability and selectivity.

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Tuning Ultramicropore Dimensions of Carbon Molecular Sieve Membranes via Electron Irradiation

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Carbon molecular sieve (CMS) membranes are considered as promising candidates to overcome the challenges that thermal based separation faced. However, effectively separating similarly sized molecules is still a challenging problem for CMS membranes. Here, by benchmarking the electron beam induced structural changes in carbonaceous materials, we reports tuning 6FDA:BPDA(1:1)-DAM derived CMS membranes ultramicropore with electron beam irradiation. The electron beam develops oxygen-rich layer on CMS surface that impose transport resistance on the large gas penetrants, providing high C_2H_4/C_2H_6 separation performance. Increase of beam dose leads to explicit diffusivity difference between C_2H_4 and C_2H_6 and is involved with diffusion selectivity enlargement, resulting in selective C_2H_6 discrimination from C_2H_4 in CMS membranes. Electron beam treated CMS also shows high stabilized permeability and selectivity than that of pristine one suffered significant performance degradation from physical aging. This study successfully highlights electron beam as a new CMS tuning method among the conventional methods mainly limited to pyrolysis conditions.

Advanced GNPs/CA Asymmetric Membrane Manufacturing via Electrohydrodynamic Emission

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Graphene nanoplatelets (GNPs) have excellent properties that can be leveraged for the fabrication of composite membranes intended for gas separation, mainly due their affinity for polar gases, hydrophilic nature and high specific surface area. Unfortunately, the fabrication of GNP composite membranes has various challenges, including the uneven dispersion of fillers, surface defects/voids, and incompatibility with the polymeric matrix. In this study, we demonstrate the potential of electrohydrodynamic emission (EHE) as a membrane manufacturing tool capable of generating ultrathin, defect-free and support-free asymmetric GNPs/cellulose acetate (CA) membranes for gas separation. The proposed methodology requires the dispersion of the GNPs via electrospraying onto a collector, followed by casting the CA solution, thus ensuring the homogeneity of the sample. We assessed the effect of GNP loading on the gas separation performance of membrane. Interestingly, the membrane displayed molecular sieving – a mechanism attributed to the GNPs layer. A solution-diffusion mechanism seemed to contribute to the process due to the presence of the CA layer. The GNPs layer displayed excellent mechanical and thermal properties. Single gas permeation tests showed a significant improvement in CO_2, CH_4 and N_2 permeability, as well as in CO_2/CH_4 and CO_2/N_2 selectivity, compared to the GNPs/CA mixed matrix membranes fabricated the same filler load. The superior features of the membranes suggest that EHE is an effective manufacturing technique that can be used to produce membranes where the homogeneity of the filler remains a challenge.

Modified Supports to Induce the Synsynthesis of High Performance ZIF-8 Membranes

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In this work, high hydrogen permeable ZIF-8 membranes were fabricated by combining the dopamine functionalized support and seed-induced growth method. In addition to providing abundant functional groups, polydopamine also can chelate metal ions (Zn^{2+}) to accelerate the nucleation on the support surface. Furthermore, polydopamine can anchor the seeds for promoting the heterogeneous nucleation on the surface of the supports, and prevent the seeds from entering into the supports porous. The characterization results by the X-ray diffraction and scanning electron microscopy showed a continuous and thin ZIF-8 layer was synthesized on the modified supports, resulting in a low gas permeance resistance. For the single gas permeation performance, the membrane showed a marked hydrogen permeance of 1.8×10^{-6} $mol\cdot m^{-2}\cdot s^{-1}\cdot Pa^{-1}$, and the idea selectivity for H_2/CO_2, H_2/N_2, H_2/CH_4, H_2/C_3H_8 is 4.1, 8.5, 11.3, 294, respectively. The H_2 permeance of the obtained membrane is about one order of magnitude higher than that of similar supports supported membrane. The membrane also demonstrated a comparable stability and separation performance for mixed gas separation. Demonstrating heterogeneous nucleation on the supports surface is an advantage for inducing the synthesis of a thin and continuous membrane.

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Layer by Layer assembly (LbL)of polyelectrolyte thin-membranes for gas separation

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Polymer membranes provide a promising opportunity in the development of an effective and sustainable technique for gas separations for various industrial application, such as, for example, Natural gas sweetening or Carbon Capture. The efficiency of gas separation membranes is strongly related to the material properties that should provide high fluxes combined with high selectivity to overcome the trade-off between permeability and selectivity [1]. In order to meet such requirements, an innovative technique has been considered for the fabrication of thin film composite membranes consisting of an ultrathin selective layer on a mechanically robust porous substrate [2]. That method, called layed by layer (LbL) assembly technique, consists in the alternated deposition of nano-sized polymer layers exploting weak electrostatic charges. In this work, Aquivion/PDDA and Aquivion/PVAm polyelectrolyte thin-membranes on porous substrates have been developed and tested for the separation of acidic compounds (such H_2S and CO_2) and N_2 from Natural gas streams. The idea is to exploit both the highly selective character of Aquivion, which exhibits interesting affinity with polar compounds and good separation performances in humid conditions (CO_2 and H_2S permeability reach values of 350 and 390 Barrer respectively at 35 °C and RH 80 %) [3], and the amine-based polymers able to interact preferably with acidic compounds with respect to Methane: the acid gases can thus diffuse much faster within the matrix. Furthermore, the nanometric size of the dense selective layer represents a unique solution able to maximize simultaneously selectivity, permeability, coupled to an intrinsic mechanical and chemical robusteness of the resulting membranes. The morphology and structural properties of the LbL membranes fabricated have been analysed by means of FT-IR and by surface ζ-potential, while the separation performance have been investaged by N_2, CO_2, H_2S and CH_4 permeation with both pure and mixed feed gas streams in humid environment and different operative conditions.

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Optimization of pretreatment and humidification methods of heterogeneous anion exchange membranes for direct air capture of CO_2

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Direct air capture (DAC) of CO_2 is one of the mobile-source CCS processes that can deal with distributed and mobile carbon emission sources, which is a variant of the conventional point-source carbon capture and storage (CCS) in terms of easing the CO_2 concentration levels and promoting negative carbon emission. The heterogeneous anion exchange membrane (AEM) is one of the most promising DAC absorbents composed of a polymer matrix with anion exchange resins functionalized with ammonium groups. The AEM can absorb CO_2 spontaneously when it is dry and release it when it is wet, based on the mechanism of the water-driven reversible hydrolysis reaction. The AEM is usually pretreated by the hydrothermal method [1] to obtain a hierarchical microporous structure to allow sufficient air access to the matrix and anion exchange resins. However, when the AEM is submerged in water, the desorption ratio (desorption quantity/adsorption quantity) of CO_2 is only 30%. In this work, to explore the effects of different pretreatment methods and testing methods on carbon capture performance, an AEM was prepared using different hydrothermal pretreatment durations and temperatures and measured for CO_2 adsorption/desorption performances using different humidification methods. The results showed that after being pretreated using the hydrothermal method under different durations, the pore structure, CO_2 capture capacity, and kinetic performance of AEM are similar to those obtained using soaking pretreatment. More importantly, we found that by using the micron-sized water droplets produced by ultrasonic atomization, the desorption ratio could be greatly promoted from ~30% to ~60% compared with the immersion humidification. The optimization of pretreatment and humidification methods in the present work reduces the energy consumption for pretreatment and significantly improves the desorption performances. Furthermore, it also provides an advanced method to enhance the efficiency of large-scale engineering implementation of DAC of CO_2.

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Effect of water vapor, pressure and temperature on the separation performance of mixed matrix iongel membranes containing azo-porous organic polymers

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One of the main challenges concerning the use of membranes in gas separation is their performance under industrial conditions, where high temperatures and pressures and presence of water vapor can affect the separation performance of the material. With this in mind, we evaluate for the first time the CO_2/N_2 and CO_2/CH_4 separation performance of iongel membranes under different experimental conditions (e. g. temperature, pressure and water vapor) to make a realistic assessment of the potential of this class of membranes for CO_2 separation processes. Mixed matrix iongel membranes were prepared by combining a high ionic liquid (IL) content (80 wt % of [C_2 mim] [TFSI]) with 20 wt % of poly(ethylene glycol) diacrylate PEGDA, to provide the necessary solid network, and 0.5 wt % azo-porous organic polymers (azo-POPs), specifically selected due to their affinity towards CO_2. The intrinsic high thermal stability and CO_2 affinity of the IL coupled with the CO_2-philic nature of the azo-POPs revealed to be of great importance to maintain the overall performance and stability of the iongel membranes under harsh conditions. For CO_2/N_2 separation, higher permeabilities were achieved with an increase in both temperature (from 30 to 80 ℃) and pressure (from 2 to 4 bar), with the opposite trend in selectivity. On the other hand, for CO_2/CH_4 separation, an increase in pressure at low temperature, lowered the permeability without a significant effect on the selectivity, showing the potential of these iongels to be used in biogas upgrading processes. The hydrophilic character of the prepared iongel membranes also revealed to have influence in their separation performance in the presence of water vapor, by significantly increasing permeability, at the expense of selectivity. Overall, the performance of mixed matrix iongel membranes containing azo-POPs is highly influenced by changes in the experimental conditions, and show promising potential to be used in gas separation processes.

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Thin film composite membranes based on PIM/Matrimid® blends for gas separation

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Membrane technology is a promising tool against greenhouse gas emissions and climate change [1]. The wide-spread use of membranes for large-scale industrial CO2 separation processes requires the development of highly permeable and selective membranes. A promising new class of gas separation membranes is formed by the polymers of intrinsic microporosity (PIMs), but so far, most studies have been focused on thick films. In this work, thin film composite TFC-membranes were developed by coating porous polyacrylonitrile (PAN) supports with a PIM solution to form a dense selective layer by solvent evaporation at 35°C [2]. We report a number of different PIMs (PIM-EA(Me2)-TB, PIM-EA(H2)-TB and AO-PIM-1) and their blends with various concentrations of Matrimid®5218 to tailor the permeability over a wide range. The effect of the PIM/Matrimid® blend ratio on the gas transport properties was studied by pure and mixed gas permeation measurements. The permeance of pure H2, He, O2, N2, CO2 and CH4 was determined in a fixed volume / variable pressure setup. CO2/CH4 and CO2/N2 mixed gas permeability measurements were performed on a variable volume setup with on-line mass spectrometric analysis of the permeate composition [3]. The TFC-membranes present higher CO2/CH4 selectivity and CO2/N2 selectivity compared to the thick films of the neat PIMs, close to the value of Matrimid®5218. Unfortunately, the first generation of TFCs has a relatively low CO2 permeance (100 GPU) compared to the value predicted on the basis of the polymer permeability and the selective-layer thickness observed by SEM, due to the low surface porosity of the supporting PAN membrane. Different approaches will therefore be investigated to improve the overall permeance of the membranes.

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Optimization of pretreatment and humidification methods of heterogeneous anion exchange membranes for direct air capture of CO_2

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Direct air capture (DAC) of CO_2 is one of the mobile-source CCS processes that can deal with distributed and mobile carbon emission sources, which is a variant of the conventional point-source carbon capture and storage (CCS) in terms of easing the CO_2 concentration levels and promoting negative carbon emission. The heterogeneous anion exchange membrane (AEM) is one of the most promising DAC absorbents composed of a polymer matrix with anion exchange resins functionalized with ammonium groups. The AEM can absorb CO_2 spontaneously when it is dry and release it when it is wet, based on the mechanism of the water-driven reversible hydrolysis reaction. The AEM is usually pretreated by the hydrothermal method [1] to obtain a hierarchical microporous structure to allow sufficient air access to the matrix and anion exchange resins. However, when the AEM is submerged in water, the desorption ratio (desorption quantity/adsorption quantity) of CO_2 is only 30%. In this work, to explore the effects of different pretreatment methods and testing methods on carbon capture performance, an AEM was prepared using different hydrothermal pretreatment durations and temperatures and measured for CO_2 adsorption/desorption performances using different humidification methods. The results showed that after being pretreated using the hydrothermal method under different durations, the pore structure, CO_2 capture capacity, and kinetic performance of AEM are similar to those obtained using soaking pretreatment. More importantly, we found that by using the micron-sized water droplets produced by ultrasonic atomization, the desorption ratio could be greatly promoted from ~30% to ~60% compared with the immersion humidification. The optimization of pretreatment and humidification methods in the present work reduces the energy consumption for pretreatment and significantly improves the desorption performances. Furthermore, it also provides an advanced method to enhance the efficiency of large-scale engineering implementation of DAC of CO_2.

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The impact of water vapor on the performance of mixed matrix membranes containing ionic liquids for gas separation

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CO_2 anthrophonic emissions have risen the need of developing effective CO_2 separation technologies to fight environmental impact. Membrane technology can efficiently separate gases, such as CO_2 from a flue gas stream. However, operating factors such as temperature, pressure and water vapor content are critical to the selective behavior and long-term stability of polymeric membranes. In this regard, mixed matrix membranes (MMMs) have been studied as an attractive alternative in CO_2 capture processes, combining the benefits of fillers within a polymeric matrix. Fillers, such as metal-organic frameworks (MOFs), have also shown benefits decreasing membrane plasticization and enhancing gas separation. Moreover, MMMs might also incorporate CO_2-selective plasticizers, such as ionic liquids (ILs), which improve the mechanical stability of membranes and their performance concerning CO_2 transport. This work combines different families of ILs with the MOF ZIF-8 with the elastomer Pebax®1657 (Arkema) to prepare MMMs using a modified phase inversion method, in order to obtain dense membranes. The effect on CO_2 selective separation has been assessed considering ILs with diverse properties and ZIF-8 at different loadings. The impact of the presence of water vapor on the binary gas mixture was also addressed. Results show the enhancement of CO_2 separation over N_2 by incorporating IL and MOF in the polymeric matrix, which was enhanced further by the presence of water vapor in the feed stream. The combination of MMMs and ILs improved the membrane stability in the presence of water at higher loadings. Future work will be focused on the preparation of the most favorable prepared MMMs as thin-film composite (TFC) membranes using a spray coating technique.

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Flat sheet and hollow fiber CO2/CH4 selective membranes based on green chemistry

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Upon the green chemistry guidelines and directives, the reduction or complete elimination of hazardous substances would be mostly favored. Subsequently, in this work, NMP was completely substituted by GBL, a non-toxic and environmentally friendly solvent. The successful polymeric co-polyimide, P84, hollow fiber (HF) and flat sheet (FS) membranes fabrication was based on Hansen solubility parameters and cloud point data from the ternary polyimide/solvent/non-solvent diagrams evaluation abetted by complete viscosity study. In this context, in order to have a spinnable dope, the sufficient viscosity of the dope solution is one of the most key parameters [1]. The appropriate viscosity is determined from the molecular concentration and weight of the polymer leading to polymer extrusion and spinning possibility without HFs disruption and discontinuity [2]. The critical concentration is defined as the intercept of the two extrapolated (tangent) lines of the corresponding linear parts of the viscosity curve [3]. The developed HFs were prepared by the dry-wet phase inversion process, which is based on the spinodal decomposition principle, in a spinning set-up, whereas dense FS membranes were prepared by the casting method. The derived membranes were characterized by SEM and their mechanical properties were evaluated. CO2/CH4 separation measurements under continuous flow were performed in real binary CO2/CH4 (10/90 vol.%) mixture under elevated pressure conditions. Furthermore, pressurization-depressurization cycle was applied in order to conceive the stability and the possible reversibility of the membranes' structure change by the pressure application. The developed green HF and FS membranes exhibited comparable results to membranes prepared using highly toxic solvents making them promising candidates for CO2/CH4 separation. Specifically, a separation factor of 27.3 and 75.4 of HF and FS membranes, respectively, were achieved. This work might render primary guidelines for the fabrication of membranes for gas separations in the context of green chemistry and sustainability employing ecofriendly solvents.

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Simulation study of single- and multi-stage membrane system based on CO2-philic composite hollow fiber membrane modules

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There are two main global problems were CO2-philic membranes can bring interesting solutions. One important problem is global warming caused by CO2-emmisions from burned fossil fuels. Here the membranes can help with removing of CO2 from thermal power plants. We have developed CO2-philic composite hollow fiber membrane based on PEBAX polymer. We have incorporated this membrane into the membrane modules and successfully tested in real flue gas. The second problem is somehow related and it is about the search of new alternative non-fossil fuels. One of these alternatives is biohydrogen production and its application as fuel. Biohydrogen is most often produced by anaerobic fermentation of organic material under specific conditions [1]. The result of this fermentation is a complex gas mixture with hydrogen and CO2 as two main components. In this work we examined several approaches how to purify raw biohydrogen using one- two- or three-stage membrane process. Simulations were carried by our own developed model of gas separation with the use of hollow fiber membranes. Our model considers separation of mixture containing several gaseous species in either cocurrent or countercurrent mode by membrane module of specified length. Model is therefore applicable for CO2 separation from flue gas and also for biohydrogen production. Model results were multiple times verified on real application data. Model's predictions were in good agreement with carried experiments and model proofs itself as suitable tool for membrane module and other experiments design. We were assuming utilization of membrane module with hollow fibers made of PEBAX material, which evinces permeances for CO2 approximately 100 GPU and selectivity CO2/H2 = 8. For example, the results show that 94% biohydrogen can be obtained from a model mixture of 50/50 H2/CO2 in two stage process with 75,7 H2 recovery.

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Separation of close-boiling point F-gas mixtures with hollow fiber membranes coated with a defect-free Pebax®1657/ Ionic Liquid composite layer

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Refrigerant mixtures of hydrofluorocarbons (HFCs) are currently in a phasing down process due to the implementation of international regulations aimed at mitigating their climate change impacts. At this juncture, novel refrigerant mixtures are being manufactured by mixing still valuable HFCs with low global warming potential (GWP) such as difluoromethane (R32, GWP = 675) with the 4th generation of fluorinated gases, namely hydrofluoroolefins (HFOs, GWP ≈ 1). Therefore, the implementation of innovative separation strategies to recover and reclaim valuable F-gases towards the manufacture of novel mixtures is urged. In this way, the environmental impact of both F-gas fabrication and management of end-of-life refrigeration would be drastically reduced. For this purpose, in our previous studies we selected polymer and composite polymer/ionic liquid (IL) membranes in flat thick film configuration, demonstrating very satisfactory results on the separation of F-gases [1,2]. In order to address the scalability of these membranes with an eye toward their industrial implementation, we have focused our efforts on designing tailor-made hollow fiber (HF) membranes for this specific application. The HF membranes were successfully developed by dip-coating of a selective layer of Pebax®1657 or combining Pebax®1657 with ILs on polypropylene porous supports. Gas permeation studies were performed with both single F-gases and refrigerant gas mixtures. These defect-free thin film HF membranes offered improved permeate fluxes, while maintaining the same selectivity as the analogous thick flat membranes. The inclusion of IL in the dense thin film coating provided an increase in both the permeate flux and the separation factor of F-gas mixtures, compared to the neat Pebax HF membranes. Ongoing work is being addressed to obtain composite HF membranes with selective layers below 1 um thickness.

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A combined molecular-level simulation and experimental approach to develop a thin-film polymer barrier for high-pressure hydrogen storage tanks

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Worldwide, it is now accepted that human civilization must switch from fossil to renewable energy sources and decarbonized forms of transportation, which include hydrogen technologies. These powered vehicles will use lightweight compressed hydrogen gas storage tanks made from plastic and fibre-reinforced composite materials. To reduce the gradual depressurization problem of such reservoirs, thin polymer films with H2 barrier properties are being developed. The two complementary approaches used for this work are 1) the pre-screening of candidate polymer networks by molecular modelling techniques in order to reduce costly laboratory experiments, and 2) the actual syntheses by interfacial polymerization (IP) for the most interesting structures. The IP technique allows for the production of thin, robust and defect-free films by localized polymer network formation at the interface of two immiscible solvents. In the literature, a number of atomistic models of varying complexity have been proposed to be used in molecular dynamics simulations of hydrogen [1-3]. However, due to this variety, it might be difficult to find those which are the most appropriate for a very large-range pressure and temperature study. As such, seven different atomistic hydrogen models were tested in order to select those which give the best agreement with experimental data related to permeation in the temperature and pressure regimes that are pertinent to storage tanks (-50°C < T < +90°C, 0 < P < 2000 bar). The effect of the anisotropy of H2 models on their ability to permeate in a polymer matrix was also assessed. At least two H2 models were selected for the next step of the project. The ongoing simulations are aimed at creating network polymer barrier models from appropriate monomers with different cross-linking densities, and these will be used later to characterize hydrogen permeation at the molecular level. In parallel, preliminary tests have been made on the interfacial polymerization part of the project. Recent results of the molecular simulations and experimental syntheses will be presented.

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ZrVTi sustainable multi-layered membranes prepared by HiPIMS

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Some metals belonging to groups IV and V show a high permeability to hydrogen and have been studied as possible alternatives to palladium in membranes for hydrogen purification/separation, in order to increase their sustainability and decrease their costs. Among the group V metals, vanadium shows a high hydrogen solubility at operational pressures, and this may affect the membrane stability due to embrittlement. To limit the hydrogen solubility, other metals with lower hydrogen solubility could be used to form vanadium-based alloys. However, up to date, very few alloys have been investigated. In this work, the deposition of dense micrometric Pd/ZrxVyTizPdw/Pd multilayers has been obtained by High Power Impulse Magnetron Sputtering onto porous alumina substrates. Dense Pd/ZrxVyTizPdw/Pd multilayers were obtained. The composition of the alloys, the morphology and structure, the hydrogen permeance, selectivity and the resistance to embrittlement were dependent on the deposition conditions, and the membrane with the enhanced performance was tuned. The environmental impact of these membranes was compared with those of more common Pd77Ag23 and V93Pd7 thin film membranes. The results showed as the partial substitution of Pd can efficiently lead to a decrease of the total environmental impact for these membranes.

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Comparative Analysis of ZIF-8 and ZIF-90 Matrimid® mixed matrix membranes for hydrogen recovery from industrial waste streams

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Membrane technology for gas separation has become a hot topic since the last decades due to advantages such as low capital and operational cost, modularity and low carbon footprint. Therefore, efforts are focused on the development of this technology with the aim of replace the traditional energy intensive processes such as distillation and absorption. Among the applications, air separation, natural gas purification, organic vapor removal from air or nitrogen. hydrogen separation stands out since its potential role as energy vector to boost the renewables contribution in the power generation sector [1]. Nevertheless, hydrogen end-uses require high performance membranes to reach the purity standars. Thus, this work provides the comparative analysis of the separation of Matrimid®/ZIF-8 and Matrimid®/ZIF-90 mixed matrix membranes (MMMs) as promising alternative for hydrogen recovery from the most suitable hydrogen-rich industrial waste gases; coke oven gas (COG), ammonia purge gas (APG) and methanol purge gas (MPG) [2,3]. Results shown that the addition of ZIF-8 and ZIF-90 increases both permeability (50% compared to pristine polymer) and selectivity (20% for H_2/CO_2 and 40-80% for H_2/N_2, H_2/CH_4 and H_2/CO). The high performance of MMMs relies on the incorporation of molecular sieve transport mechanism which enhaces H_2 permeability compared to the well-known solution-difussion transport in polymeric materials. Moreover, membranes were also characterized by scanning electron microscopy (SEM) and fourier transform infrared spectroscopy (FTIR) analysis and the influence of the temperature on the separation parameters (permeability and selectivity) was studied by the determination of the permeation activation energy of the gas compounds for each of the industrial waste streams. Thus, new knowledge on mixed matrix membranes separation performance under real process conditions was assessed. Moreover, the synthesis of mixed matrix membranes in hollow fiber configuration is being currently investigated since the promising results of Matrimid®/ZIFs for hydrogen recovery.

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Development of membranes for alkaline water electrolysis

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Alkaline water electrolysis is one of the easiest methods for generating high purity hydrogen. An alkaline, noble-metal free medium for electrolysis is more economical and less corrosive than acidic medium. The challenges in water electrolysis are to reduce the cost, energy consumption and maintenance, hence increase efficiency of the overall process. In electrolysis operation, the voltage drops that occur due to the cell components, add to the loss of energy. The objective of this study is to develop a membrane separator with low ohmic resistance and hydrogen permeability, essential for the alkaline electrolysis operation. A separator in an electrolyzer cell prevents mixing of the gases across the anodic and cathodic chambers, but also contributes to the voltage drop in the cell. A thorough analysis of the membrane's resistance is done based on an understanding of how synthesis parameters and structure determine physical and electrochemical properties and influence water electrolysis. Three categories of separators are being studied and reported for the alkaline for the alkaline water electrolysis: porous separators, anion exchange membranes, ion-solvating membranes. Porous separators are physical diaphragms which separate the anode and cathode, although allowing some mixing of gases and electrolyte across chambers. We report here our studies on a porous composite Zirfon membrane [1] (polysulfone and zirconium dioxide) separator with 80% loading of zirconium dioxide and a nylon mesh support. The average thickness and ohmic area-resistance obtained for the zircon membrane is 320um and 4.5ohm-cm^2 (measured in 0.5M NaOH via Electrochemical Impedance Spectroscopy), respectively. The membranes were tested for alkaline electrolysis and a quantitative relationship between the cell voltage, current and impedance results is established and analysed by calculating the expected ohmic losses, Tafel kinetics and purity of hydrogen gas evolved. The results obtained for the in-house membranes are benchmarked against the commercial Zirfon membrane.

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Application of facilitated transport membranes for hydrogen separation in HT-PEM Fuel cell plants

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Fuel cells (FCs) are used in co-generation plants as an environmentally benign and cost effective alternative for the production of heat and electricity. Currently hydrogen is produced via methane steam reforming followed by a Water Gas Shift reactor where CO is converted to CO2 and then is fed to a Pressure Swing Adsorption (PSA) process for purification. PSA systems are complex to run and energy intensive and therefore the use of other separation technologies would be attractive. Compared to normal Polymer Electrolyte Membrane FCs (PEM FC), HT-PEM FC are able to withstand lower H2 purity in the feed and therefore they allow the investigation of less energy intensive and complex separation technologies after the water gas shift reactor. One promising option, is a membrane process using CO2-selective membranes and in particular Facilitated Transport Membranes (FTMs). FTMs show enhanced CO2 separation performances and selectivity compared to conventional solution-diffusion membranes due to the facilitation mechanism; the fixed carriers present in the membrane, typically amine groups, react with CO2 in the presence of water creating an additional specific pathway for the transport of carbon dioxide. The efficiency of the separation is therefore heavily affected not only by the intrinsic properties of the material but also by the management of water in the membrane module and by the operating conditions. The integration of FTMs with HT-PEM FCs is investigated in detail in order to establish the effect of the membrane characteristics on the efficiency of the HT-PEM FC. The influence of various materials and design parameters is evaluated including effective permeance, water content, sweep configuration and operating temperature and pressure. The results are compared to the use of Pd-based H2-selective membranes in the process that has been investigated in the literature.

Carbon capture via high performance ZIF-302/PSF interfacial composite membranes

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Owing to its thermal and chemical durability, mesoporous zeolitic imidazole framework ZIF-302 synthesized via solvothermal process efficiently captures carbon dioxide (CO_2) from flue gas. High performance carbon capturing thin-film interfacial composite (TFIC) membranes were prepared by coating thermoplastic polysulfone (PSF) matrix with vertically aligned array of ZIF-302 via dip coating method. Thin-film interfacial composite membranes prepared through this technique improved CO_2 permeability by adopting shortest transport channels formed in membranes structure. CO_2 transport mainly takes place through mesopores of ZIF-302 via molecular sieving phenomena. As compared to its bare PSF counterpart, ZIF-302/PSF composite membrane improved CO_2 permeability and CO_2/N_2 selectivity by a factor of almost 1.6 and 2.7 respectively. Morphological analysis depicted uniform distribution of ZIF-302 arrays on PSF substrate. Furthermore, the synthesized membranes were found to be thermally stable subjected to flue gas conditions.

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Innovative Direct Air Capture (DAC) technology based on non-dispersive CO_2 absorption using membrane contactors and ionic liquids

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Reaching net-zero greenhouse gas emissions by 2050 is critical to mitigating climate change, as COP26 recently claimed. For this purpose, a set of targets have been established focusing on the decarbonization of industrial and energy sectors. In this scenario, the Direct Air Capture (DAC) technology is presented as a promising strategy to achieve this challenge. DAC is based on directly removing CO_2 from the atmosphere and, permanent storage in geological cavities. Two different approaches are currently being used to carry out this process, emphasizing solid S-DAC or liquid L-DAC sorbents. In this context, L-DAC using amine-based solvents is the preferred alternative for large-scale plants, due to the possibility of continuous operation mode and higher CO_2 capture capacity [1]. However, this technology still presents some troubles like high energy consumption due to regeneration temperature requirements, large equipment needed and solvent loss by evaporation [2]. Thus, this work, presents for the first time a novel approach of L-DAC through the use of non-dispersive absorption using the same ionic liquids (ILs) and membrane contactors previously tested successfully in the research group [3]. First, the design and construction of non-dispersive L-DAC experimental setup was carried out employing a characterized IL (1-ethyl-3-methylimidazolium acetate ([emim][Ac])). Consequently, the CO_2 capture capacity and energy consumption were evaluated at different operating conditions such as (i) gas/liquid flow-rate ratio and (ii) mass fraction of water in the IL at different experiments with a duration of 48 hours. IL characterization shows promising results on solvent stability (90 % integrity up to 500 K), moderate viscosity (40-50 cP) and favorable contact angle (110o-120o). Moreover, preliminary results demonstrate an excellent performance for the intensification of conventional DAC, obtaining a CO_2 capture capacity (up to 9.4 $gCO_2 \cdot L$-1), and lower energy consumption (30% reduction).

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CCSEL Research Infrastructure for evaluation of membranes and barrier materials relevant within the CCUS value chain

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ECCSEL is the European Research Infrastructure for CO_2 Capture, Utilisation, Transport and Storage (CCUS) encompassing interlinked transnational scientific facilities and national nodes. ECCSEL offers open access to over 80 world class CCUS research facilities across Europe. ECCSEL was established in June 2017 as a permanent pan-European distributed research infrastructure, with the main objective of enhancing European science, technology development, innovation and education in the field of CCUS, in order to combat climate change. Great effort has been recently put on CO_2 capture using gas separation membranes, and many examples of membrane developments are found in literature. Through the ongoing ECCSELERATE project, membrane-related infrastructure is being opened for Transnational Access. For example, SINTEF MLAB (NO3.3) is dedicated to fabrication, testing and characterisation of membranes for separation of gases. It offers a broad range of fabrication equipment applicable for producing polymeric, ceramic and metallic membranes, as well as characterisation and test installations for studying the performance and durability of all kinds of membranes. The laboratory includes installations for testing of membrane durability under harsh and poisonous chemical conditions, such as in H_2S [1], SO_2 and NH_3 [2]. The MEMLAB (IT4.1) unit is also devoted to capture, targeting the use of polymeric membranes for CO_2 separation that are characterized from both direct permeation or sorption with pure gases or mixtures. Recently, both infrastructures have been applied to investigate effects of CO_2 on polymeric materials in the CO_2 transport chain [3]. The current contribution will introduce the ECCSELERATE project, and present selected examples of membrane investigations that have applied the ECCSEL infrastructure.

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H_2/CO_2 Separation Using polyPOSS-imide and Hybrid Silica Based Ceramic Multi-Channel Membranes

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Atmospheric CO_2 is becoming a serious global issue. The shift from fossil fuels to renewables is quite slow due to technological barriers. In the meantime, the demand for energy is increasing exponentially making us more dependent on fossil fuels leading to high amount of CO_2 emissions. Therefore, immediate intermediate solutions are essential to address CO_2 emission of fossil fuelled plants fast and in a cost-effective way. CO_2 capture technologies are recognized as one of the direct answers to this problem. However, they are mostly cost- and energy-intensive. In this study, as an outcome of GENESIS project, two advanced materials (polyPOSSimide and hybrid silica) were used to develop and upscale ceramic-supported membranes to reduce costs and increase efficiency of CO_2 capture. In the GENESIS project, iPOSS (polyPOSSimide) membranes are upscaled on multi-channel ceramic-supports by CTI and evaluated in a relevant capture process for pre-combustion in the steel industry. Also, HybSi (Hybrid Silica) membranes are upscaled on multi-channel supports with comparable geometry (7-channel) by TNO and the performance and stability of both membranes were evaluated for H_2/CO_2 separation. The results show a stable performance of both membranes for >250 hours in a simulated COG (Cokes Oven Gas) mixture and a predictable behavior to different process conditions. At 150°C, both the H_2 permeance and selectivity towards H_2 are lower with wet gas mixtures compared to the dry gas mixtures. At 250°C, the hydrogen permeance was found to be 1322 GPU for the iPOSS membrane which is comparable to previous test results with single channel iPOSS membranes, while it was 1606 GPU for the HybSi membrane. The H_2/CO_2 selectivity was 8.0 vs. 6.2 for iPOSS and HybSi, respectively.

Facilitated direct air capture of CO_2 in polysulfone membrane contactors by improving stability and hydrophobicity of amine additives

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Atmospheric CO_2 concentration has become a subject of worldwide concern because it seriously contributed to global warming and climate change, which resulted in serious environmental problems. Several methods have been identified for the removal and capture of CO_2 based on physical and chemical processes: absorption, adsorption, conversion, cryogenic separation, and membrane techniques [1]. In this communication we report the preparation and characterization of asymmetric membranes for CO_2 direct air capture contactors, based on Polysulfone (PSf) and UG20, an additive based on the hyperbranched Polyethyleneimine Lupasol G20, grafted with phenyl isocyanate via urea linkages. Membranes with different amount of additive were prepared by phase inversion precipitation and characterized by several techniques in terms of morphology, porosity, wettability, and stability to aqueous KOH. Moreover, thermal, dielectrical, and mechanical properties were investigated. Finally, CO_2 solubility and mass transfer coefficient were determined and correlated with membranes' performance in the CO_2 capture device. In general, it was found that the presence of the additive greatly improved the characteristics of the membranes as regards the CO_2 capture, especially solubility, hydrophobicity and chemical resistance to the aqueous alkaline absorbent solution, giving the better results for 5% additive content This translated into a greatly improved direct air capture efficiency and robustness, reaching absorption fluxes above 150 mmol(CO_2)/$m^2 \cdot s$, largely outperforming results obtained with another Lupasol G20-based additive, mG20, previously reported [2]. The introduction of urea linkages in the modified polyamine UG20 is shown to be a successful strategy for the design of additives for facilitated CO_2 direct air capture using polymeric membranes.

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Postcombustion carbon capture via nanoporous graphene membranes: a techno-economic analysis

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Membrane processes are emerging as an alternative to absorption for postcombustion CO_2 capture. Membranes based on nanoporous graphene atom-thick selective layer have shown high promise in this regard with extremely large CO_2 permeance (10000 GPU) and attractive CO_2/N_2 selectivity (30) [1,2]. We report a techno-economic model and assess the most performing process configuration for CO_2 capture from wet flue gas (CO_2 concentration of 13.5%). We analyze the role of feed and permeate pressure on energy consumption and capture penalty, by comparing scenarios with compression in the feed channel, vacuum in the permeate channel or a combination of these. When the permeate is under vacuum, the energy consumption corresponding to a certain pressure ratio is lower than when feed is compressed, but the membrane area increases. However, the high permeance of graphene membranes uniquely makes this scenario more competitive than feed compression in most cases (large range of pressure ratio and membrane cost) [3]. Optimizing the double-stage process configuration, we find a minimum capture penalty of 34.3 \$/ton with permeate pressures of 0.05 and 0.1 bar in the first and in the second stage, with membrane module cost of 500 $\$/m^2$. This already promising value of capture penalty can be further reduced by taking into account improved membrane performances, already achieved in our lab. When the selectivity increases to 85, the energy consumption decreases by 25% and the capture penalty goes down to 28.9 \$/ton. Instead, with increased permeance of 15000 GPU, the membrane area decreases by 33% and the capture penalty results 30.6 \$/ton. These results show that graphene membrane-based CO_2 capture is competitive with state-of-art amine absorption and the optimised polymeric membrane-based systems.

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(Per-)fluorinated MOF-based MMMs for CO_2 capture

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Membrane-based gas separation is used in many industrial applications such as H_2 recovery and oxygen/nitrogen enrichment. It is emerging in biogas treatment and is among the most promising technologies for carbon capture. However, the implementation of this technology for large-scale applications is still not economically feasible since the fluxes produced by commercially available membranes are too low for treating large gas volumes. In the last decade, mixed matrix membranes (MMMs) have been recognized as promising next-generation membranes since they can be based on industrially reliable and already commercial polymers blended with inorganic materials with superior performance. In this paper, we show the preliminary results of the "doMino" project, which aims to develop next-generation MMMs based on novel (per-)fluorinated metal-organic frameworks (F-MOFs) for energy efficient carbon capture from flue gas (CO_2/N_2) and biogas (CO_2/CH_4). Two main features of F-MOFs, which make them promising fillers for CO_2 separation using MMMs, drive the choice of these materials: i) fluorine atoms in microporous frameworks are known to enhance the affinity to CO_2 over gases like N_2 and CH_4; ii) hydrophobicity of F-MOFs can be crucial to both make them stable and maintain high CO_2 selectivity when exposed to humid gas streams, ubiquitous in industrial settings. Herein we present the gas transport properties of MMMs prepared from Hyflon AD60x and two perfluorinated Ce-based MOFs having UiO-66 and MIL-140 topology, namely F4_UiO-66(Ce) and F4_MIL-140A(Ce) [1]. The physico-chemical characterization of these MMMs will be presented, as well as a deep discussion of gas transport properties, also as a function of the temperature. We anticipate that the F-MOF-based MMMs show higher CO_2/N_2 and CO_2/CH_4 selectivity with respect to that of the neat polymer, without having a dramatic impact on permeability.

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Membrane, gas hydrate and hybrid technologies for CO_2 capture from flue gases

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Over the past century, the carbon dioxide concentration in the atmosphere has dramatically increased from 275 to 387 ppm and already led to a tangible increase in temperatures on the planet, namely, the average temperature of the Earth's surface in the 21st century exceeds the same value of the 20th century by 0.8 - 1.2°C. If the build-up of CO_2 continues at current rates, by 2060 it will have passed 560 ppm – more than double the level of pre-industrial times. The developed climate models predict that the established trend will negatively affect the global climate by 2100 [1]. As its source is anthropogenic action, nowadays the CCS project is a worldwide accepted strategy under that is about 50 operational facilities today. In that context, special attention should be paid to membrane-based, gas hydrate and hybrid processes which are favorable stands out among the conventional energy-intensive technique based on absorption using the aqueous solutions of amino alcohols. In addition to high energy demand, which tends to 30% of plant generated power, it is characterized by high capital and maintenance costs. In the light of above, a number of technological schemes [2] were designed to overcome the drawbacks connected with phase transition separation processes. The present study deals with CO_2 capture technologies based on membrane and gas hydrate methods. Number of technological schemes were studied through the simulation using the Aspen Plus software, where the special custom models of membrane and gas hydrate units were integrated. Sensitivity and techno-economical study was performed for each plant under the conditions, which allows to achieve both, not less than 90 % CO_2 recovery rate and 95 mol.% CO_2 content in product stream. As a result of present complex study, three, membrane, gas hydrate and the hybrid technologies were proposed for efficient CO_2 capture.

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Wastewater treatment and resource recovery in the food industry by membrane technology

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The food sector plays an important role in ensuring the general people's good health and nutritional needs by maintaining food security and food safety. However, the growing worldwide population has increased the need for food, but at the same time, there has been a significant increase in the output of waste generation of the food industry, which represents an ultimate challenge. Mainly, two types of wastes, liquid waste (wastewater) and solid waste (food waste), are observed on a large scale, but by treating these wastes, various potential benefits could be obtained in terms of clean water production and resource recovery, respectively. Therefore, in the current paper, novel electrospun nanofibers membranes have been applied in treating wastewater and pressure-driven membrane filtration used for resource recovery by modulating their process intensification. At first, the authors, using an environmentally friendly approach for eliminating methylene blue from an aqueous solution, have developed a unique electrospun nanofiber membrane made of a combination of polyethersulfone and hydroxypropyl cellulose (PES/HPC). The highest adsorption capacity was found to be 259.74 mg/g at neutral pH under room temperature, and the pseudo-second-order model was found to be accurate. Secondly, the nanofiltration process has been applied to the potential of organic waste streams (i.e., food waste) for the sustainable production of precursor chemicals such as volatile fatty acids (VFAs) using anaerobic digestion (AD) within a consortium of resource recovery. Experiments were carried out using two commercial nanofiltration membranes of 200-300 Da and 300-500 Da under various pH, applying constant pressure and temperature of 15 bar and 20-21oC, respectively. As noticed, the membrane with the molecular weight cut-off (MWCO) of 200-100 Da appeared to be more effective with an increased concentration of total VFAs (16.94 g/L) and recovery percentage above 90% at pH 9.

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Polystyrene nanoplastics removal from urban wastewater by aerobic membrane bioreactor

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Micronanoplastics as emerging contaminants have become an increasing environmental concern given their persistent and ecotoxicological effects on the aquatic environment. Although the retention capacity of identified microplastics in wastewater treatment plants (WWTPs) is ranged between 98 % - 99 % [1, 2], WWTPs are still considered the main pathway for microplastics to get into the aquatic environment. In this context, membrane-based advanced technologies for wastewater treatment, such as membrane bioreactors (MBRs) have been demonstrated to reach a removal capacity of 99.9 % of microplastics (MPs). However, major efforts have been devoted to studying the removal of MP debris (> 250 µm) [3], whereas the fate of nanoplastics (NPs) (< 1 µm) has been scarcely investigated. The present work aimed to evaluate the removal capacity of NPs by an aerobic MBR of submerged-membrane configuration. For that purpose, two systems of 30 L, with an effective membrane area of 0.11 m^2 each have been employed. One reactor was used as a control (i.e., without polystyrene (PS) addition), and the other one with the addition of PS nanospheres (120 nm). The experimental conditions of the reactors have been set at continuous operation with a steady-flux value of 18 LMH, applying cycles of 8 min of suction followed by 2 min of relaxation. The effects on the microbial community (i.e., bioaccumulation and toxicity) in presence of NPs, fouling of the membranes, as well as the quality of the MBR effluent, are being studied. A rigorous protocol of cleaning is being arranged to minimize possible air- and cross-contamination. Overall, this work fills some knowledge gaps in relation to the identification and quantification of NPs and will provide important insights into NPs removal by advanced MBR technology.

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Validation of recycled nanofiltration and anion-exchange membranes for the treatment of high salinity urban wastewaters for crop irrigation

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One of the unconventional alternative sources to tackle the problem of water shortage is the use of reclaimed water from wastewater treatment plants (WWTPs) for irrigation purposes. To improve and reach the water quality established for irrigation in terms of salinity, the secondary-treated wastewater effluents from WWTPs must be further treated with advanced technologies such as membrane-based processes. However, the management of the end-of-life membranes is still based on a linear model, which assumes the tendency to discard the membranes once their separation capacity is unrecoverable [1]. Under this framework, in this work, recycled Nanofiltration (rNF) membranes and Anion-Exchange Membranes (rAEM) obtained from end-of-life reverse osmosis membranes were employed to evaluate their application capability in saline wastewater treatment. Both recycled membranes (rNF and rAEM) were applied in crossflow filtration and electrodialysis (ED), respectively. The results obtained in the NF process were similar to the separation capacity of commercial NF90 membranes in terms of permeability and total salt rejection, obtaining a high selective rejection of divalent ions (i.e., SO_4^{2-} (> 96 %); Ca^{2+} and Mg^{2+} (> 93 %)). In the case of the ED process, the comparison between rAEM and commercial anion-exchange membranes (Ralex®) showed a suitable demineralization rate for irrigation of crops without compromising the power consumption. The quality of both system effluents was evaluated by comparing the quality of the water reused for irrigation, which was based on the World Health Organization guideline [2], and validated by studying lettuce cultivation for 7 weeks. No significant differences were observed in individual macronutrients (i.e., total N, P, Ca, and Mg) of leaves of the lettuce of each treatment studied. Therefore, the implementation of recycled membranes could be considered a promising approach in real applications, enabling the valorization of wastewater for irrigation purposes, and increasing the sustainability of water separation processes.

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Reusable composite membranes for highly efficient chromium removal from real water matrixes

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Nowadays, more than 1.5 million deaths are directly or indirectly associated with health problems from water pollution. Natural or industrial hexavalent chromium water pollution remains an unresolved, worldwide threat. There is intense research on new active and cost-effective sorbents for Cr(VI), but most still exhibit a critical limitation: their powdered nature makes their recovery from water cost and energy-consuming. In this work, Al(OH)3, MIL-88-B(Fe), and UiO-66-NH_2 Cr(VI) sorbents were immobilized into a poly (vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) polymeric substrate to develop an easily reactivable and reusable water filtering technology. The immobilization of the sorbents into the PVDF-HFP porous matrix modified the polymeric matrix's macro and mesoporous structure, tuning its wettability in parallel. Although a partial blocking of the Cr(VI) adsorptive capacity was observed for Al(OH)3 and MIL-88-B(Fe) when immobilized into composite membranes, PVDF-HFP/UiO-66-NH_2 filter (i) exceeded the full capacity of the non-immobilized sorbent to trap Cr(VI), (ii) could be reactivated and reusable, and (iii) it was fully functional when applied in real water effluents.

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The gases emissions by raw and membrane-treated vinasse applied into soil

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Vinasse is rich in nutrients, particularly nitrogen, phosphorus, and potassium, besides a high organic carbon load, reasons which it is considered for fertigation. With this composition, another alternative for the disposal of this residue is its transformation into organo-mineral fertilizer (FOM). The FOMs, recognized by the higher nutrients' composition, stability, and uniformity, allows greater use of the fertilizer in the soil and has advantages for the soil such as the increase of its organic matter, cation exchange capacity (CEC), water storage capacity, besides an improvement of its structure and aggregation capacity. Besides, the concentration of vinasse nutrients by integrated membrane separation processes such as ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) can reduce the vinasse volume to be transported in fertigation and make a nutritionally richer fertilizer. However, the two ways of disposing of vinasse can bring harm to the environment such as emissions of gases such as SO_2 and CO_2, which can contribute to global warming. This study aimed to estimate the emissions of these gases in the use of raw and treated vinasse, by the UF, NF, and RO, both in the application via fertigation and as FOM. For fertigation was analyzed a vinasse application rate (VAR) of 184 m^3/ha for raw vinasse and vinasse concentrates of 115 m^3/ha for UF, 136 m^3/ha for RO and 133 m^3/ha for NF. For FOM the VAR estimated was 12 m^3/ha for raw vinasse and its concentrates. In all scenarios analyzed, the CO_2 emission was greater when raw vinasse was considered given its greater organic matter concentration and similar observations can be made for SO_2 emissions. In addition, in all scenarios analyzed, the emissions generated by fertigation were higher when compared to those generated by the application via FOM.

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Toward Sustainable and Cost-Effective Production of Drinking Water from Eutrophic and Micro-Polluted Water Using the PAC-UF Membrane Hybrid Process

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So far, the hybrid process of powdered activated carbon adsorption and ultrafiltration (PAC-UF) is not considered the most suitable drinking water treatment process in case surface water is contaminated with both, microalgae and micropollutants, because irreversible fouling accrues atop of the membrane, which is cost effective to remove. Often, a number of conventional treatment methods are employed, including flocculation processes or adsorption on activated carbon, a material that is in many cases produced from fossil raw material. Replacing conventional technologies with the PAC-UF hybrid process, combined with the usage of PAC produced from drinking water sludge, offers a cost-effective and sustainable solution. Four sludges were collected from different drinking water treatment plants in Germany. The sludge samples, with different elemental carbon content, were activated at various conditions in a rotary kiln. The produced sorbents were tested for their micropollutant adsorption capacity using selected model compounds (i.e., caffeine, benzophenone, benzophenone-3, ibuprofen, diclofenac, triclosan), and compared with conventionally available PAC. Benzophenone and benzophenone-3 were found to be removed using the produced sorbents to the same extent as with the commercial PAC. A good removal of caffeine was also measured, while the other micropollutants were hardly eliminated. A semi-pilot plant was constructed to examine the treatment of microalgae-contaminated water employing the PAC-UF hybrid process. The motivation was to investigate whether irreversible membrane fouling can be reduced or avoided by a combination of PAC and coagulation, combined with an improvement of the permeate quality. Based on previous expertise, a series of jar tests was performed using ferric chloride and polyaluminium hydroxide chloride to determine the optimal coagulant dose, pH, and G-values. Membrane filtration experiments were conducted, whereas the impact of different operational parameters (e.g., PAC concentration, PAC dosing rate, and feed flow rate) on the performance of the hybrid process was studied.

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Development of an innovative PVDF based hollow fiber membrane with incorporated graphene oxide to combine ultrafiltration and adsorption

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In the frame of Graphil project, European project aiming to develop innovative graphene related material, Polymem has developed a PVDF hollow fiber composite membranes incorporating Graphene Oxide (GO) nanoparticles. This innovative membrane allows to combine suspended solids and virus removal with a UF cut-off and at the same time an adsorption of micropollutants thanks to the graphene oxide. This membrane could be used advantageously inside domestic and outdoor ultrafiltration cartridges. As the contact time of water and graphene oxide would be increased drastically if the GO flakes are small and perfectly dispersed in the porous structure of the membranes, a better adsorption performance is expected with this all-in-one composite membrane compared with the coupling of an adsorption bed followed by a filtration on membrane. To obtain such membrane, the dispersion and quantity of GO has been studied. A PVDF/GO hollow fiber has been prepared by a Nonsolvent Induced Phase Separation (NIPS) technic. GO nanoparticles dispersion has been studied and the combine effects of the stirring and the sonication of the dope solution investigated. Dope composition and membrane spinning parameters has been finely tuned to optimize the membrane characteristics and to fabricate membranes at industrial scale. Geometry (thickness, macro voids), filtration performances (pure water permeability, virus retention), adsorption performances (ciprofloxacin, PFAS and heavy metals removal) and stability (mechanical resistance, ageing tests) have been measured and the performances of these membranes evaluated.

Effectiveness of hybrid Adsorption/Coagulation/Ceramic MF towards contaminants of current and emerging concern in drinking water

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Resilient, advanced water treatment is crucial to face increasingly limited water resources and challenging water quality requirements. Even optimized, conventional water treatment has limitations against a growing number of synthetic and natural organic microcontaminants in water sources and for ensuring disinfection with minimal disinfection by-products' formation. Pressurized powdered activated carbon/coagulation/ceramic microfiltration (PAC/Alum/MF) was investigated at pilot scale for treating low turbidity and low natural organic matter (NOM) surface waters spiked with organic microcontaminants of current or emerging concern. A total of 11 trials with clarified or non-clarified waters spiked with pesticides, microcystins, or pharmaceutical compounds were conducted to assess the removal of these microcontaminants, NOM (as A254 nm absorbance, A254, and dissolved organic carbon, DOC), trihalomethane formation potential (THMFP), aerobic endospores as protozoan (oo)cysts indicators (chlorine-resistant), bacteriophages as virus indicators, and regular drinking water quality parameters [1]. PAC/Alum/MF achieved high microcontaminants' removal, between > 75% and > 97% for total-pesticides, microcystin-LReq and total-pharmaceuticals, with 4–18 mg/L of a mesoporous PAC and 2 h minimum contact time. Microcontaminants showed different amenabilities to PAC adsorption, much depending on contaminant key properties, such as charge, hydrophobicity (Log Kow), polar surface area, and aromatic rings count. Compounds less amenable to adsorption showed higher vulnerability to NOM competition, more expressive in non-clarified, A254-rich waters, and benefited the most with the DOC-normalized PAC dose (mgPAC/mgDOC) increase. PAC/Alum/MF was a full barrier against aerobic endospores (protozoan indicators) and PAC dosing was crucial for removing bacteriophages (≥ 1.1-log reduction). Both PAC/MF and Alum/MF attained a considerable NOM removal (14–30%, median), important for reducing the THMFP (22–27%, median) and, in the case of Alum/MF, also important for controlling membrane fouling. In PAC/Alum/MF PAC complemented coagulation, adding 15–19% to NOM median removal, whereas no substantial improvement was observed towards THMFP and membrane fouling control.

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Decentralized water treatment by low pressure recycled reverse osmosis

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Although essential for human development, the safe supply of drinking water depends on advanced treatment processes, often prohibitive for low-income countries and isolated areas. In this sense, procedures for recycling residential end-of-life reverse osmosis (EoL-RO) modules were investigated, which were validated in a long-term operation for groundwater treatment. The devices developed were characterized by ease of use, low maintenance, and independence from the electrical energy supply. Furthermore, the advantages of using recycled membranes can be extended to environmental aspects, as it avoids the simple disposal of EoL-RO membranes in sanitary landfills. A survey of imported materials and membrane inventories in Brazil predicted that around 1,800 t of reverse osmosis modules would be disposed of in the country by 2024. From a global point of view, it is estimated that the disposal rate of EoL-RO membranes would reach over 14,000 t annually. On the other hand, great challenges also generate great opportunities. Monitoring data demonstrate constant permeate flux (21.2 ± 0.46 L/m^{2}h), and filtration resistance (6.37 ± 0.16 × 1014 1/m), yielding a total permeate volume (3,000 L) comparable to commercial filtration systems and complying with potability standards (109 parameters analyzed in total). The fouling, even observed by the FTIR, SEM, and EDS techniques did not compromise the system's performance. As a low-cost and decentralized technology, recycled modules can be considered for point-of-use applications or even in remote regions and developing countries.

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F-rich groundwaters in Calabria Region (Southern Italy): from sources to remediation

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Fluorine is commonly present as fluoride ion (F-) in drinking waters and represents an essential costituent for human metabolism; however, an excessive intake of fluoride via ingestion of contaminated water could cause several negative effects on human health. Fluoride is widespread into the environment as a result of water-rock interaction processes. Commonly, the main F-bearing minerals like fluorite, muscovite, biotite, apatite are present in considerable quantities into crystalline rocks in which the F concentration ranges from 20 to 2700 mg/kg [1]. The present work represents a multidisciplinary study to understand the conditions of F release into the environment, exploiting the geochemical approach for the targeted of the materials and operating conditions to apply during the water treatment. A geochemical modelling was elaborated reproducing satisfactory the chemistry of several water representing the studied crystalline aquifers [2]. Two water samples (labled Pc and Bg) were classified as a F enriched Na-HCO_3 type of water. In detail, the following F-concentrations were observed: 8.9 mg/L and 30 mg/L for the Bg and Pc samples, respectively. Based on the acquired geochemical characterization knowledge, Bg and Pc samples were treated by two thin-film composite NF membranes, namely SPR 10114 and SPR 10113 which are not yet tested for water defluoridation. The result showed that the SPR 10114 membrane was able to lower F- contents below the threshold value of 1.5 mg/L (WHO value) for both treated waters, whereas the fluoride content remained above the threshold value when the Pc sample was treated using the SPR 10113 membrane [3]. The obtained permeates can be classified by a low ionic load waters and are not suitable for long-term consumption as drinking water. However, all of the produced waters did not need any further re-mineralizing processes to be used as agricultural or other purposes.

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Graphene oxide membrane on Nylon support for PFAS removal from water by filtration

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Emerging contaminants have generated great concern in recent years. Recently, one of the most alarming is per- and poly-fluoroalkyl substances (PFAS). Due to the stability of C-F bonds, their removal from water is complicated [1-2]. Graphene oxide (GO) has been proposed in recent years as a promising material for membrane preparation since its properties can be adjusted by chemical modifications such as reduction or oxidation [3]. GO membranes with different load (0.04-0.06 $mg \cdot cm^{-2}$) and different GO solution concentration (2.5-40 $mg \cdot L^{-1}$) were prepared in this work by pressure-assisted filtration. Membranes were subjected to different chemical treatments: ozone oxidation and/or UV radiation reduction. SEM, XRD, and XPS were used for membrane characterization. Filtration tests were carried out in a cross-flow device using 2 $mL \cdot min^{-1}$ of 10 ppm solutions of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS) at 2 bar. Membrane fabricated with a GO suspension concentration of 10 mg/L and a GO membrane loading of 0.04 $mg \cdot cm^{-2}$ showed the highest PFAS rejection, reaching 80 % for PFOA and 90 % for PFOS, with flux of 4.2 and 3.2 $L \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$, respectively. Membrane preparation conditions didn't alter significatively the oxygen groups distribution but vary the interlayer spacing between 8.1 and 8.5 Å with a result in decrease of PFOA and PFOS rejection. Oxidation by ozone led to a modification of oxygenated groups distribution in GO membranes (XPS analysis), while reduction led to a decrease in atomic percentage of oxygen (31 to 20 %). XRD analysis showed a decrease in interlayer spacing of membranes after both treatments, from 8.1 to 7.8-7.7 Å. Among chemical modifications, the combination of reduction and oxidation led to the most significant changes in membrane performance, increasing flux up to 9 $L \cdot h^{-1} \cdot m^{-2} \cdot bar^{-1}$, but with a decrease in rejection (45 %).

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Prepare of layer by layer polymer membrane for high permeation rate separation membrane for water purification

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Demand for clean water is increasing worldwide, and accordingly, the demand for water purifiers at home is increasing. However, a tubular membrane is mainly used in filters used for home use, and the tubular membrane has a disadvantage in that the removal rate is lower than that of the reverse osmosis membrane. In addition, in order to use a reverse osmosis membrane, a module that is 6 to 7 times larger than the existing one is used, so the miniaturization that can be used in home water purifiers is not achieved. In this study, we tried to develop a polymer membrane that can be used for water purification by coating the layer by layer polymer membrane, and we tried to develop a layer-by-layer polymer membrane by preparing membranes according to various materials and coating them. As a characteristic evaluation, the mechanical strength, permeability, and removal rate of the membrane were measured to determine the performance of the membrane for water purification.

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Laboratory Pilot of SophiA Water Technologies for Drinking and Deionised Water

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The European Union's (EU) Horizon 2020 project 'SophiA – Sustainable Off-Grid Solutions for Pharmacies and Hospitals in Africa' aims to enable African people access to safe and clean drinking water, off-grid carbon-neutral electricity and heating and cooling of food and medicine, increasing quality of life in a sustainable way. It will be demonstrated at 4 rural health facilities – Burkina Faso, Cameroon, Uganda and Malawi covering 4 different climate and geographical regions across the African continent. Broad implementation of SophiA systems comprise drinking and deionised water production system, cooling system and steam generation system that will bring vast environmental, economic, social and especially health benefits. The concept of water treatment system is based on two stages of low-pressure technologies, namely ultrafiltration (UF) to address microbial safety and membrane capacitive deionization (MCDI) for water softening. Polyethersulfone (PES) membrane modules called Cube® mini and FM6® of 0.45 m^2 and 6.25 m^2 active filtration area respectively were manufactured by Martin Systems GmbH for fouling tests at laboratory scale for filtration of suspended impurities. Further, MCDI module C12 manufactured by Voltea® was tested for removal of fluoride from model drinking water and production of deionised water with electrical conductivity below 20 µS/cm. The developed laboratory pilot serves as the groundwork to SophiA's containerised scheme of water treatment system which integrates both UF and MCDI technologies equipped with sensors for monitoring and autonomous operation. The innovative, affordable and efficient SophiA renewable energy solutions will support Africa in achieving sustainable development growth and economic transformation. SophiA systems will be developed by a multi-national interdisciplinary team of 13 partners well balanced between academia and industry, and Africa and Europe.

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Perspectives of the Extracorporeal Membrane Oxygenation from the Mathematical Modelling

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Extracorporeal membrane oxygenation is a life-saving critical medical equipment for cardio-pulmonary support, in the case of severe chronic respiratory distress. The equipment performance is dependent on the type of membrane, flow configuration, blood physiology, etc. A lot of research and innovation is focussed on the development of novel membrane material. However, there are two other major challenges affecting the performance of these systems – flow arrangement and the nature of the flow coupled to the blood physiological conditions. The oxygenator design originally involves flow of blood through the lumen of the thin-walled hollow fibres (intra-luminal flow ILF) and oxygen (or oxygen enriched air) in the outside [1]. Increasing the blood flow in the ILF reduces the oxygen saturation, and the local partial pressure of O_2 diffusing into the fibres in affected by the fibre arrangement [2]. On the contrast, it has been observed that the oxygen transfer efficiency is improved with the oxygen flowing inside the lumen and the blood outside the hollow fibre (extra-luminal flow ELF) [3]. This is primarily because of the increased flow convection in the blood side resulting in higher mass transfer coefficient. But with increased shear, there is a possibility of haemolysis. The pulsatile nature of the flow and the blood physiological conditions is also important, which was not explored significantly. The impact of the frequency of the pulsatile flow on the oxygenation profile is also relevant. The effect of the blood haematocrit factor on the hydrodynamic and diffusivity properties, is significant as it affects the rheology and the haemodynamic conditions. The present effort in understanding the process, can lead to improved performance and design of this life-supporting equipment. The results provide clear recommendations from the application perspectives, and helps in better design, performance prediction and control of the critical life saving device.

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Characterization under dynamic conditions of cellulose acetate-based monophasic hybrid membrane for improved blood purification devices

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The preparation of integrally skinned asymmetric CA membranes by means of the wet-phase inversion technique paved the way to the synthesis of membranes with a multitude of structures ranging from ultrafiltration to reverse osmosis as well as for the development of low-flux hemodialyzers and high-flux hemodialyzers. Nevertheless, CA membranes exhibit drawbacks such as limited chemical resistance, low mechanical strength, low shelf life, and small pore size. To overcome these limitations, our research group has focused on the development of novel monophasic hybrid CA-based membranes, which combine the high mechanical and thermal stability of the inorganic material with the flexibility, ductility, and processability of CA [1]. In this work, a modified version of the phase inversion technique coupled with the sol-gel method was used to synthesize novel integral asymmetric monophasic hybrid amine-functionalized CA-SiO_2-$(CH_2)_3NH_2$ membranes [2, 3]. For comparison purposes, a integral asymmetric CA membrane was also prepared and characterized. The morphological characterization of membranes was performed by scanning electron microscopy (SEM). Permeation experiments were performed in an in-house-built single hemodialysis membrane module (SHDMM) under dynamic conditions. The membranes were characterized in terms of hydraulic permeability (Lp), molecular weight cut-off (MWCO) and the rejection coefficients to urea, creatinine, uric acid, and albumin [3]. SEM confirmed asymmetric cross-section structures composed of a very thin dense layer (< 1 µm) supported by a much thicker porous layer. Permeation studies revealed that the Lp of the CA-SiO_2-$(CH_2)_3NH_2$ membrane was 66.61 kg·h−1·m−2·bar−1, 1.8 times higher compared to CA membrane (37.09 kg·h−1·m−2·bar−1). MWCO increased from 18.1 kDa for the CA membrane to 24.5 kDa for monophasic hybrid membrane. Moreover, the CA-SiO_2-$(CH_2)_3NH_2$ membrane fully permeated urea, creatinine, and uric acid while completely retaining albumin and long-term filtration studies of albumin solutions suggested that fouling does not occur at the surface of the CA-SiO_2-$(CH_2)_3NH_2$ membrane [3].

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Controlling Polylactide Layer Parameters Covered Hyaluronic Acid-Alginate Structures in Drug Carriers

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Using a controlled-release drug carrier is an innovative solution for treating local infections, e.g. in dentistry. The biocompatibility, biodegradability, possibility of a large amount of drug adsorbed (especially those with hydrophilic properties), and ability to create structures of any shape and size cause hydrogels to be frequently studied. The main disadvantage of hydrogel carriers is the rapid rate of drug release. The answer to reducing the mass flux typical for hydrogel structure is to surround it with a polymer layer working as a selective membrane. The work presents hydrogel HA-SAL carriers covered with a layer of polylactide (PLA) using Caleva's Dry Cover (Dorset, UK). PLA solution with a concentration of 1.5 w/v was prepared in methylene chloride. The process temperature was changed in the range 45-55°C. Capsule coating was carried out for 10-30 minutes. The quality of the obtained PLA membrane (thickness, pore size) was evaluated by SEM. Transport properties were tested on selected antibiotics: amoxicillin and doxycycline. It allowed specifying optimal conditions for the PLA membrane preparation. Finally, the carriers were tested on the pathogenic strain Enterobacter faecalis.

Hydrogen Production by Ammonia Scission

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The Process of Hydrogen production by direct NH_3 scission is a real innovation technology. It is developed in a special Plasma Torch reactor, without wastes or polluting emissions. Hydrogen is really clean, because it is obtained without CO_2 production. Inside the Plasma Torch reaction unit, a special membranes package must be installed, to separate immediately H_2 and N_2 gases. This installation is very easy as process engineering, but it is very important the right construction of the Plasma Torch reactor, that need expertise engineers to design mechanical structure. It is necessary to have a right supplier to build this special Plasma Torch Reactor, in all over the world there are few Companies, that are able in these kinds of constructions.

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Monophasic coupling of nanofiltration and O_3/H_2O_2 advanced oxidation process

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The ubiquitous presence of organic contaminants (TrOCs) in wastewaters and the environment is a major concern for freshwater production as well as water reuse. Among the available techniques for TrOCs removal, nanofiltration (NF) and advanced oxidation processes (AOP) have emerged as suitable separative and destructive techniques respectively. The aim of this study is to develop a hybrid process coupling NF and an ozonation AOP. An original configuration was adopted, consisting of mixing pre-ozonated water with the water to be treated shortly before entering the membrane cell. This single-phase configuration makes it possible to avoid direct bubbling of ozone gas into the membrane cell in order to preserve its integrity, thus allowing the selection of polymeric membranes that are less expensive than ceramic membranes. The aim of this study was to select the most adequate polymeric membrane material based on their ozone stability and to evaluate the performance of the proposed coupling in terms of TrOCs elimination. In the first part of the study, polyethersulfone (PES) and polyamide based NF membranes were exposed to O_3 (10 ppm for 1 h) at pH 3 (favoring molecular ozone oxidation) and 7 (favoring hydroxyl radical production resulting from O_3 decomposition). A thorough characterization of membrane physicochemical and morphological properties before and after exposure has demonstrated the good stability of PES to ozone contrarily to polyamide. In the second part of the study, the monophasic coupling of nanofiltration (using PES membrane) and O_3 (with and without H2O2) has been studied at lab-scale for the removal of carbamazepine, sulfamethoxazole and deethylatrazine (used as HO· radical probe) from drinking water and surface water. The proposed monophasic configuration allowed an intensified hydroxyl radicals production, a fouling reduction as well as a protection of the membrane surface to ozone degradation.

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Life-cycle impact assessment of end-of-life reverse osmosis membranes: method selection

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Desalination has become an integral part of water management worldwide. As any other process, it causes environmental impacts, including the disposal of end-of-life reverse osmosis (EoL-RO) membranes. To move desalination towards more sustainable practices, life cycle impact assessment (LCIA) is an important tool to compare different disposal options for of EoL-RO and their associated impacts. This allows organizations to use that information to intervene in positive ways in the membrane life-cycle, and also invest into new processes. However, the vast majority of LCIA models were developed in European countries, USA, and Canada, which prevents countries outside those regions from producing LCIA results with specific characteristics. Hence, the present study aims to analyze different models in the Brazilian LCA context, in order to help understand how this regionalization sets specific to which model. To this, the results of six different LCIA methods (ReCiPe 2016, IMPACT 2002+, IMPACT World +, EDIP 2003, CML-IA, and ICLD Midpoint 2011+) were compared in terms of common categories, namely global warming and ozone depletion. To evaluate if there was any significant difference between the methods, the Monte Carlo simulation (1000 runs) was applied. Then, the respective distribution functions were compared using the non-parametric Anderson-Darling test. To run comparison, statistical tests were performed using Python and Sci-Py. It was found that CML, ILCD, and Impact 2002+ did not present any significant difference at a confidence level of 95%, for the ozone depletion category. By using the box-plot visualization, it was also seen that the distribution function for Impact 2002+ was distant from the other two for the global warming category, narrowing down the options for CLM-IA and ILCD. Finally, since CML-IA presents worldwide application and was amongst the recommended methods for the Brazilian context by Mendes [1], it was the method chosen to perform further LCIA tests.

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Plasmonics phenomena in membrane technology

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Membrane distillation (MD) is a promising separation technology for treating saline water and wastewater with high rejection factors [1]. In addition, MD technology has the potential to produce "green" results and provide solutions to environmental problems. Currently, performance of conventional MD systems is affected by temperature polarization which reduces the feed temperature at the membrane surface involving a decrease of the driving force to water evaporation and a reduction of the overall efficiency of MD. Due to these negative effects, the technological applications of MD are still limited. In this context, the advent of thermoplasmonics, an emerging field in photonics which aims at harnessing the kinetic energy of light to generate heat at nanoscale, can offer interesting prospects. Localized surface plasmon resonance (LSPR) supported by nanostructures greatly improves the interactions between light and structure. By engineering the size, morphology and composition of nanostructures, the absorption of light can be triggered and maximized, resulting in a fully controllable nano-source of heat [2]. In this work, we explore the impact of thermoplasmonics in membrane technology with special emphasis on the thermoplasmonic effects promoted by photothermal excitations of advanced nanomaterials incorporated into microporous polymeric membranes.

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A tunable fabrication method towards catalytic polymer-based porous multi-composite membranes with promising properties for pollutant degradation and hydrogen production

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Porous catalytic composite materials are fundamental for promising solutions to current challenges such as green energy production or pollution reduction. A prerequisite are methods for incorporating multiple different functional components into a stable material that exhibits good mass transport properties. Here, we present a new approach for the fabrication of catalytically active multi-composite membranes that incorporate nickel nanoparticles, carbon nanoparticles and a cationic ionomer. Based on the film casting and subsequent phase separation of solutions of the matrix material polyethersulfone in presence of all other components, we were able to prepare porous membranes with tuneable morphology and chemical compositions. Via elemental analysis a complete integration of the components was confirmed, and additional EDX mapping and SEM images showed a homogeneous distribution of the different components. We further managed to control the pore structure and pore size distribution by using different phase separation conditions and additives. The composite membranes exhibit a high porosity of over 70% and an extraordinarily high stability in 6M KOH and at elevated temperature over 30 days. All these properties make our new membranes promising candidates for various catalytic applications. We could show high activity for the reduction of p-nitrophenol in water in batch mode and observed a significant synergy between the different components. We found that at constant nickel catalyst content anisotropic morphologies as well as an increase in carbon or ionomer content led to higher catalytic activity. Additionally, we used our materials as flow-through membrane reactors for p-nitrophenol reduction and achieved an about 1000fold increase in activity compared to the batch mode as well. Based on the promising results in this model study, ongoing work is devoted to tune the novel membranes for a catalytic membrane contactor for hydrogen recovery from the storage material NaBH4 and as catalytic electrodes for hydrogen production via water electrolysis.

Intensification of the antisolvent crystallization process using membrane technology

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The control of antisolvent crystallization is a perplex that has not been resolved yet. To date, the process relies on the addition of downstream processing units such as grinding. Membranes, on the contrary, have the ability to control the transport of the antisolvent and provide excellent mixing that inhibit the formation of local supersaturations that are responsible for the random properties of the resulting crystals. In a previous work, we demonstrated that membranes can indeed provide in one step, a narrow crystal size distribution and a uniform shape; also, membrane properties like hydrophobicity, thickness and porosity play an important role in controlling the crystal size, and crystal size distribution [1-2]. We noticed that the flow rate had an impact on the hydrodynamics of the crystallizing solution, so this begs the question, what other operating conditions impact the crystallization process? This work investigated – besides the impact of flow rate – the impact of antisolvent composition, the temperature and gravity. Results were impressive such that in any condition, membranes were consistent in providing a narrow CSD. The prism-like shape of glycine crystals was maintained as well, but slightly altered when operating at a temperature of 35 °C. Finally, for the crystal size and the crystalline structure, it was demonstrated that the size does not change significantly ca. 5 um, and the monoclinic form was perfectly maintained in all conditions. The study shows that membrane is indeed a robust technology that can offer one-step uniform crystal properties. Most of all, it demonstrates from a kinetic perspective, that operational time can be extended given a specific combination of the four factors —flow rate, antisolvent composition, temperature or gravity. This membrane function is much-needed for the formation of organic compounds overall, particularly heat-sensitive ones as in pharma- and agro-industries, saving energy, time and footprint.

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Continuous and intensified membrane crystallization process: Quality control of an active pharmaceutical ingredient

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Crystallization from solution is a solid-liquid separation technology based on obtaining a solid phase from a supersaturated solution by means of a change of state. Supersaturation is the driving force of crystallization. It is achieved by various means, mainly antisolvent addition and solvent evaporation, and can have an impact on the final product quality [1]. Thus, controlling the supersaturation is a promising alternative for achieving the desired product characteristics in terms of size, size distribution, polymorphism, shape, specific surface area, density, purity, and stability [2]. In this aim, this project seeks to develop a continuous, easily scalable, and fully modular membrane crystallization process using membrane modules allowing fine control of supersaturation, while minimizing industrial risks and environmental footprint, in particular by reducing the amount of solvent used.

Owing to their large and known exchange surface, membrane processes allow fine local control of the mass and heat transfer, and consequently control of the supersaturation. Two processes will be investigated involving different supersaturation methods: the addition of an antisolvent and selective evaporation of the solvent. The first part of this project focuses on the second method, the good solvent is extracted from a supersaturated solute/solvent/antisolvent mixture by selective evaporation through a composite membrane by pervaporation. This technology offers temperature flexibility, high selectivity, and non-sensitivity to wetting [3]. The initial stage involves the control of paracetamol polymorphism. Once the desired polymorphic form is obtained, the final product quality in terms of crystal size distribution, crystal shape will be assessed . Meanwhile, the process performance will be evaluated by the crystallization yield, permeate flux and membrane selectivity with a particular focus on fouling, concentration and temperature polarization.

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