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Prof. Dr.-Ing. Matthias Wessling

Florian Pradellok, Malte Wehner, Susanne Offermann

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The proceedings of the 19th Aachener Membran Kolloquium contain the articles and/or abstracts to all oral and poster presentations. The authors are responsible for content and layout of these.

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Preface – Proceedings AMK 2024

Welcome to Aachen. Welcome to the 19th Aachener Membran Kolloquium.

Already for the 19th time, we are happy to bring the membrane community – end-users, technology providers and academics – back together in Aachen for the AMK to discuss the newest developments in product and process development. As we strive to tackle the grand global challenges such as energy and water supply, health and climate control, we are well advised to foster synergies between the key players in industry and academia. It is the mission of AMK to be a hub for experts from different fields, to spur creativity and to initiate co-operations. In this spirit, we are looking forward to an inspiring conference.

The present proceedings contain the abstracts and papers of the oral and poster presentations, giving you a summary of the topics and providing detailed background information. You will also find contact details for all companies participating in the industrial exhibition.

We are glad to announce our two keynote speakers: Tim Merkel, Vice President of Technology at Membrane Technology and Research Inc., will talk about the experience of MTR to bring carbon capture membranes to a commercial scale – a technology which will remain highly relevant even after complete replacement of fossil fuels. Over 15 years the collaboration of MTR and the U.S. Department of Energy advanced the capture technology from concept to the cusp of commercialization. What are the future research directions and critical challenges for developing efficient carbon capture processes?

Fausto Gallucci, Full Professor at Eindhoven University of Technology, will present his results on process intensification using membrane reactors in chemical production. A topic of high significance as process intensification in the chemical industry can lead to an increase in energy efficiency and hence a decrease of anthropogenic CO₂ emissions. The current research focuses on the use of membranes for dehydrogenation reactions and for chemical production. Is process intensification the key to a more efficient chemical industry?

The Aachener Membran Kolloquium is the only international conference where industrial lectures prevail, where industry meets and inspires academia and vice versa on the full breadth of membrane applications. We work hard to continuously shape the AMK such that it remains one of the leading international conferences on industrial membrane applications.

We succeeded in composing a well-balanced and exquisite program that will hopefully convince you to rejoin our Aachener Membran Kolloquium in the future. Until then, enjoy the lectures, the poster session, the industrial exhibition and the accompanying social events.

We thank you for your open and inspiring contributions.

Matthias Wessling

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Univ.-Prof. Dr.-Ing. habil. **T. Wintgens**
Institut für Siedlungswasserwirtschaft
(ISA) der RWTH Aachen University

Univ.-Prof. Dr.-Ing. **M. Wessling**
Aachener Verfahrenstechnik
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16. AACHENER TAGUNG



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Session 1

Keynote I

MTR's Experience Bringing Carbon Capture Membranes to Commercial-Scale

Timothy C. Merkel, Ph.D¹ (tim.merkel@mtrinc.com)

Abstract

There is widespread recognition that the world needs to decarbonize the power and industrial sectors to mitigate the adverse impacts of global climate change. A key aspect of any realistic carbon management strategy is to capture the CO₂ produced by fossil fuel combustion or industrial processes and utilize or store the CO₂ underground in a process known as carbon capture, utilization, and storage (CCUS). This CCUS approach is needed even after complete replacement of fossil fuels with renewable energy because industrial processes where CO₂ is produced regardless of energy source (such as steel and cement production) will continue to operate and generate CO₂ emissions for the foreseeable future. Carbon capture plants can reduce emissions from these persistent industrial sources, as well as from the large installed base of fossil fuel energy plants until they are replaced by renewable alternatives. Also, combined with bioenergy production, CCUS is the most economical way to achieve negative CO₂ emissions, which appears increasingly necessary as a backstop to meet net-zero emission targets.

Membranes as a CO₂ capture technology have many attractive features, including simple passive operation, environmental friendliness (no chemical handling, storage, or emissions), compact size, low water requirements, and the use of only electricity. Over the past 15 years, MTR has worked with the U.S. Department of Energy (DOE) to advance membrane capture technology from concept to the cusp of commercialization. This work has included membrane, module, and process improvements validated in field tests of progressively larger size at sites including the U.S. National Carbon Capture Center (NCCC) and Technology Centre Mongstad (TCM) in Norway. Over this time, the capacity of these field systems has been scaled up by more than 3 orders of magnitude. Today, the world's largest membrane capture system is installed at the Wyoming Integrated Test Center (WITC) in Gillette, WY. This 150 tonnes CO₂/day capture system is now in commissioning with a planned 12 months of operations starting before the end of this year. Concurrently, ongoing front-end engineering and design (FEED) studies are evaluating costs for commercial systems ranging in size from the WITC unit to approximately 10 times larger capacity.

¹Membrane Technology and Research, Inc. 39630 Eureka Drive, Newark CA 94560-4805

This presentation will review the development of MTR's membrane capture technology, describe where capture membranes are best suited for use, and discuss remaining commercialization challenges and opportunities for improvements.



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Session 2

Energy Applications

Systematic material design and development for polymer electrolyte fuel cells and anion exchange membrane water electrolysis

Takeo Yamaguchi¹ (yamag@res.titech.ac.jp)

Abstract

For large-scale use of renewable energy, a hydrogen society is necessary to overcome the supply and demand mismatch in time and space. Renewable energy should be converted into hydrogen or hydrogen carriers by water electrolysis, stored and transported, and used as electricity by fuel cells at the required time and place. Polymer-electrolyte fuel cells (PEFC) and anion exchange membrane water electrolysis (AEMWE) represent superior systems that exhibit high efficiency, offer better power generation, and meet the desired levels of demand for renewable energies through hydrogen. However, to facilitate the widespread use of water electrolysis and fuel cells, efficiency, cost, and lifetime problems must be resolved.

We are systematically designing and developing new materials from the molecular level to the device level. In both fuel cells and water electrolysis, different components such as membranes, catalysts, and catalyst layers share significant functions and work in a well-coordinated manner; hence, the total cell system must be optimized for the best performance. The systematic design and development approaches concerning materials for PEFC and AEMWE will be proposed.

Pore-filling thin electrolyte membranes enable us to achieve high performances [1,2] for polymer electrolyte fuel cells. Carbon-free nano Pt alloy connected catalysts showed high ORR activity and durability [3,4,5]. Water electrolysis using anion exchange membranes can efficiently produce hydrogen without using precious metals, and the development of highly durable anion exchange membranes is key to achieving the technology [6,7,8,9]. Non-precious metal electrocatalysts can be designed using materials informatics methodology [10,11,12,13]. These new membrane, catalyst, and cell design strategies will be explained.

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¹Institute of Integrated Research, Laboratory for Chemistry and Life Science, Institute of Science Tokyo (former Tokyo Institute of Technology)

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Polymer-composite membranes as porous thin-film electrodes for electrochemical applications

Anna Volz¹ (Lukas.Fischer@uni-due.de), Ignacio Sanjuán², Corina Andronesco², Mathias Ulbricht¹, Lukas Fischer¹

Abstract

In light of the transition toward renewable energy and the electrification of production processes, various electrolyzer technologies have emerged, specifically for water, CO₂, and N₂ electrolysis. However, these technologies still face significant challenges, particularly in the design of porous catalyst layers (CLs) and gas diffusion electrodes (GDEs). In each of these systems, gas-liquid transport processes determine the supply of substrate to electrocatalytically active sites and the release of formed products. Consequently, the pore properties of CLs and GDEs are crucial factors in governing this mass transport. However, current fabrication methods offer limited possibilities for adjusting the pore structure of CLs and GDEs.

Therefore, we developed a fabrication approach for intrinsically conductive polymer-composite membranes containing carbon filler, enabling a pore structure variation through film casting cum phase separation protocols [1]. These porous composite membranes are a new class of free-standing thin-film electrodes with potential use in a variety of electrochemical applications.

For their use as functional GDEs in CO₂ electrolysis, we further established a coating procedure to present Cu active sites within defined regions of the conductive pore system. When used as free-standing cathodes in a CO₂ flow electrolyzer, these membrane electrodes achieved Faradaic efficiencies greater than 70% for CO₂ electroreduction products at current densities up to 200 mA/cm². Our data suggests that the presentation of copper in an electrolyte-wetted, electrically conductive

¹Lehrstuhl für Technische Chemie II, Universität Duisburg-Essen, Germany

²Technische Chemie III, Universität Duisburg-Essen, Duisburg, Germany

pore structure can promote C–C coupling, enhancing C_2H_2/CO selectivity by up to threefold. Furthermore, we observed that a large pore size is essential to facilitate CO_2 gas transport to Cu sites after electrolyte infiltration into the pore. Additionally, our findings indicate that an anisotropic structure inhibits liquid distribution within the pore system, reducing the number of CO_2 transport pathways blocked by excessive electrolyte wetting. Similarly, coating a dense Cu film on the outer surface of an anisotropic membrane limited electrolyte uptake into the pore system of the underlying gas-distributing membrane, which further prevented CO_2 transport pathway blockage due to electrowetting at higher current densities.

To prepare free-standing CLs for water electrolysis, nickel nanoparticles can be directly blended with a carbon filler-containing polymer casting solution. This yields porous, conductive membrane electrodes integrated with an electrocatalyst and exhibiting either anisotropic or isotropic pore morphology. We showed that these can be employed as cathodic CLs in an anion exchange membrane (AEM) water electrolyzer without negatively impacting the overall cell resistance compared to a spray-coated Pt/C CL. Additionally, we found that the pore morphology of the membrane electrodes significantly affects H_2 crossover from the cathode to the anode, highlighting the role of gas-liquid transport. We attributed this phenomenon to the capillary pressure at the gas-liquid interface within the pore system, where factors such as pore size gradient, hydrophobicity, and surface roughness collectively influence H_2 bubble transport out of the CL. When this transport is inhibited, bubbles accumulate, increasing contact with the AEM, which in turn enhances H_2 solution into the AEM and ultimately promotes diffusion to the anode side.

In conclusion, our results underscore the potential of membrane fabrication protocols for producing porous thin-film electrodes with tunable pore structures, as well as the substantial impact that the pore structure in CLs and GDEs can have on performance in water and CO_2 electrolysis.

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Prediction of Conductivity vs. Selectivity Trends for Membranes Using Non-Equilibrium *Ab Initio* Molecular Dynamics

Pang Xiaoxian¹, Benedict Schier¹, Abhishek Khetan¹
(askhetan@modes.rwth-aachen.de)

KEYWORDS: Ion transport, molecular dynamics, ion selectivity, conductivity, polymeric membranes, CCC-NEMD

Abstract

Membranes that separate the electrolytes while allowing ion transport are central to the performance of low-cost flow batteries. However, the design of membranes with optimal ion selectivity, conductivity, and stability remains a challenge. Particularly, the trade-off between conductivity and ion selectivity is a bottleneck. Polymeric membranes are especially difficult to model in MD simulations because of the heterogeneous structure and long simulation time scales. To observe bulk conductivity in practical simulations, an often-employed approach is to perform simulations at elevated temperatures and extrapolate to room temperature using the Arrhenius relation. The higher kinetic energies of the atoms at higher temperatures make these jumps more likely at higher temperatures. However, this method cannot be applied when phase changes occur when increasing the temperature or the structure gets abnormally distorted, which makes the application of an extrapolation nonphysical. We utilize Chemical-Color-Coded Non-equilibrium molecular dynamics (CCC-NEMD) simulations to elucidate the mechanisms of ion transport through various polymeric membrane materials. The CCC-NEMD method applies a field that encourages the occurrence of these jump events. By using fields with different strengths, it is possible to identify a region with linear behavior between the field strengths and the corresponding values for the observed ionic conductivity. This region can then be used to apply an extrapolation towards a value for a field strength of zero.

Further, we investigate the effects of membrane structure, charge density, and functional groups on ion selectivity and permeability. Our work paves the way for relatively fast prediction of the conductivity vs selectivity trends across any class of membrane materials and for any type of ion. Using these methods, it would be possible to perform throughput virtual screening for a diverse range of membranes at the *ab initio* level

¹MODES – Multiscale Modelling of Heterogeneous Catalysis in Energy Systems, RWTH Aachen, Schinkelstraße 8, 52062 Aachen, Germany

Energy applications

and to identify promising candidates for experimental validation based on their predicted conductivity and selectivity.

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Session 3

Water treatment I

New rules and challenges in the water sector – what’s in for membranes?

Thomas Wintgens¹ (wintgens@isa.rwth-aachen.de)

KEYWORDS: Wastewater treatment, water reuse, membrane processes

Abstract

The water sector is facing huge challenges due to different pressures such as climate change leading to more frequent extreme weather events such as floods and droughts, degradation of ecosystems which provide essential services in the water cycle, as well as pollution with an enormous spectrum of pollutants, many being persistent in the environment [1].

At the same time, environmental regulations are becoming more stringent and require additional treatment and monitoring efforts both in water supply and wastewater treatment. Among the new regulatory frameworks within the European Union is, for example, the Drinking Water Directive (2020/2184), which had to be transposed into national law by 2023 and includes, e.g., limit values for perfluorinated substances (e.g., PFAS below 0.5 micrograms per liter), which will pose a challenge to many utilities having to achieve this target by 2026 [2]. Among the options to remove this class of mobile and persistent pollutants are two specific activated carbon types, ion exchange resins, and dense membrane processes such as nanofiltration and reverse osmosis [3].

With respect to wastewater treatment, the re-cast of the Urban Wastewater Treatment Directive (UWWTD) is expected to bring about several new requirements and indicative targets [4]. The need to remove micropollutants in larger plants (e.g., above 150,000 population equivalents) in connection with stricter targets for nitrogen removal calls for widespread upgrades and extensions of municipal plants, which are nowadays largely equipped with conventional activated sludge systems. While ozonation and activated carbon (AC) adsorption or combinations of both are already applied for micropollutant removal in a number of full-scale references, the implementation of membrane bioreactor technology with simultaneous powdered AC dosing is a promising hybrid process with a number of benefits [5].

Membrane processes become particularly attractive in municipal wastewater treatment when water reuse is envisaged with beneficial use of reclaimed water in agricultural or urban greenspace irrigation, industry

¹Institut für Siedlungswasserwirtschaft / Institute of Environmental Engineering, RWTH Aachen University, Germany

(e.g., cooling or process water), or managed aquifer recharge. The European Regulation (2020/741) for the first time established minimum requirements for water reuse in agricultural irrigation and defines water quality classes for different applications. The strictest water quality class A for unrestricted irrigation requires an *E. Coli* count below 10 per 100 mL and a process performance validation procedure including, e.g., a 6 log₁₀ reduction target for Coliphages as indicators of viral pathogens [6]. This ambitious target can typically only be achieved by a combination of mechanical-biological (secondary) treatment plus filtration (tertiary treatment) and disinfection (e.g., UV-based). The filtration step can be carried out by membranes (e.g., micro- or ultrafiltration, also in membrane bioreactors) but, also due to the multi-barrier concept, porous membranes do not replace a final disinfection step. Dense membrane processes play a role in very high-quality applications such as indirect potable reuse, but only a few cases exist so far in Europe [7]. Membranes are also widely used in industrial water systems and have possible applications in resource recovery from waste streams (e.g., phosphorus from sewage sludge ash) [8]. A key sustainability aspect is related to the management of brines and the options to recover valuable compounds from them [9]. While there is a range of prospects for membrane applications in the water sector, challenges remain mainly related to investment and operation cost, operational ease, as well as energy demand and concentrate disposal or utilization.

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Polymer Membrane Modification Using Electron Beam Irradiation for the Removal of Endocrine Disruptors from Water

Zahra Niavarani¹ (zahra.niavarani@iom-leipzig.de), Daniel Breite, Agnes Schulze

KEYWORDS: Microfiltration, Water treatment, Electron beam, Endocrine disrupting chemicals

Introduction

The increasing detection of endocrine-disrupting chemicals (EDCs) in water sources has raised significant environmental concerns. These chemicals can interfere with hormonal systems at low concentrations, posing risks to wildlife and human health [1]. Conventional water treatment processes often fail to remove these persistent pollutants, necessitating more effective and economically feasible technologies.

¹Leibniz Institute of Surface Engineering (IOM), Germany

Membrane technology has shown great promise in removing EDCs from water. In this study, electron beam irradiation is used to modify microfiltration membranes, significantly enhancing their adsorption capacity and selectivity for EDCs.

Materials & Methods

To enhance the adsorption efficiency of polyethersulfone (PES) membranes, urethane functional groups were introduced through surface modification. The process involved soaking the PES membrane in a polyvinyl alcohol solution followed by electron beam irradiation to create reactive sites. Subsequently, the membrane was treated with hexamethylene diisocyanates in n-hexane to form urethane groups. Another approach utilized precipitation polymerization to synthesize adsorber and selective polymer particles, which were integrated into the membrane using nonsolvent-induced phase inversion and secured by electron beam irradiation. The modified membranes underwent characterization using SEM, FTIR, and contact angle measurements to confirm successful modification. Their adsorption efficiency was tested through dynamic adsorption experiments simulating real-world water treatment conditions.

Results & Discussion

Adsorber microfiltration membranes were developed to efficiently remove EDCs from water. Modifying the surface of PES microfiltration membranes with urethane functional groups significantly enhanced their adsorption capacity towards specific EDCs, likely due to the formation of hydrogen bonds [2][3]. Additionally, incorporating adsorber polymer particles within the membrane structure led to a substantial increase in adsorption capacity [4]. The introduction of molecularly imprinted polymer (MIP) particles into the membrane matrices imparted selectivity, enabling the membranes to selectively adsorb target molecules [5]. The modified membranes demonstrated superior adsorption capacities compared to conventional nanofiltration membranes and offered the advantages of high water permeability and operation without the need for high pressure. Figure 1 demonstrates the dynamic removal mechanisms of the modified membranes. Furthermore, these membranes maintained their adsorption capacity across multiple adsorption cycles after undergoing a regeneration procedure, highlighting their potential for sustainable and efficient water treatment applications.

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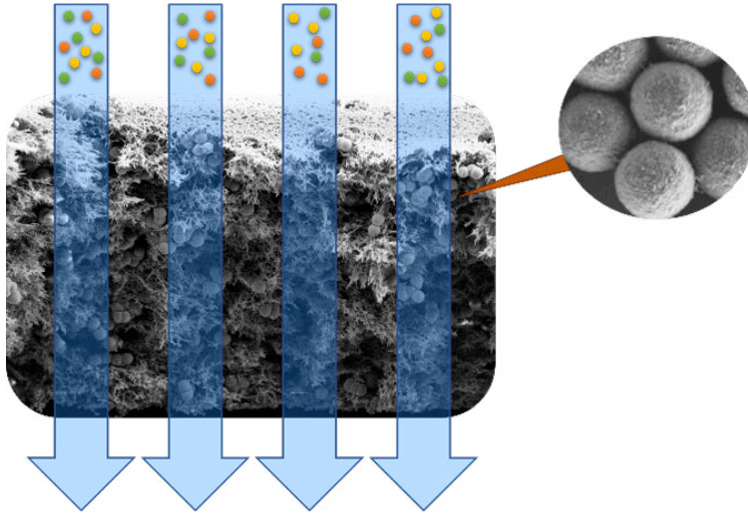


Figure 1: PES membrane integrated with polymer adsorber particles for the dynamic adsorption of EDCs from water.

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Introducing Powdered Activated Carbon Counter Flow to an Inline-Dosing Membrane Hybrid Process – Impacts on Membrane Performance

M. Zimmermann¹ (zimmermann@isa.rwth-aachen.de), M. Hoffmann², C. Staaks², B. Aumeier³, T. Wintgens¹

KEYWORDS: Membrane Hybrid Process, Ultrafiltration, Adsorption, Advanced Wastewater Treatment

Abstract

Drastic changes in climatic conditions and the hereby increased pressure on existing water resources require the exploitation of alternative water resources. The reuse of municipal wastewater in agricultural irrigation can provide a stable water supply in water-scarce areas. This non-potable reuse requires advanced treatment of wastewater treatment plant effluents, including disinfection and removal of organic micropollutants [1]. Ultrafiltration membrane hybrid processes allow for near-complete disinfection, as well as organic micropollutant removal when utilizing powdered activated carbon (PAC) as an adsorbent [2].

Such a hybrid process has been operated in pilot scale in this study for a duration of over two years, utilizing real wastewater as influent. After conventional biological treatment, PAC was dosed prior to a DuPont Water Solutions/ inge GmbH Multibore[®] hollow fiber membrane. The treatment capacity of the process was 500 L/h respectively for two treatment trains. One mimicking a conventional Ulm Process including a contact reactor for activated carbon accumulation, the other representing a novel inline-dosing concept. In this inline-dosing approach, the required contact volume is reduced to less than one minute, which is possible by utilizing fine PAC products with increased adsorption kinetics. Ultimately, a PAC counter-flow scheme was introduced to the process, which allowed for a vast reduction in required PAC dosage by up to 50% compared to the inline process without recirculation.

Within this study, we investigated the impact of variations in the inline adsorption parameters (contact time, PAC dose, PAC size) and of the PAC counter flow on the performance of the ultrafiltration membrane.

¹Institute of Environmental Engineering (ISA), RWTH Aachen University, Mies-van-der-Rohe-Str. 1, 52074 Aachen, Germany

²Dupont Water Solutions / inge GmbH, Germany

³Urban Water Systems Engineering, TU München, Germany

Depending on the prevailing setting, permeabilities (normalized to 20°C) of around 300 L/(m² h bar) could be reached despite a challenging water matrix. The vast reduction of required carbon dosage (compared to other studies without a recirculation [3][4]) by the counter-flow scheme does not seem to influence the performance of the membrane plant meaningfully.

It is expected that the combined treatment that this hybrid process offers will provide meaningful cost synergy and improved ecological evaluation, offering a long-term resilient treatment option, with a life cycle assessment pending.

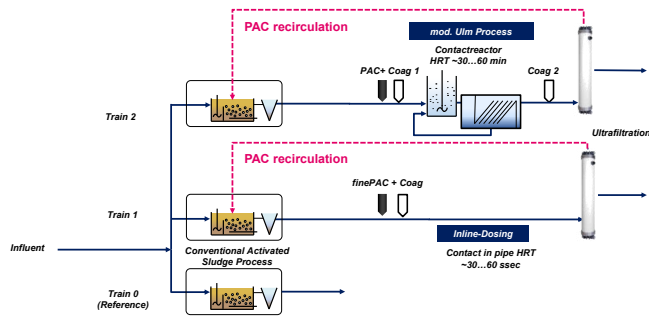


Figure 1: Process flow chart of pilot system including three parallel treatment trains for comparative investigation of conventional activated sludge process, Ulm Process, and PAC inline-dosing process. Treatment trains shown were operated in pilot scale, including the biological stages.

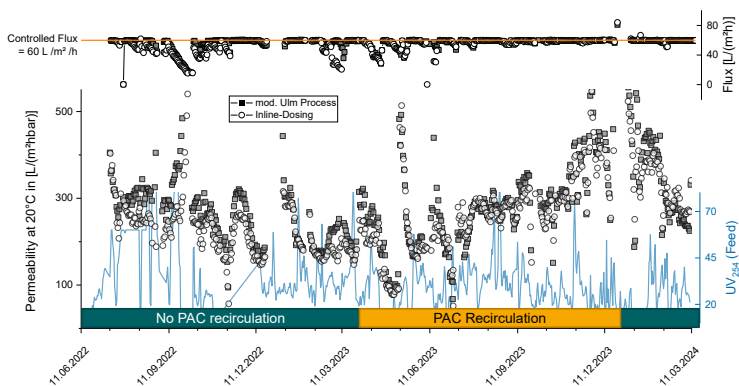


Figure 2: Operational parameters and performance indicators of the pilot system during the duration of operation.

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Session 4

Water treatment II

Polyelectrolyte multilayer based Nanofiltration Membranes, Made to Order

Wiebe M. de Vos¹ (w.m.devos@utwente.nl)

Abstract

The intensification of the use and reuse of fresh water resources comes at a cost. Reports on rising levels of emerging contaminants in our water are numerous. New and better membranes are urgently needed to achieve more sustainable water treatment processes. Over the past decade, polyelectrolyte multilayers have established themselves as one of the most promising new materials in membrane science and technology [1]. Novel nanofiltration membranes are prepared by the self-assembly of oppositely charged polyelectrolytes at the interfaces of a porous ultrafiltration support membrane. In this so-called Layer-by-Layer (LbL) assembly, the support membrane is alternately exposed to polycations and polyanions to build polyelectrolyte multilayers (PEMs) of controllable thickness. After coating, the separation properties of the membrane are completely determined by the applied PEM layer.

A real strength of this approach has been found to be its high versatility [2]. The material properties of the polyelectrolyte multilayer, and thus its separation properties, can be tuned by many parameters, including the choice of polyelectrolytes, the ionic strength, the pH, and the coating method. Moreover, by coating membranes in an asymmetric fashion, building one more dense PEM on top of a more open PEM, we can even combine the separation properties of the two layers in a single system [3].

With this high degree of versatility, we can ask the question: how far away are we from producing membranes on demand? Creating membranes with a very specific combination of size exclusion (MWCO), Donnan exclusion, and dielectric exclusion that fit exactly with the desired application. Moreover, is it then possible to add a final coating that limits the degree of fouling in challenging separations while leaving the separation properties unaffected?

In this talk, I will discuss a modular way of preparing PEM-based membranes that could enable the production of PEM-based nanofiltration membranes, made to order.

¹Membrane Surface Science, University of Twente, The Netherlands

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Composite Hollow Fiber Nanofiltration Membranes via Chemistry in a Spinneret

Hannah Roth¹ (h.roth@utwente.nl)

KEYWORDS: Nanofiltration, Composite hollow fiber membranes, Cross-linking, Phase inversion, Polyelectrolytes, Chemistry in a spinneret

Abstract

The fabrication of composite membrane structures typically requires several procedurally complex membrane modification steps. In recent years, we have demonstrated that the scalable technology basis of "Chemistry in a Spinneret" serves as a one-step fabrication process for composite hollow membranes, providing an intelligent alternative for the multi-step membrane modification processes. The formation of a porous hollow fiber membrane acts as an underlying process superimposed with chemical reactions and/or ionic interactions of additives occurring at the interface of the polymer solution and lumen fluid.

In this paper, we present three different material systems that have been successfully converted into composite hollow fiber nanofiltration membranes using the "Chemistry in a Spinneret" technology.

¹University of Twente, The Netherlands

The formation of composite hollow fiber membranes with crosslinked separation layers is enabled by applying the crosslinking reactions of polyethylenimine with trimesoyl chloride and glutaraldehyde during hollow fiber spinning. Both created layers exhibit similar nanofiltration properties and were investigated for solvent pH and backwash stability. The combination of ionic crosslinking of oppositely charged polyelectrolytes and covalent crosslinking of the polyelectrolytes with glutaraldehyde during hollow fiber spinning results in nanofiltration membranes. The fibers show high salt retentions and a low molecular weight cut-off of 280 Da.

In the last material system, sulfonated polyethersulfone (SPES) serves as a polyanionic additive in the polymer solution that ionically complexes with polycations in the lumen fluid during fiber spinning. The spun fibers have a positively charged lumen surface, which enables subsequent coating with the polyanion polystyrene sulfonate (PSS) in a coating bath. The resulting composite hollow fiber membranes have open to dense nanofiltration characteristics depending on the used polyelectrolytes. The various material systems investigated establish the "Chemistry in a Spinneret" as a universal technology base for the fabrication of nanofiltration hollow fiber membranes. The results provide a broad basis for further membrane material and morphology developments.

Early Scaling Detection in Conventional Plug Flow and Upcoming Closed-Circuit Reverse Osmosis for Antiscalant-Free Operation

Martin Futterlieb¹ (martin.futterlieb@uni-due.de), Stefan Panglisch

KEYWORDS: Closed-circuit Reverse Osmosis, Membrane scaling

Abstract

Membrane scaling, i.e., the precipitation of sparingly soluble salts, is typically the limiting factor for the recovery of inland membrane desalination plants, e.g., for brackish fresh or wastewater treatment. The state-of-the-art method to mitigate scaling is the dosage of antiscalants (ASs). However, the application of ASs has various drawbacks. For one, the discharge of AS-bearing concentrates into receiving water bodies is disputed and connected to environmental concerns [1]. Moreover, ASs may promote biofouling [2]. Additionally, traces of ASs can be detected

¹University of Duisburg-Essen, Germany

in the permeate [3], which may form harmful disinfection byproducts [4]. Early scaling detection, i.e., before measurable permeability loss, could allow adjusting the operation in time to counter incipient scaling, e.g., by lowering the recovery or performing a forward flush. This could presumably reduce or even avoid the addition of ASs.

This study investigated various options for early CaCO_3 -scaling detection in conventional and continuous plug flow reverse osmosis (PFRO) and upcoming discontinuous operation as closed-circuit RO (CCRO) on a pilot and bench scale. The pilot plant was equipped with two series of four-inch spiral wound RO elements ($\sim 16 \text{ m}^2$). The bench-scale plant has one 2.5-inch RO element ($\sim 2.6 \text{ m}^2$). Both plants can be operated as conventional PFRO with concentrate recirculation or as CCRO and were fed with a real water matrix inducing CaCO_3 -scaling ($\text{SI}_{\text{Feed}} \sim 0.4$).

The results of the PFRO operation showed that CaCO_3 -scaling can be seen early in dropping concentrate pH, typically multiple hours before the permeability starts to fall. Other parameters manifesting membrane scaling, e.g., lowered retention and increasing pressure loss, reacted significantly later than the pH drop. CCRO operation confirmed early detection by the concentrate pH. Moreover, the cyclic nature of CCRO facilitates incipient CaCO_3 -scaling detection. A steady increase in concentrate pH showed no scaling tendency within a closed-circuit mode (i.e., no concentrate discharge). In contrast, flattening the pH and even dropping the pH in the closed-circuit mode indicated incipient CaCO_3 -scaling way before dropping permeability. Using the concentrate pH as an early CaCO_3 -scaling detection tool allowed both PFRO and CCRO to operate on the edge of CaCO_3 -scaling without AS dosage. A CCRO experiment using the concentrate pH as a control parameter was conducted for over 50 days. Therefore, the recovery was lowered (i.e., reduced closed-circuit time) whenever a pH drop was detected, which allowed to counter membrane scaling and prevent heavy permeability drops.

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Session 5

Process Engineering

Hollow Fiber Nanofiltration Membranes - From colour removal to efficient total solutions

Henk Koops¹ (h.koops@nxfiltration.com)

KEYWORDS: hollow fiber nanofiltration, total solution, wastewater, surface water, micropollutants, PFAS, colour removal

Abstract

NX Filtration started in 2016 with industrial scale production of hollow fibre nanofiltration (HFNF) membranes, which were developed at the University of Twente. Now, 8 years later, the company is listed at the Euronext Amsterdam Stock market, moved into a brand new large scale production facility, has 3 product portfolio's, and is growing fast, both in the number of projects sold, and in size of the projects. This presentation shows how NX Filtration developed as a company in those 8 years, starting with HFNF removing colour from rivers in Indonesia up to total solutions for producing high quality water from secondary municipal wastewater and surface water with HFNF at the core: the two sweet spot applications for HFNF. The hollow fibres are kept clean easily (forward flush and backwash) as it has a smooth flow path (spacer free) and are chlorine tolerant for disinfection. This makes NX Filtration's HFNF a key enabler for sustainable and affordable water treatment.

Initially, with its low fouling properties, NX Filtration focussed on HFNF for direct surface water treatment, and booked its first large scale success in Dumai, Indonesia (4 MLD) on river water treatment in 2020. A simple strainer (130 micron) as pre-treatment followed by HFNF, pH adjustment and post chlorination resulted in perfectly clear, clean and safe drinking water. The main challenge here was removing colour (humic acids). Several similar large scale applications followed since; Fossmark, Sweden (0.9 MLD), multiple containerized solutions in Canada, and three more in Indonesia: Meranti (1 MLD), Bengkalis (4.8 MLD) and Medang Kampai (10 MLD). And at the same time many more projects are in the pipeline, varying from pilot phase, up to project development, in South East Asia, Scandinavia, and North America.

NX Filtration quickly learned that their HFNF is a key enabler in municipal wastewater treatment and drinking water production from surface water. The application of HFNF often creates an efficient total solution when the membrane is more open and combined with other treatment technologies. This might seem counterintuitive as one wants to remove as much organic molecules as possible. True, but the HFNF does not

¹NX Filtration, The Netherlands

have to do this all by itself. In the treatment of municipal wastewater this becomes evident.

The combination of HFNF and AOP (UV/H₂O₂, NanoX) creates a perfect synergy for the process of converting wastewater into high quality re-use water. The open HFNF dNF80 membrane removes the colour, and up to 50–90% of the micropollutants, while passing monovalent ions and some bivalent ions. On the one side, this results in a concentrate composition that can be returned in front of the biology leaving no waste stream to be further treated. A tighter HFNF membrane would create a concentrate with too high TDS levels that would accumulate in the biology process and ultimately causing harm. On the other side, the micropollutants still passing the dNF80 will be destroyed by the UV/H₂O₂ process, where the dNF80 creates a permeate of extremely high quality with UVT > 95%. This results in a nearly complete removal of all micropollutants with relatively low energy consumption and no waste.

To remove the smallest PFAS molecules, the addition of a GAC filter after HFNF is a reliable and good solution as UV/AOP does not destroy these types of molecules. Since the water after UV/AOP is super clean and the GAC filter only needs to remove the small PFAS molecules, the lifetime of this GAC filter will increase significantly, making this step a relatively cheap one.

In short, a more open HFNF, that allows a relatively high passage of mono- and bi-valent ions, while retaining micropollutants including PFAS up to 50–90%, makes post-treatment processes like AOP, UV/H₂O₂, GAG/PAC very efficient and cost effective, and results in an easy-to-treat brine.

Our largest HFNF application to date is part of an Indirect Potable Reuse (IPR) project in Sapal, Mexico (17 MLD). Biologically treated wastewater effluent is screened by 20 μm before entering the HFNF. After ozone and GAC, the water is collected in a large reservoir, from where water is further treated to drinking water quality, a major step in the Mexican battle against severe drought and water scarcity.

This concept of combining HFNF + AOP + GAC can also be applied to the production of drinking water from surface water. Since the treatment train for surface water is less stringent for total removal of protozoa and viruses compared to upcycling wastewater, one could choose to leave out the UV/AOP, as the GAC filter will remove the remainder of the micropollutants, including PFAS. UV or post-chlorination can be added as a safety disinfection step, and the strainer could be replaced by a drum screen or even a chemical-free rapid sand filter. In the latter case, naturally present biomass will convert assimilable organic carbon, ammonia, and manganese. Additionally, more effective particle removal may be expected, and the combined effects allow for stable HFNF operation with higher flux and a reduced frequency of backwashing.

In comparison to spiral wound NF and RO, HFNF works very well with

limited pre-treatment, making the addition of coagulants and flocculants obsolete. The HFNF allows monovalent ions to pass. The limited bi-valent ion retention ensures that the process does not require anti-scalants, remineralization, and results in a relatively easy-to-discharge concentrate stream. Last but not least, the hollow fibres can be kept clean easily (forward flush and backwash) as it has a smooth flow path (spacer free), are chlorine tolerant (for disinfection) and back-washable. This makes NX Filtration's HFNF a key enabler for sustainable and affordable water treatment.

Microsieve bulk filtration (an update)

Franz Liebermann¹ (fl@microsieve.net)

Abstract

Microsieves are membranes made from silicon wafers using micro-engineered manufacturing processes known in the semiconductor industry for micro-filtration. Microsieves are almost ideal and inert membranes characterized by uniform pores from 0.1 to 10 microns. Bulk filtration based on microsieves is a difficult task. Challenges include very fast pore clogging and blocking, breaking membranes, up-scaling to industrial level volumes, in-line hydrophilization, membrane integrity testing, and more. MicroSieve Technology has solved these challenges by developing new microsieves adapted to its proprietary, patented, and scalable high frequency flow reversal (HFFR) technology. This technology is designed to keep the microsieve pores open and allow stabilizing the high flux specific to microsieves without breaking.

¹MicroSieve Technologies

Recent and Potential Use of Membrane Contactors for the Hydrogen Industry

Norbert Selzer¹ (nselzer@solventum.com), Martin Ulbricht¹

KEYWORDS: Hydrogen, membrane contactor, water treatment, degassing

Introduction

The increasing generation and use of hydrogen plays an important role as part of the necessary technology changes towards climate protection. This also requires an adaptation of auxiliaries, such as the treatment of water. Among several other membrane technologies, membrane contactors are already regularly used for the specific requirements of hydrogen generation. This presentation provides an overview of already established applications in the generation of grey, blue, green, etc. H₂, and mentions recent developments and potential applications on both the generation and use sides of the hydrogen industry.

Feed Water to Electrolysis

The dominant application is CO₂ removal from water. PEM electrolyzers typically require the highest efficiency of ion removal, making CO₂ removal essential upstream of polishing steps like Electrodeionisation (EDI) or mixed resin beds. Alkaline stacks are less sensitive to residual ions but require CO₂ removal up to very low levels to avoid precipitation of carbonates. Membrane degassing is the state-of-the-art technology for physical inline CO₂ removal from water. Recent projects also investigate N₂ removal from feed water to support higher H₂ gas purity.

Hydrogen Generation by Steam Reforming

The established use of membrane degassing involves O₂ removal as part of the water treatment for the steam system. Compared to traditional thermal degassing, a higher level of flexibility and efficiency may be achieved with lower installation efforts. The potential use of membrane contactors for capturing CO₂ to produce blue H₂ from grey H₂ is a recent research target.

Flexible H₂-Fueled Combined Cycle Thermal Power

Beyond hydrogen use in industrial processes or transportation, its opportunities for balancing/buffering renewable electric power are of major

¹3M Healthcare Germany GmbH, Germany

interest. Over the next decades, this will require a significant number of H₂-fueled combined cycle (gas+steam turbine) power stations. Membrane degassing may help increase the flexibility of such systems, which is one of the most important criteria for their grid balancing function.



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Session 6

Desalination I

Electromembrane technologies for sustainable chemical industry: technology ranking and industrial perspectives

Michele Tedesco¹ (michele.tedesco@tno.nl)

Abstract

Electromembrane technologies can be broadly defined as technology aiming to (selectively) recover or remove a target compound by means of ion exchange membranes under the effect of an applied electric field. Initially developed for desalination and water treatment applications, electromembrane technologies (such as, e.g., electrodialysis, bipolar electrodialysis, electrodialysis metathesis, shock ED) are receiving increasing attention across different sectors, with the possibility to unlock novel sustainable applications [1]. In fact, such technologies offer several advantages, i.e., modularity (ease of scale-up), flexibility (rapid ramp-up/down), mild operating conditions, enhanced mass transfer given by the electric field, easy reaction kinetics control, and metering of products.

In this contribution, we will present the results of our analysis on different electromembrane technologies (e.g., electrodialysis and related processes), including a qualitative ranking based on a list of technical and environmental key performance indicators, as well as a number of promising industrial applications (including, e.g., metal recovery, carbon capture [2], biobased downstream processing). Based on this initial ranking, we have identified the most promising electromembrane technology for each specific application and experimentally investigated bipolar electrodialysis for selective carboxylic acid recovery as a case study.

Finally, we will present our results of the techno-economic analysis, to assess the economic viability of each investigated concept. Notably, although energy efficiency is often mentioned as an advantage, our analysis shows that such a statement is strictly process-dependent and therefore cannot be generalized. CAPEX/OPEX should be estimated case by case and benchmarked against the best available thermally-driven alternative.

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Project RIKovery – Recycling of Industrial Saline Waters through Ion Separation Concentration and Intelligent Monitoring

Yuliya Schießer¹ (yuliya.schiesser@covestro.com), Christine Kl-effner, Gerd Braun², Herbert Bassek³, Matthias Woyciechowski⁴, Christoph Kirsch⁵, Christian Linnartz, Lukas Mankertz⁶

KEYWORDS: Ion Separation, Concentration, Intelligent Monitoring, Water Reuse, Salt Recovery

Abstract

Chemical production and mineral processing industries are closely related to the use of a significant amount of water. This produces large quantities of wastewater containing high concentrations of inert salts. Currently, more than 6 million tons of chloride are discharged into surface waters via wastewater in Germany every year. More than 3/4 of these come from the chemical (51%) and mineral processing (26%) industries.

The rising water scarcity and competing use of water resources increase the need to reduce the discharge of these high salt loads, especially into surface waters, and to reuse the water and its valuable components. Since the salt contamination is often present as a mixture of different salts and/or the concentration is too low for direct reuse, treatment processes are necessary to enable further application.

Of great importance are the separation of the ions or the setting of a defined composition, as well as more energy-efficient processes (and combinations thereof) for concentrating the salt solutions to a suitable concentration level. Especially in the area of high salt concentrations (higher than 10 wt.% NaCl), there is a lack of suitable technologies. The state of the art is still thermal evaporation (e.g., MVR mechanical

¹Covestro Deutschland AG, Leverkusen, Germany

²Technical University of Applied Sciences Cologne, Cologne, Germany

³BWS Water, Oberndorf a. N., Germany

⁴Evonik Operations GmbH, Hanau-Wolfgang, Germany

⁵K+S AG, Unterbreizbach, Germany

⁶RWTH Aachen, Aachen, Germany

vapor recompression), which is neither sustainable nor allows in most cases the reuse of produced concentrates. It is not expected that all fields of application can be covered with a single technology.

Within the framework of the German Ministry for Education and Research (BMBF) funding measure "Water Technologies: Reuse (WavE II)" in the RIKovery project, the potentials of innovative technologies (Osmotic Assisted Reverse Osmosis OARO, High Pressure Low-Salt-Rejection Reverse Osmosis HPLSRRO, High Pressure Nanofiltration HPNF, Forward Osmosis FO, Flow-Electrode Capacitive Deionization FCDI) are systematically investigated and the promising areas of application are developed.

In order to achieve widely transferable project results, the project consortium has identified industrially relevant applications that cover the main discharges (polymer chemistry, specialty chemicals, mineral industry) but differ significantly in terms of challenges (volume flows, concentration ranges, salt composition, purity requirements for the reuse of salts or water) and thus cover the majority of the industrial salt discharges.

The main goal of the project is to create an economically and technically well-founded basis for decision-making for the implementation of salt and water recovery processes on a production scale. Based on real relevant examples from practice with modules on a technical scale, it was shown that innovative technologies significantly expand the possibilities of concentrate processing:

- In particular, the operation of nanofiltration beyond the usual operating pressure of 41 bar proves to be particularly attractive. Here it was shown that the separation of sulfates and chlorides is feasible for almost saturated salt solutions.
- The concentration of monovalent neutral salts by means of HPLSRRO succeeds at an application pressure of 120 bar up to 15 wt.%, and thus well beyond the limits of high-pressure reverse osmosis.
- It was shown how clever process concepts can be used to achieve concentration up to the crystallization of monovalent and divalent ions with low energy demand by means of FO.
- FCDI could be used to separate and concentrate chloride ions from sulfate ions in a single step. However, the process is currently at a significantly lower TRL level compared to the pressure-driven membrane processes.

Partial Desalination with the Membrane Capacitive Deionisation for Monovalent Ions

Hanna Rosentreter¹ (hanna.rosentreter@tu-dresden.de), David Schödel, André Lerch

KEYWORDS: Chloride, Electrochemical Desalination, Groundwater, Monovalent Selective Ion Exchange Membranes, Nitrate, Sodium

Abstract

Groundwater with high concentrations of sodium, chloride, or nitrate, caused by natural or anthropogenic processes, needs desalination before domestic use or for irrigation. Slightly saline groundwater resources are coming into focus due to shortages of freshwater resources. However, conventional desalination by ion exchange, nanofiltration, or brackish water reverse osmosis is energy-intensive, requires remineralisation, and specific concentrate treatment [1].

Within the BMBF-funded project innovatION (02WV1572), self-made membranes by FUMATECH BWT GmbH and the Leibniz Institute of Polymer Research (IPF) in Dresden are placed into Capacitive Deionisation to enable selective desalination of monovalent ions. The methods for membrane preparation were already presented at the last AMK 2022. In particular, the additional neutral- and polyamide layers on standard ion exchange membranes or nanofiltration membranes have shown in lab-scale experiments an up to >10-fold higher removal of monovalent ions due to their preferential transport through the membranes and increased adsorption on the electrodes. The energy efficiency varies between 0.1-1 kWh/m³ depending on the water recovery and feed concentration.

Long-term experiments and tracer tests have shown that the geometry and accuracy of electrode production impact the monovalent selectivity and irreversible scaling on membranes and electrodes caused by calcium carbonate or calcium hydroxide. In our presentation, we will present the results of the lab-scale and pilot-scale experiments on Langeoog and in Nienburg.

¹TUD Dresden University of Technology, Germany

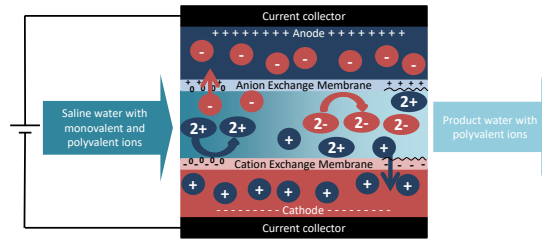


Figure 1: Monovalent Ion Selective Membrane Capacitive Deionisation during Adsorption

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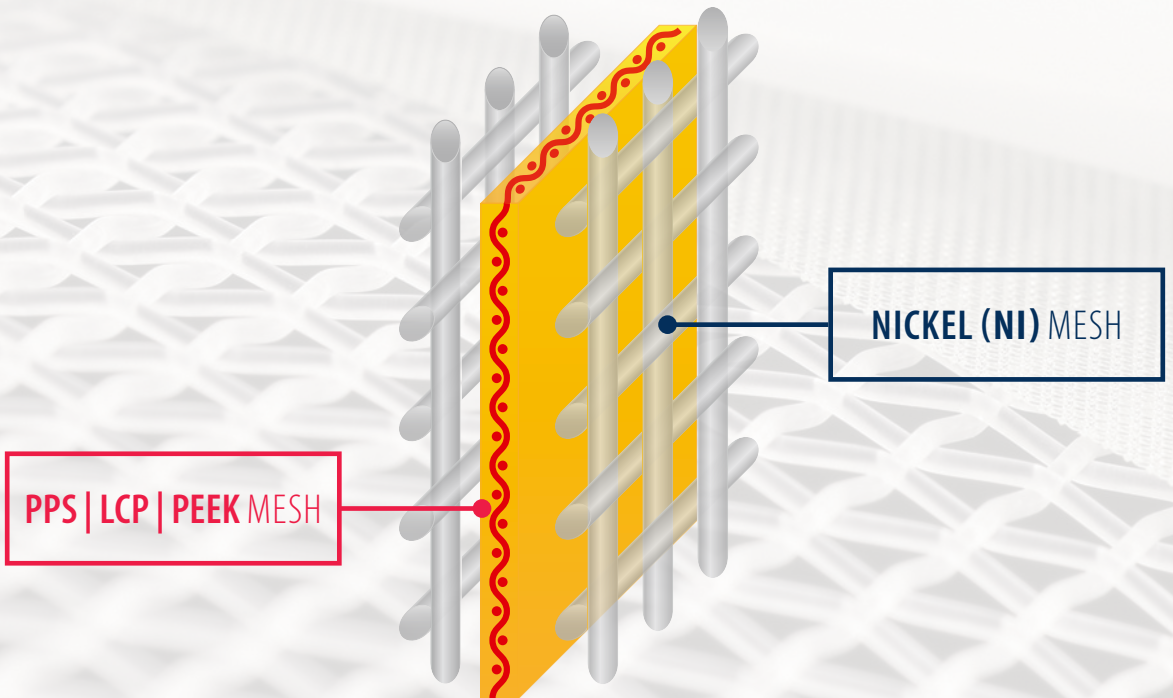
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Session 7

Health

Blood purification beyond Low- and High Flux membranes for ESRD patients

Bernd Krause¹ (bernd_krause@baxter.com) , Markus Storr

KEYWORDS: Blood Purification, Medical Devices, Membranes

Abstract

The majority of the approximately 5 million patients worldwide with end-stage renal disease (ESRD) currently undergo treatment via extracorporeal blood purification using low- or high-flux membranes. However, are there innovative approaches to enhance morbidity and mortality outcomes for patients reliant on blood purification? This presentation explores three distinct concepts where advancements in membrane technology hold the potential to significantly improve patient outcomes.

(i) As dialysis membrane technologies have advanced, the ability to clear increasing numbers of uremic toxins has occurred. To date, however, the class of uremic toxins known as large middle-molecules has been classified as “difficult to remove.” Expanded hemodialysis (HDx) utilizes a new generation of MCO membranes; these membranes provide the ability to remove large middle-molecules effectively for the first time, without significant albumin loss. Clinical studies indicate that HDx can improve clinical outcomes such as patient mortality and morbidity versus conventional high-flux hemodialysis. HDx has been shown to improve patient-reported quality of life aspects of kidney disease, including symptom burden, restless legs syndrome criteria, pruritus, and dialysis recovery time. HDx therapy has the potential to reduce the total cost of care, primarily driven by potential reduction of cardiovascular events, infections, medication usage, all-cause hospitalizations, hospitalization rate and length of stay.

(ii) Liver failure (LF) is associated with significant morbidity and mortality and can occur in the presence or absence of underlying chronic liver disease. Acute-on-chronic liver failure (ACLF) generally presents after an acute decompensation in a patient with preexisting liver disease or cirrhosis. In contrast, acute liver failure (ALF) arises in the absence of underlying chronic liver disease. The underlying mechanisms involved in LF are multifactorial and are associated with the accumulation of hepatotoxins, including proinflammatory cytokines, and endotoxins released from intestinal flora and with alterations in the functional capacity of albumin. Impaired hepatic clearance of these toxins leads to a cascade

¹Gambro Dialysatoren GmbH, Holger-Crafoord-Straße 26, 72379 Hechingen, Deutschland

of biochemical processes, including oxidative stress, increased capillary permeability, immune dysregulation, and cellular damage. Novel extracorporeal liver support devices aim to remove circulating toxins and exchange dysfunctional albumin with the intention of stabilizing liver function while allowing for recovery of native hepatic function or bridging to liver transplantation.

(iii) The therapeutic target of extracorporeal carbon dioxide removal (ECCO₂R) is the elimination of carbon dioxide (CO₂) from the blood across a gas exchange membrane. Clinical data suggest that mechanical ventilation (MV) can contribute to the negative outcomes in patients with acute respiratory distress syndrome (ARDS). ECCO₂R has the potential to facilitate ultra-protective lung ventilation (UPLV) strategies in these patients and to decrease injury from mechanical ventilation while limiting hypercapnia (abnormally elevated carbon dioxide levels in the blood) and respiratory acidosis.

Polymer Membrane Surface Functionalization with Proteins and Enzymes by Electron Beam Irradiation

M. Schmidt¹ (martin.schmidt@iom-leipzig.de), A. Prager, A. Abdul Latif, S. Zahn, A. Schulze

KEYWORDS: Polymer surface, Grafting, Biofunctionalization, Enzyme membrane reactor, Anti-fouling, Electron beam

Introduction

Surface functionalization of porous polymer membranes plays a decisive role in tailoring material properties. Conventional approaches to immobilize substances on polymer membranes are usually based on chemical methods which can be time-consuming and costly due to long reaction times and expensive reagents. A novel electron beam-based method has been developed to couple biomolecules such as proteins or enzymes directly to polymer membranes in a fast one-step process requiring only an aqueous solution of the biomolecules.

Materials & Methods

Polyvinylidene fluoride (PVDF) microfiltration membranes were modified by electron beam-induced grafting with biomolecules such as al-

¹Leibniz-Institut für Oberflächenmodifizierung e.V. (IOM)

bumin protein or industrial lipase enzyme. By employing a design of experiments approach, models were developed to predict the protein grafting yield or enzymatic activity respectively. The effectiveness of a lipase-modified membrane was demonstrated by the enzymatic removal of olive oil fouling in successive filtration and self-cleaning cycles. To understand the mechanism of this radiation-based functionalization, static quantum chemical calculations were performed to investigate the coupling of two small model molecules, glycine and taurine.

Results & Discussion

PVDF membranes were effectively modified with albumin protein, resulting in an optimized coverage of approximately 1000 mg/m² (using 11.5 g/L albumin, 2.7 min incubation time, 117 kGy electron beam dose) [1]. A model to predict the grafting yield was confirmed and used to reduce process costs significantly compared to literature. Subsequently, this technique was applied to immobilize an industrial lipase on PVDF to create lipolytic membranes used in fouling tests with olive oil [2]. By placing the strongly fouled membranes in an aqueous buffer at 37 °C, complete restoration of filtration performance was achieved within 3 hours. Surface characterization using SEM, XPS, and FTIR spectroscopy confirmed the results. Finally, a reaction mechanism was proposed which was supported by density functional theory calculations, molecular dynamics simulations, and experimental investigations [3]: Exposure to high-energy electrons leads to activation of the polymer material, primarily by generating radical sites. Furthermore, radiolysis of water as solvent results in the generation of reactive species like OH and H radicals or solvated electrons, which subsequently activate the dissolved biomolecules mainly through H-abstraction reactions. Ultimately, immobilization via the establishment of covalent bonds is accomplished through radical recombination between activated polymer and biomolecules.

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Hexagonal Membrane Bundle Design: A Novel Approach to Combined Lung and Kidney Support for Preterm Neonates

Danny van Galen¹ (d.j.m.vangalen@utwente.nl), Niels Rochow², Ulrich Haag³, Christoph Fusch², Ana Martins Costa¹, Frank Halfwerk^{1,4}, Jutta Arens¹, on behalf of the ArtPlac Research Consortium⁵

KEYWORDS: Artificial Placenta, Pulmonary, Renal, Hexagonal, Neonatal

Introduction

Although neonatal mortality has declined worldwide in recent years, preterm birth remains the leading cause of neonatal death [1]. Many of these deaths are attributed to lung immaturity, occasionally exacerbated by renal failure [2][3]. Despite available therapies such as mechanical ventilation, extracorporeal membrane oxygenation (ECMO), and continuous renal replacement therapy (CRRT), the needs of extremely preterm infants (>24 weeks gestational age) are not fully met. These interventions are characterized by their invasiveness, association with lifelong disability, and reliance on multiple human-machine interfaces that hinder family integration [4][5].

To address these limitations, the ArtPlac project aims to develop a combined lung-kidney assist device that mitigates lifelong disabilities through a less invasive and family-centered approach. This abstract provides insight into the initial hexagonal membrane bundle design of the ArtPlac device.

Materials & Methods

User requirements were delineated through stakeholder analysis and subsequently converted into design requirements. Preliminary iterations of the blood inlet/outlet configuration and device housing were conceptualized and then transformed into 3D designs.

¹Dept. of Biomechanical Engineering, Engineering Organ Support Technologies group, University of Twente, Enschede, the Netherlands

²Dept. of Neonatology, Klinikum Nurnberg, Nuremberg, Germany

³Getinge, Hechingen, Germany

⁴Dept. of Cardiothoracic Surgery, Thorax Centrum Twente, Medisch Spectrum Twente, Enschede, the Netherlands

⁵HORIZON-EIC-2022-PATHFINDEROPEN-01, Grant agreement ID: 101099596

Results & Discussion

The device will be designed to be connected through the umbilical vessels, eliminating the need for additional vascular access in neonates. The system will operate without a pump, relying solely on the neonate's heart to reduce the risk of hemolysis. A hexagonal membrane bundle configuration will be used to accommodate three fluids (blood, gas, dialysate), with a 60° angle between the oxygenation and dialysis layers within the fiber bundle (see Figure 1). The potting will be done in a round fashion to avoid dead water areas.

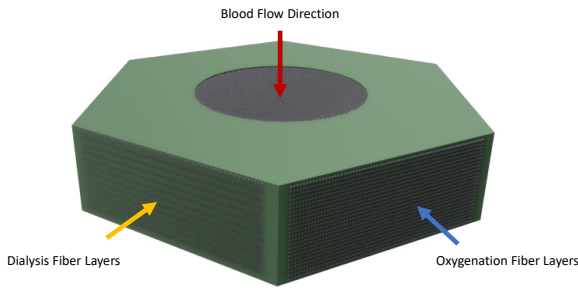


Figure 1: 3D render illustrating the configuration of oxygenation and dialysis hollow fiber mats encapsulated in potting material for phase separation of fluids and gas forming a hexagonal membrane bundle design.

This innovative hexagonal membrane design integrates both oxygenation and dialysis functionality in a single housing (see Figure 2). Future design objectives include minimizing pressure drop and limiting hemodilution by minimizing priming volume. ArtPlac is a breakthrough treatment paradigm designed to improve both survival and quality of life in the neonatal intensive care unit.

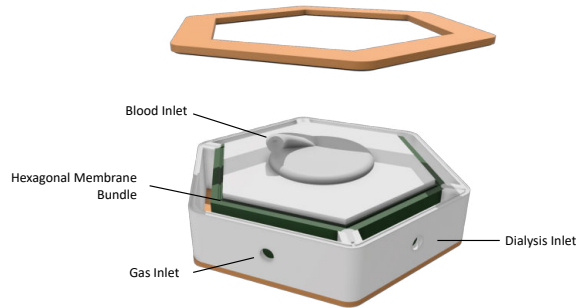


Figure 2: Initial concept design of the combined lung and kidney assist device.

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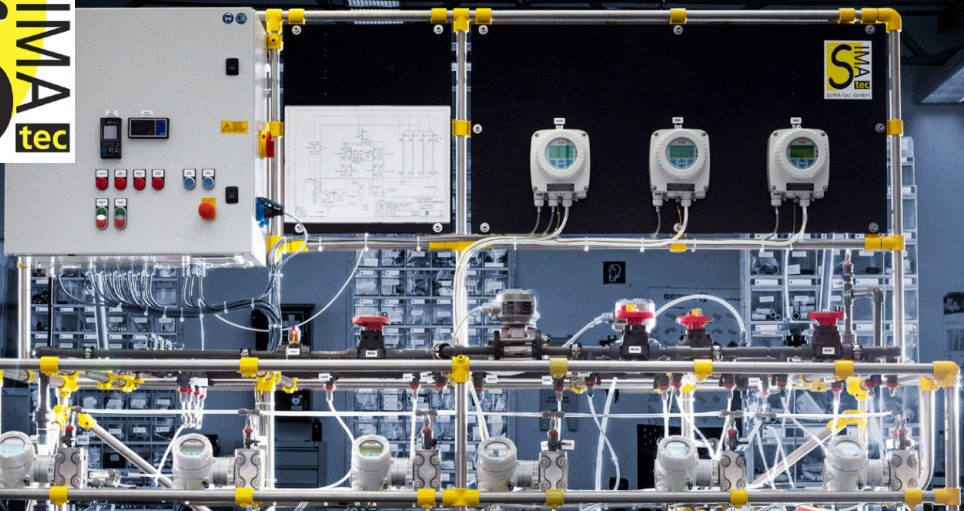
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Session 8

Keynote II

Membrane reactors for chemical production

F. Gallucci¹ (f.gallucci@tue.nl)

KEYWORDS: Membrane reactors, carbon membranes, Pd membranes

Abstract

Process Intensification is an important topic for the chemical industry as it can lead to an increase of energy efficiency and hence a decrease of anthropogenic CO₂ emissions. Process intensification can be achieved in different ways, and one is the integration of process functions in a single unit. In this respect, one can integrate the reaction and the separation in a single unit (a membrane reactor is an example).

Membrane reactors have been explored for different chemical production. Examples are CO₂ conversion, ammonia production, hydrogen production, etc.

In this talk, we will be focusing on the use of Pd membranes and carbon membranes both for dehydrogenation reactions such as reforming, cracking, and dehydrogenation reactions, where hydrogen is the main product or a by-product. Additionally, we will discuss the use of porous membranes for the production of chemicals such as DME and methanol production from CO₂, ammonia production as energy storage, etc.

The talk will summarize the research results of different projects such as Arena, Biocomem, Andreah, Macbeth, and Ambher, which have received funding from Horizon Europe or Horizon2020 workprogrammes.

¹Inorganic Membrane and Membrane Reactors, Sustainable Process Engineering, Eindhoven University of Technology, Eindhoven, The Netherlands

How Charged Membranes Recover Lithium: Separation of Ionic Mixtures

K.Brökelmann¹ (kerstin.broekelmann@avt.rwth-aachen.de), C.J. Linnartz^{2,1}, A. Wallraff¹, H. Scheulen¹, H. Roth^{1,2}, M. Wessling^{1,2}

KEYWORDS: Lithium recovery, Battery recycling, Salt mix separation, Polyelectrolyte multilayers, Nanofiltration hollow fiber membranes, Flow-electrode capacitive deionization, Salt metathesis

Abstract

In the transition to a fossil-free economy, lithium (Li^+) is emerging as a vital resource due to its primary utilization in Li^+ -ion batteries. However, aqueous ion separation is a major challenge in Li^+ -mining and Li^+ -recovery from Li^+ -ion battery (LIB) recycling processes. Specifically, the separation of Li^+ and magnesium (Mg^{2+}) is problematic for the former, and the separation of fluoride (F^-) from sulfate (SO_4^{2-}) and phosphate (PO_3^{2-}) is challenging for the latter. [1]

A suitable approach for the separation of monovalent and multivalent ions is nanofiltration. Polyelectrolyte multilayer hollow fiber nanofiltration membranes are a promising alternative to conventional nanofiltration membranes, offering ease for further tuning. The Layer-by-Layer (LbL) technique fabrication describes the alternating coating of oppositely charged polyelectrolytes onto a membrane support structure. By this, the pore size is reduced to a nanometer scale, and the electric charge of the membrane surface is customized in order to obtain a separation mechanism not only by size but also by electric charge of the dissolved species. [2,3]

Flow-electrode capacitive deionization (FCDI) utilizes ion exchange membranes (IEMs). With their incorporated fixed electric charge, they selectively separate anions and cations. In an electric field, ions migrate across the respective IEM towards the oppositely charged electrode. In the electrode chambers, the ions adsorb onto the flow electrode particles, which are pumped in a suspension to the other electrode, where the ions desorb and migrate through an IEM to the neighboring chamber. FCDI only transports ions, making it suitable for salt metathesis. The process water from LIB recycling contains mainly the low-solubility salt lithium fluoride (LiF). Within an FCDI module, a salt metathesis can be

¹Chair of Chemical Process Engineering RWTH Aachen University, Aachen, Germany

²DWI – Leibniz Institute for Interactive Materials, Aachen, Germany

performed to produce the more soluble salt lithium hydroxide (LiOH), which can be concentrated simultaneously. [4]

LbL nanofiltration membranes show promising separation performance for monovalent/multivalent cation and anion separation. FCDI provides excellent anion/cation separation, enabling an innovative LIB recycling process. Both membrane technologies enable Li⁺ recovery from salt lake brines and an innovative LIB recycling process for sustainable, continuous, and energy-efficient lithium recovery. In this study, we showcase LbL membranes as well as IEMs incorporated into FCDI as a suitable technology for Li⁺ recovery from various sources.

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Session 9

Gas and Vapor Separation

Design of High-Performance Gas Separation Membranes: From Research to Translation and Commercialization

Ingo Pinnau¹ (ingo.pinnau@kaust.edu.sa)

Abstract

Membrane-based gas separation systems were first introduced in the early 1980s for hydrogen recovery with polydimethylsiloxane-coated polysulfone hollow fibers and carbon dioxide removal from natural gas using cellulose acetate flat-sheet membranes. Since then, the gas separation application spectrum has continuously grown by applying glassy polymer membranes (e.g., tetrabromo-polycarbonate, polyimides, poly(phenylene oxide)) for nitrogen production and dehydration of air, CO₂ removal from biogas, etc. Reverse-selective rubbery silicone-based thin-film composite membranes were introduced for the recovery of organic vapors such as olefins from nitrogen-containing off-gases in the petrochemical industry in the mid-1990s. Currently, the annual membrane gas separation business has been estimated to be in the range of 1–1.5 billion USD.

Although hundreds of advanced membrane materials with significantly improved permeability and selectivity have been developed over the past three decades for a wide variety of industrial applications, only very few have been translated into high-performance membranes with properties sufficiently suitable for scale-up and commercial deployment. In fact, most industrially successful gas separation membranes are still made from commercial polymers with materials performance below the 1991 Robeson performance ‘upper bounds’. The limitations of newly developed membrane materials for gas separation applications are often linked to (i) materials scale-up, (ii) poor processability, (iii) insufficient mechanical properties, (iv) poor long-term stability, e.g., physical aging, (v) materials cost, and others.

This presentation will highlight some examples that overcame these bottlenecks and strategies to more efficiently translate and commercialize academic materials research in necessary collaboration with industrial membrane producers.

¹Chemical Engineering Program, Physical Science and Engineering Division, King Abdullah University of Science and Technology, Thuwal, Saudi Arabia

Industrial adaptation of polymeric membranes in humid high H₂S natural gas feed streams

Thijs Peters¹ (thijs.peters@sintef.no)

KEYWORDS: Natural gas sweetening, H₂S, CO₂, contaminants, performance

Abstract

The understanding of polymer membrane separation performance at high levels of H₂S and moisture containing natural gas is limited. For industry adaptation of commercial membranes in these fields, it is crucial to understand the membrane separation behavior under relevant field conditions. This presentation will highlight the separation performance of various membrane materials in H₂S-containing natural gas mixtures (up to 15 mol% H₂S) including various levels of humidity and in the presence of higher hydrocarbons such as toluene.

The systematic study of the gas separation performance has been carried out on self-standing films and asymmetric hollow fiber membranes. The separation performance in H₂S-containing mixtures (CH₄/CO₂/H₂S 80/5/15 mol%) was evaluated at 30 and 50 °C at 20, 35, and 50 bar in the presence of various levels of humidity up to 4500 ppm or 80% RH. Based on different feed-gas process conditions, the separation performance and the stability of the investigated materials are promising. Thanks to the combination of H₂S plasticization and competitive sorption in the presence of humidity observed experimentally, membrane technology can be attractive for humid natural gas streams with high H₂S content.

Introduction

Membrane-based natural gas sweetening is a technology with low capital cost, potentially high energy efficiency, small footprint, modularity, simple operation, and low maintenance, as well as minimal environmental impact. In addition, membrane technology, being modular, can be integrated with the rest of the gas treatment plant for de-bottlenecking the plants and to reduce the carbon footprint by diverting the H₂S and CO₂ molecules back to the reservoir through reinjection. As moisture and higher hydrocarbons are important constituents of natural gas fields, it becomes vital to understand the impact of contaminants on the membrane performance in an H₂S environment for fundamental understanding and application of membranes for industry adaptation. The current

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study thus aims to provide a thorough understanding of the separation performance of various membrane materials (Cellulose Triacetate (CTA), Pebax® MH1657 (a trademark of Arkema France), PolyActive™ 1500 (a trademark of Helmholtz-Zentrum Geesthacht)) in H₂S-containing mixtures (CH₄/CO₂/H₂S 80/5/15 mol%) including various levels of humidity and in the presence of higher hydrocarbons such as toluene.

Materials and Methods

The study has applied both self-standing symmetric films (CTA, Pebax® MH1657, PolyActive™ 1500) and asymmetric CTA hollow fiber membranes. All gas permeation measurements were conducted using the constant-pressure method analog to ASTM D3985 – 17. Investigated gas mixtures include a mixture containing 5% CO₂ in CH₄, a mixture containing 15% H₂S and 5% CO₂ in CH₄, and a mixture containing butane (3%) and a trace amount of toluene (300 ppm) maintaining 15% H₂S and 5% CO₂ in CH₄. Two temperatures, i.e., 30 °C and 50 °C, and three pressures ranging from 20 bar to 50 bar were considered.

Results and Discussion

The results have shown that the addition of humidity to the sour feed gas results in a slight increase of both H₂S/CH₄ and CO₂/CH₄ selectivity for all membrane materials. Figure 1 shows as an example the separation performance of CTA in the presence of 15% H₂S at 30 °C during humidity exposure up to 90% RH at 20 bar. Initially, the performance is left to stabilize in the sour feed gas (5% CO₂, 15% H₂S in CH₄) for around 4 days. This establishes a proper baseline prior to the humidity introduction. Then the performance in humid 15% H₂S is investigated at varying water vapor content over a period of around 200 hours. Finally, the performance in the dry sour feed gas (5% CO₂, 15% H₂S in CH₄) is again verified to investigate any hysteresis effects. The introduction of H₂S results in a minor gradually increasing behavior of both the CO₂ and H₂S permeability over time combined with a decrease in CO₂/CH₄ selectivity. Compared to the permeability prior to any H₂S exposure, the H₂S introduction results in an increase in CO₂ permeability from 8.1 to 9.6 Barrer at the expense of a reduced CO₂/CH₄ selectivity from 33.6 to 28.6 under the current conditions (30 °C and 20 bar).

Also for Pebax® MH1657, compared to dry operation, the CO₂/CH₄ and H₂S/CH₄ selectivity shows an increase upon humidity addition with 5% (CO₂/CH₄) and 10% (H₂S/CH₄). The CO₂ and H₂S permeability at 1700 ppm humidity (20 bar, 30 °C, 80% RH) decreases from respectively 96.7 and 507 Barrer to 90.5 (-6%) and 496 (-2%) Barrer.

Our hypothesis is that in the higher H₂S environment, the combination of H₂S plasticizing and hydrophilic nature of the polymers enables

the swelling of polymer chains, which allows the H₂S molecules to pass through, but there is still significant competitive sorption between condensable gases (H₂S, CO₂, and H₂O) versus CH₄, which helps in preserving or slightly increasing selectivity. Based on the obtained results at various pressures, temperatures, and different gas mixtures of sour gas (15% H₂S) with heavy hydrocarbons (up to butane, toluene), and up to high levels of humidity, it is evident that the performance and the stability of the investigated materials is promising. With the improvement of selectivity due to the combination of H₂S plasticization and competitive sorption in the presence of humidity, membrane technology can be attractive for humid high H₂S natural gas feed streams.

Conclusion

In the current work, the performance of various membrane materials in H₂S-containing natural gas mixtures (up to 15 mol% H₂S) including various levels of humidity and in the presence of higher hydrocarbons such as toluene. Based on different feed-gas process conditions, the separation performance and the stability of the investigated materials are promising. Thanks to the combination of H₂S plasticization and competitive sorption in the presence of humidity observed experimentally, membrane technology can be attractive for humid natural gas streams with high H₂S content.

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High Throughput Simulation (HTS) of Gas Separation Processes and the Application of AI

Peter Kreis¹ (peter.pk.kreis@evonik.com), Leif Nett¹, Markus Priske²

KEYWORDS: High throughput simulation, Gas separation, Membrane processes, Artificial intelligence, Multi-stage processes

Abstract

Resource efficiency is a decisive factor for the success of the chemical industry. One approach for the development of more efficient downstream processes is the early consideration of innovative separation processes, such as membrane processes, in process development. Membrane processes are increasingly applied, especially for the separation of gaseous mixtures. Industrial examples include upgrading of technical gases (hydrogen, helium, synthesis gas), biogas and natural gas, recovery of condensable valuable products from exhaust gases, and CO₂ capture.

Existing limitations in single-stage processes in terms of purity and yield can be overcome by intelligently combining membrane processes into multi-stage processes. Here, detailed/rigorous mathematical models are used for process design, which are usually integrated into commercial process simulation software. In order to take into account relevant influences of the operating parameters and non-idealities on the membrane separation and thus also to determine an optimal process configuration, very extensive simulations are necessary. This is where a method for High Throughput Simulation (HTS) starts, in order to run the complete parameter field in a very short time despite a combinatorial explosion in the parameter variation.

Examples of this include simple one-stage separations up to 2, 3 or 4 stage membrane processes, as well as the application of machine learning tools to predict the relevant parameters in (milli)seconds.

¹Evonik Operations GmbH, Process Technology & Engineering, Germany

²Evonik Fibres GmbH

Session 10

Membranes I

Reactive coating of desalination membranes in spiral-wound modules: Increasing antifouling properties and potentially enabling “second life” applications

Mathias Ulbricht¹ (mathias.ulbricht@uni-essen.de), Pascale Wünsch¹, Qirong Ke¹, Kibrom Alebel Gebru¹, Kevin Bojaryn¹, Franziska Blauth²

KEYWORDS: Water desalination, Membrane fouling, Hydrogel coating, Polyamide thin-film composite membrane, Spiral-wound module

Abstract

Fouling of reverse osmosis (RO) or nanofiltration (NF) membranes in water desalination or other water treatment applications, with negative effects on process performance and efficiency as well as membrane lifetime, is a challenge that is addressed by a number of different approaches. Building on prior own research [1], this study presents further developments of an *in situ* antifouling coating methodology for commercial RO and NF flat sheet membranes and spiral-wound modules, deploying a concentration polarization-driven reactive (“click”) coating formation process [2] and results from the first phase of a project devoted to recycling of used RO modules to enable their use for certain NF applications [3]. The transfer from lab-scale cross-flow experiments with membrane coupons to successful *in situ* reactive filtration cum coating of full-scale spiral-wound modules will be in the focus of this presentation. The study was performed using different commercially available polyamide thin-film composite membranes and spiral-wound modules. Upscaling to large-scale modules was studied with BW30 membranes and BW30-4040 spiral-wound modules from DuPont. “Clickable” polyzwitterionic building blocks [1] and analogous new polymers that can also be covalently cross-linked into antifouling hydrogels, in particular an easily accessible polyvinylalcohol (PVA) derivative [2], were used. The influences of “chemical” (type of polymeric building block and complementary cross-linker to “click” into a hydrogel; surface linker; concentrations; ...) and “engineering” parameters (initial flux, cross-flow velocity and feed spacer to impact the degree of concentration polarization; duration of reactive

¹Lehrstuhl für Technische Chemie II, Universität Duisburg-Essen, Essen, Germany

²Institut für Umwelt & Energie, Technik & Analytik e.V. (IUTA), Duisburg, Germany

filtration; ...) on coated membrane structure and separation performance were studied in detail. Correlations between the extent of flux decline during reactive coating as a function of parameters and the resulting coating thickness with impact on coated membrane permeance were established. Information from such correlations was then utilized for the transfer to spiral-wound modules, for example by keeping all “chemical” and almost all “engineering” parameters constant and only varying the duration of reactive filtration, to ensure on the one hand a sufficient thickness of the coating to improve fouling resistance but on the other hand to limit the reduction of permeance to $< 10\%$. Detailed characterizations, including autopsies of modules, yielded information about structure and homogeneity of the coating. Salt rejection was slightly improved, which can be explained by the “repair” of defects in the membranes by the coating. Lab- and pilot-scale studies confirmed the improvement of overall separation performance due to the antifouling coatings for most investigated application scenarios. Parallel lab-scale studies also yielded deeper insights into the interplay of different foulants and the specific antifouling coating structure, with consequences for the success of fouling mitigation [4]. The chemical stability of the polyacrylate-based zwitterionic hydrogel coating allows the use of acid cleaning, but it also enables the removal of a used (and no longer functional) coating under alkaline conditions, with the option to thereafter recoat the membrane to renew the antifouling functionality.

Finally, implications of the results of this work for methodologies that will enable a “second life” of the rapidly growing number of end-of-life (spiral-wound and other) membrane modules will be outlined, and first results toward recycling of used RO membranes by initially removing the polyamide layer and then applying a NF-selective layer will be presented.

Acknowledgments

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Development of Surface-Patterned RO Membranes with Pronounced Regular Microstructures and Their Anti-Scaling Performance in Feed Spacer-Filled Channels

M. Patel¹ (maharshi.patel@uni-due.de), S. Panglisch¹, I.M.A. ElSherbiny¹ (ibrahim.elsherbiny@uni-due.de)

KEYWORDS: Surface-patterning, thin-film composite membranes, feed spacer, scaling

Introduction

Numerous studies in literature have emphasized the potential advantages of surface-patterned TFC membranes in water treatment and desalination [1]. Imprinted surface patterns can induce certain mixing effects in the direct membrane vicinity that are claimed to mitigate biofouling and scaling [1][2]. Surface patterns can also increase membranes' active surface area, resulting in higher pure water permeability compared to their flat counterparts [4]. However, none of the respective studies investigated the performance of surface-patterned TFC membranes in combination with feed spacers, which are typical components in spiral-wound modules. Additionally, direct patterning of dense RO membranes (via thermal embossing methods using hard stamps) has always been challenging, since TFC membranes are exposed to significant

¹Chair for Mechanical Process Engineering / Water Technology, University of Duisburg-Essen, Duisburg, Germany

plastic deformation that can damage the crosslinked polyamide layer (as reflected by a drop in water permeability (PWP) and/or permselectivity [5]). So far, the preparation of surface-patterned TFC membranes via a two-step approach [3][4] (i.e., surface patterning of the membrane support followed by interfacial polymerization) has been the most reliable option. In this presentation, the development of surface-patterned RO TFC membranes with pronounced regular microstructures via the direct patterning thermal embossing method will be discussed. A systematic study of the imprinting conditions and their impacts on both the topographical characteristics and permselectivity of TFC membranes was conducted. Additionally, the results of lab-scale scaling experiments using surface-patterned TFC membranes, combined with typical diamond-shaped feed spacer, will be presented for the first time.

Experimental

Surface-patterned TFC membranes were prepared via hot embossing micro-imprinting lithography using brackish water FilmTec™ LC LE-4040 membranes and a brass mold (27 x 7 cm²) with a regular lines-and-grooves pattern (line width: 20 μm, line-to-line distance: 20 μm, groove depth: 10 μm). Different imprinting parameters were examined. The patterning fidelity was analyzed using a 3D optical profilometer, while topographical characteristics (e.g., roughness) were analyzed using scanning electron microscopy and atomic force microscopy. PWP and NaCl retention were examined using a lab-scale membrane testing unit. Anti-scaling performance, with/without feed spacer, was compared vs. two reference flat-sheet membranes (pristine membrane and compacted membrane under analogous conditions as surface-patterned membrane).

Results and Discussion

Surface-patterned TFC membranes with pronounced microstructures (pattern height up to 5 μm) were successfully achieved while maintaining a dense polyamide layer, see Figure ???. A correlation between pattern dimensions and PWP could be established. Additionally, surface-patterned membranes exhibited generally better anti-scaling performance than flat membranes, while performance results in cases of feed spacer-free and feed spacer-filled channels differ substantially. Effective combination of surface patterning and feed spacer design can potentially enhance RO membranes' performance.

Acknowledgments

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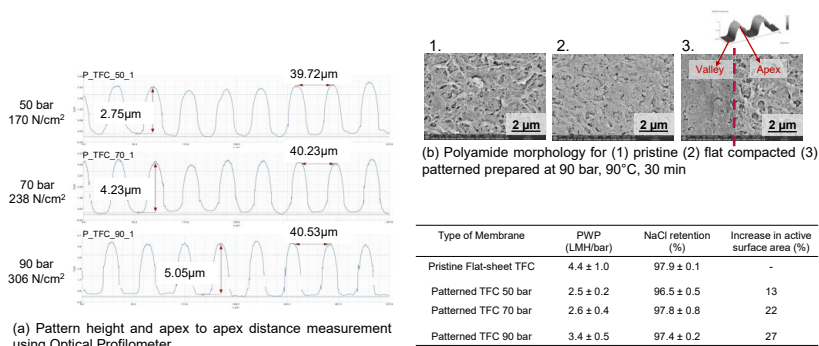


Fig.1. Pattern fidelity, topographical analysis, and performance for surface-patterned TFC membranes vs. pristine flat membrane.

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Scale-Up of Carbon Molecular Sieve Membranes Based on Polymers of Intrinsic Microporosity Precursors

Wojciech Ogieglo¹ (w.ogieglo@gmail.com)

KEYWORDS: Carbon molecular sieve membranes, thin-film composites, gas separation, scale-up, thin films, polymers of intrinsic microporosity

Abstract

In this contribution, we will present insights gained from our attempt to scale-up carbon molecular sieve (CMS) membranes based on polymers of intrinsic microporosity precursors from laboratory samples to pilot-scale modules. We will discuss the challenges encountered along the way which can be divided into challenges resulting from the inherent physics of thin microporous amorphous films as well as those related to the mechanical stability mismatches in multilayer composites. Those issues become quite severe when the active membrane area of the module grows beyond several cm². Finally, we will touch upon the attractive potential of fine-tuning of the separation properties CMS membranes by doping/surface modification as well as utilizing CMS membranes in other less explored application areas (e.g., pervaporation).

Introduction

Carbon molecular sieve (CMS) membranes are made by a pyrolysis process where a suitable organic polymer precursor is transformed into a microporous amorphous material during slow heating (>500 °C) under an inert (e.g., N₂, Ar, vacuum) atmosphere (see Figure 1). The resulting turbostratic carbon membrane material contains a significant volume fraction of relatively well-defined micropores which are able to efficiently discriminate permeating gas molecules based predominantly on their diffusivities (molecular sieving).

CMS membrane materials have been rather extensively investigated in the literature for a variety of separation problems such as natural gas processing, air separation, olefin/paraffin separations, and others. However, scale-up and commercialization efforts were unsuccessful mostly as a result of technical challenges related to their high cost, brittle character, but also to the propensity of traditional CMS precursor hollow fiber

¹King Abdullah University of Science and Technology, Saudi Arabia

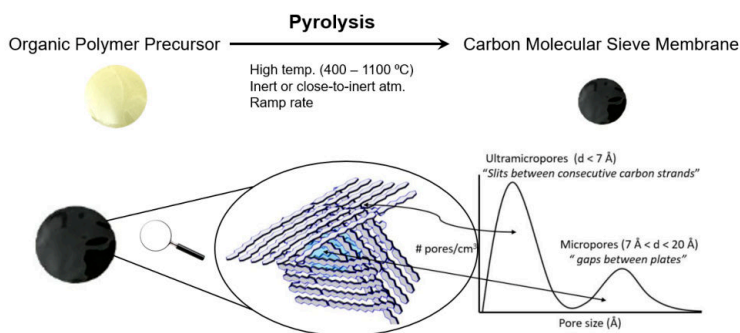


Figure 1: A scheme of CMS pyrolysis as well as their microporous morphology

membranes (polyimides, cellulose acetate) to collapse thereby forming dense low permeability structures.

In our approach, we attempted to address those challenges by:

- Employing a functionalized polymer of intrinsic microporosity (PIM) precursor which shows a much more limited tendency to structurally collapse before reaching thermal decomposition.
- Developing thin-film composite CMS membranes where the properties of the selective layer (CMS) can be varied and optimized independently from those of the underlying relatively inexpensive mechanical porous support.

Materials and Methods

Several kinds of polymer of intrinsic microporosity precursors were employed for the formation of CMS selective layers. Among the most promising ones were PIM-6FDA-OH and 6FDA-HTB polymers – both containing ortho-positioned -OH as well as -CF₃ functional groups. The -OH groups enable thermal rearrangement into polybenzoxazole structures which provides a further mechanism of micropore evolution (see Figure 2). The -CF₃ functional groups, on the other hand, are thought to play a significant role by providing cross-linking sites.

As a support for the selective CMS layers, we have explored several types of in-house synthesized and commercial porous inorganic structures including porous alumina discs and tubes and inexpensive porous stainless steel supports. In some cases, the supports contained separately fabricated interlayers of intermediate pore sizes (<100 nm) in comparison to the much larger pores in the bulk of the supports (several microns).

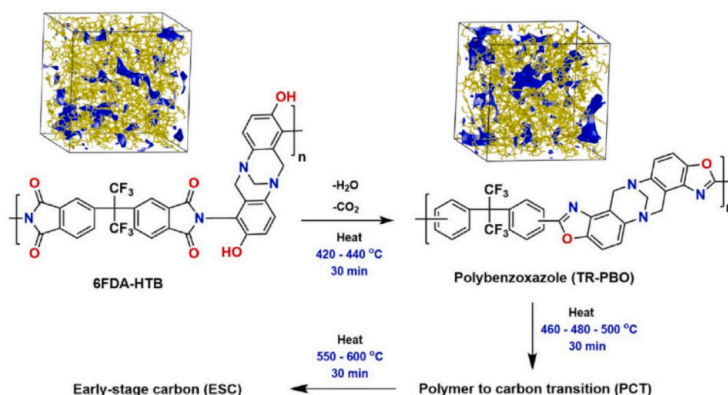


Figure 2: Structure of 6FDA-HTB CMS precursor as well as the scheme of the chemical reactions during the pyrolysis process

Results and Discussion

In contrast to a monolithic hollow fiber, CMS thin-film composite (TFC) structure allows independent optimization of the selective layer (usually in the 1-3 μm thickness range) and the supporting structure. In the course of the project, it became clear that the proper substrate interface preparation is necessary for the subsequent deposition of a defect-free separation layer – especially for larger active surface areas.

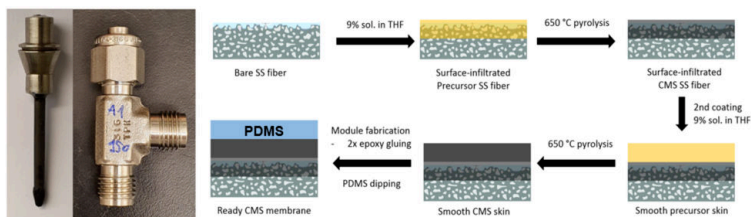


Figure 3: Stepwise development of the CMS selective layer by sequential polymer precursor coating pyrolysis (repeated twice) with a final application of a protective PDMS layer [1]

Figure 3 shows the simplest reliable TFC CMS membrane fabrication process where the substrate (in this case, porous stainless steel fiber outer surface) is exposed to the polymer precursor solution which after the 1st pyrolysis step forms the interlayer. This interlayer is significantly infiltrated into the pores of the support and would not by itself be sufficiently selective. In the 2nd coating and pyrolysis step, the actual well-defined separating layer is created which is further covered with the defect-sealing PDMS layer.

The process parameters space for this approach is relatively broad and needs to be individually tuned depending on the polymer precursor (solvent type, concentration, viscosity, target pyrolysis temperature, target film thickness, etc.). For example, the properties of the polymer precursor solution used to create the interlayer may differ from those for the final separating layer.

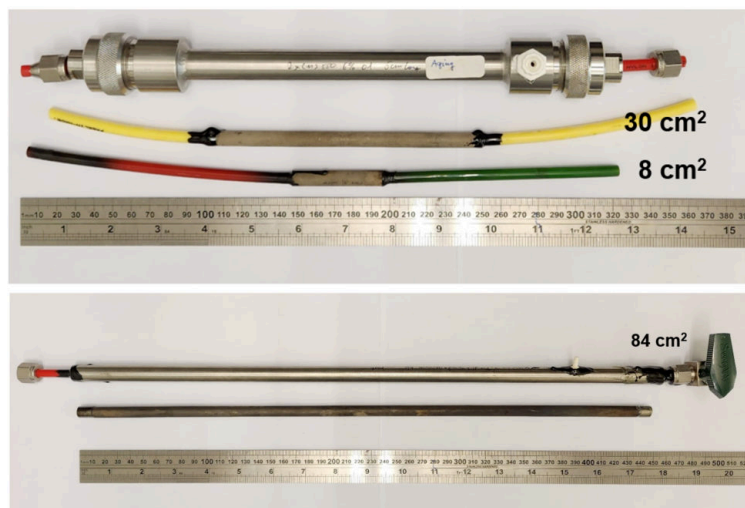


Figure 4: Illustration of a gradual progress in development of larger scale modules up to 84 cm² of active membrane surface

Figure 4 shows the gradual development of larger CMS active membrane surface areas up to the pilot-scale size (employing 45 cm long zirconia-coated stainless-steel microfiltration supports). In this case, to obtain acceptable gas separation properties, the fabrication parameters (especially the polymer solution concentration and pyrolysis ramp rate) were meticulously fine-tuned and an additional defect-control method based on the in-situ interfacial polymerization reaction was employed.

Although the fabricated TFC CMS PIMs-based CMS membranes were prepared for gas separation applications, they can also potentially be used in other more specialized application areas (such as aqueous-organic pervaporation [2]). Moreover, the separation properties of the presented CMS membranes can be further attractively tuned by surface modification/doping techniques.

Conclusion

The potential of scale-up of thin-film composite carbon molecular sieve membranes based on polymers of intrinsic microporosity precursors was

extensively explored. It was shown that careful optimization of the fabrication parameters as well as appropriate defect control allows for the preparation of pilot-scale CMS membrane modules. Subsequent flexible and facile surface modification techniques may further boost the performance of the prepared CMS membranes and extend their applicability to new separation problems.

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Session 11

EU Projects I

Development of a Ceramic Membrane-Based Process to Re-Refine Used Lubricant Oil

A. Buekenhoudt¹ (anita.buekenhoudt@vito.be), P. Vandezande, H. Richter, M. Weyd, C. Pflieger, A. Perez, W. Riedl

KEYWORDS: Ceramic membranes, Membrane extraction, Grafting, Lubricant oil, Pilot demonstration

Abstract

The EU project CUMERI aims to develop and demonstrate innovative customized membranes for greener and more resilient industries. Developments focus on two specific applications: H₂ recovery with CO₂ capture from coke oven gas in the steel industry, and two-step liquid filtration to re-refine Used Lubricant Oil (ULO) in the oil & gas industry. This contribution focuses on the Liquid Treatment Line where we envision that ultrafiltration (UF) followed by membrane extraction (ME) can replace current energy-intensive distillation-based re-refining.

ULO is a highly viscous stream containing, in addition to the base lubricant oil, some water, diesel, naphtha, and a high level of metal-containing impurities from lubricant additives, fouling, and wear. The viscosity directs to a high-temperature treatment with robust ceramic membranes for both UF and ME steps. Tuning of the chemistry of the membranes by grafting can further optimize process performance and stability in the low polarity oil. Experiments have shown that UF leads to a first important purification step, while ME can be used as a further metal polishing step. Results encompass fluxes, retentions, extraction rates, and efficiencies in lab-scale screening and parameter optimization in medium-scale proof-of-concept tests. Modeling is used for understanding and simulations. The processes are fine-tuned in collaboration with an industrial partner and end-user in the project.

In the next step, the two-step process will be demonstrated at TRL7 on-site at the end-user. To this end, an existing mobile ATEX filtration pilot unit from VITO will be used, adapted to the specific CUMERI needs. TEA and LCA have already been applied to gain first insights into economic and ecological benefits compared to the benchmark, and to pinpoint the most important membrane and process parameters to optimize.

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¹VITO, Belgium

Development of Membranes and Membrane Processes for H₂ and CO₂ Separation from Coke Oven Gas of Steel Industry

H. Richter¹ (hannes.richter@ikts.fraunhofer.de), O. David, R. Wyrwa, M. Etxeberria, M. Weyd, A. Vincent, G. Massasso, H. Sparkman, K. van Tilburg, T. Türken, F. Arslan, E. Unal, A. Buekenhoudt

KEYWORDS: SiC/SiCN-membranes, Bio-based PEBA membranes, H₂-separation, CO₂-separation, Coke oven gas

Abstract

CUMERI proposes a global approach for bridging separation needs in the steel industry and in particular on the coke oven gas, allowing the recovery of valuable H₂ while preventing CO₂ emissions from this waste stream. The typical composition of the coke oven gas from partner Erdemir, which is currently burnt for steam production, is 60.1% H₂, 20.4% CH₄, 7.8% CO, 4.9% N₂, 3.3% C_xH_y, 2.4% CO₂, 0.2% O₂, and 47 ppm H₂S. CUMERI partners propose to shift this towards a more circular value chain by developing SiC/SiCN membranes allowing H₂ permeation, whereas the relatively high concentration of H₂ in the coke oven gas, as well as its high temperature, is beneficial for the separation process. The separated H₂ in the permeate can then be reused in the steel-making process as a reducing agent. The retentate enriched in CH₄ and CO can be valorized in steam boilers. As this retentate stream is half of the coke oven gas stream, smaller combustion equipment is needed, thus reducing CAPEX. After burning (oxy-combustion), a typical CO₂ (80 vol.%) and N₂ (20 vol.%) containing waste gas is obtained. The relatively high CO₂ content of such a gas is a perfect match for the PEBA-polymer membranes developed in CUMERI, allowing CO₂ permeation and being highly selective for CO₂/N₂. The membranes will avoid CO₂ release into the atmosphere at low cost. Overall, the hybrid demonstration foreseen will cover the whole value chain: recovery and reuse of valuable H₂ using new SiC/SiCN membranes on tubular SiC-supports while avoiding CO₂ emissions due to the CO₂ separation from the burnt retentate using PEBA hollow-fibre membranes based on 100% bio-based polymer and produced in a green process.

¹Fraunhofer IKTS, Germany

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Preparation and Testing of Sustainable Membranes for MD Applications

A. Figoli, F. Galiano, M. Frappa¹ (m.frappa@itm.cnr.it), E. Drioli, O. Lorain, L. Leva, W. Yave, A. Angelini, A. Ali, H. F. Sulaiman, M. Fasano, A. Tiraferri, F. Macedonio

KEYWORDS: Green solvent, Sustainability, Membrane preparation, Membrane Distillation

Introduction

MEloDIZER is a European project funded under the Horizon Europe programme with the aim of implementing high-performance membranes and modules in strategic membrane distillation (MD) applications [1]. The overall goal of MEloDIZER is to provide the necessary step to transform membrane distillation (MD) into products that benefit industry and society [2]. The best design of this technology, when combined with the most appropriate membranes and mix of renewable energy resources, is the key to its effective application in different fields, both in industry and on a domestic or community scale.

Experimental/Methodological

One of the main activities of the first 18 months of the project was the fabrication and testing of PVDF-based membranes through the use of non-toxic solvents (e.g., N-butylpyrrolidone with the trade name Tami-solve) in both hollow fiber and flat-sheet configurations. Subsequently, in order to improve the MD performance of the membranes, they were coated with a fluorinated oligomer (trade name Fluorlink®), and the performance of the coated and uncoated membranes was compared. Moreover, the preparation and coating procedure were optimized to promote industrial scale-up.

¹Institute on Membrane Technology - National Research Council (ITM-CNR) c/o University of Calabria, Italy

Results and Discussion

The characterization of the fabricated membranes proved that the PFPE coating improved the hydrophobicity of the PVDF membranes. In particular, for commercial-based PVDF membranes, the contact angle increased from 132° to 150°. The performance of the various PVDF membranes was evaluated in MD devices using different synthetic and real wastewater solutions as feed in strong cooperation between the various partners involved in this activity (e.g., CNR-ITM, Aalborg University, Deltamem, Polytechnic of Turin, Polymem).

The comparison of the results achieved in the experiments allowed the identification of the best-performing membranes and operating conditions. For example, it was determined that the commercial-based PVDF membranes with coating did not get wet and maintained very high rejections when used in VMD configuration at permeate pressure not lower than 10 kPa or at feed temperature equal to or lower than 60-63°C. Furthermore, the analysis of real wastewater samples made it possible to identify which samples require pretreatment before concentration via the MD process.

Acknowledgments

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Session 12

Membranes II

Epoxide-based membranes: an emerging platform for liquid and gas separations

Dr. Rhea Verbeke¹ (rhea.verbeke@kuleuven.be)

KEYWORDS: Epoxide chemistry, robust membranes, interfacial polymerization, nanofiltration, gas separation

Abstract

Epoxide chemistry has been applied for many decades in a variety of industries owing to its excellent chemical, physical, and thermal resistance and the simple tunability of its chemical composition. However, in membrane technology, the use of epoxides for membrane synthesis has only recently been demonstrated by our group. In this presentation, I would like to give an overview of our research on epoxide-based membranes [1-5] and demonstrate that it can serve as a novel synthesis platform for liquid and gas separations.

The different membrane synthesis techniques that can be used to make epoxide-based membranes, such as thin-film composite (TFC) membranes and integrally skinned asymmetric (ISA) membranes, will be highlighted. To underline the versatility of epoxide chemistry and the available polymerization mechanisms, membranes with vastly different performances and physicochemical properties will be shown. To improve salt rejection for desalination applications and achieve CO₂/N₂ selectivity for gas separations, further densification of the interfacially polymerized thin film by inducing additional cross-links is required.

Besides desalination and CO₂/N₂ separations, the epoxide-based membranes could also purify aqueous streams containing solvents and were stable for 5 days in organic solvents. The membranes are also intrinsically thermally stable (up to 350 °C) and chemically stable in oxidizing, caustic, and acidic environments, as demonstrated by their full stability in sodium hypochlorite, NaOH (pH 14), and HCl (pH 0), respectively. Thanks 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 liquid and gas separations.

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¹KU Leuven, Belgium

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Tips for Developing Thin Film Composite Hollow Fiber Membranes by Dip Coating

Oguz Karvan¹ (o.karvan@emi-twente.nl), Jurjen Regenspurg, Ilse Pierik, Ozlem Kap, Patrick de Wit

KEYWORDS: Thin film composite, Hollow fiber, Gutter layer, Intermediate layer, Dip coating

Abstract

The thin film composite (TFC) approach using the dip coating technique is a simple and flexible option to utilize selective membrane materials that are either unable to fabricate self-standing films or fibers or are too expensive to use as the porous supports of integrally asymmetric structures. Besides the porous support and the selective layer, depending

¹EMI Twente BV

on the compatibility, thickness of the selective layer, and the level of pore penetration, an intermediate (gutter) layer may be necessary to apply. However, there is no off-the-shelf support and/or gutter layer to fabricate a TFC membrane. It requires an iterative total approach to tune the components for each other.

After proving the compatibility of the components, surface pore size, and pore size distribution of the support, compositions, and viscosities of the coating solutions are the most critical material parameters to optimize, while the residence time in the dip coating solution and the withdrawal speed are the key process parameters to investigate. Additionally, the wettability of the fibers plays a significant role in the coating process. It affects how well the coating solution spreads and adheres to the fibers, directly impacting the performance of the gutter layer. High capillary pressure can lead to excessive pore penetration and therefore reduce the permeance. Hence, controlling capillary pressure and optimizing fiber wettability are essential strategies to ensure uniform and defect-free coatings.

Starting from the targeted application and the materials selection, this project discusses the concerns, considerations, characterization techniques, and methodology for developing TFC hollow fiber membranes for gas separation applications, drawing conclusions from several R&D projects.

Visible-Light Active and Stable Bi_2WO_6 Composite Polymer Membranes for Photocatalytic Micropollutant Removal

Kristina Fischer, Zahra Niavarani¹
(zahra.niavarani@iom-leipzig.de), Amira Abdul Latif, Jan Griebel, Andrea Prager, Omid Shayestehpour, Stefan Zahn, Agnes Schulze

KEYWORDS: Photocatalysis, Microfiltration Membrane, Bismuth Tungstate

Introduction

Micropollutants pose a growing challenge in wastewater treatment. Traditional methods have limitations while photocatalysis with titanium dioxide (TiO_2) applied to membranes suffers from UV dependence and membrane degradation. Bismuth tungstate (Bi_2WO_6) offers a visible-light active alternative. This study explores Bi_2WO_6 immobilized on

¹Leibniz Institute of Surface Engineering (IOM), Germany

polymer membranes as a potential solution focusing on synthesis, characterization, and degradation experiments.

Materials & Methods

Bi_2WO_6 synthesis was optimized by varying reactant salt concentrations. It was then immobilized on polyethersulfone (PES) and polyvinylidene fluoride (PVDF) membranes. The membrane and the photocatalyst were thoroughly examined regarding their performance in degrading micropollutants from water.

Results & Discussion

Increasing reactant salt concentration enhanced Bi_2WO_6 crystallinity, leading to a significant boost in methylene blue degradation under UV light. Degradation increased from 16% to 95% after 80 minutes [1]. A higher amount of Bi_2WO_6 immobilized on a PES membrane surface improved its photocatalytic activity. Methylene blue degradation rose from 57% to 80% after 30 minutes of UV light activation [1]. The $\text{Bi}_2\text{WO}_6/\text{PVDF}$ achieved around 90% removal within 120 minutes under UV light, maintaining stable removal rates over 10 cycles (Figure 1) [1]. Bi_2WO_6 allowed propranolol degradation under visible light [1]. The $\text{Bi}_2\text{WO}_6/\text{PVDF}$ membrane exhibited excellent stability. Unlike TiO_2/PVDF membranes, which degraded after 7 days of UV exposure, the $\text{Bi}_2\text{WO}_6/\text{PVDF}$ membrane remained intact, highlighting its potential as a more durable alternative [1].

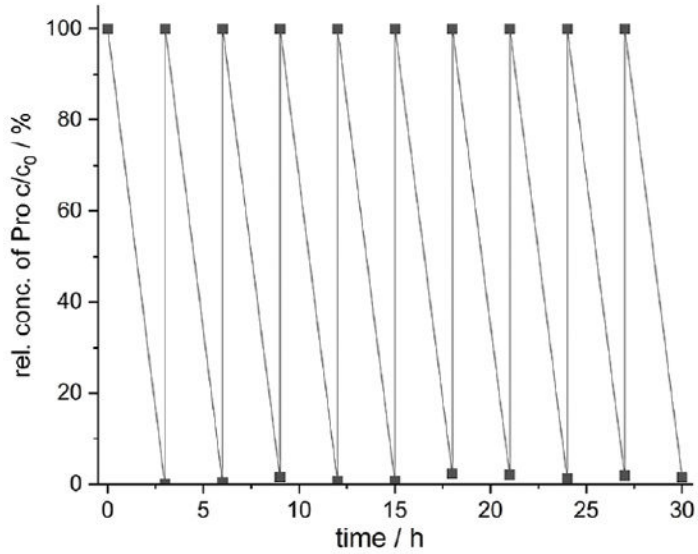


Figure 1: Degradation of propranolol over time for 10 cycles (each 3 h) under UV light activation.

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Session 13

EU Projects II

From Membranes to Processes Lifetime: An Overview of the Modelling Activities within MEASURED Project

Michele Ongis, Andraž Pavlišič, Felipe Parada Molina, Yvonne Van Delft, Blaž Likozar, Vladimir Dikic, Zanaçat Sahin¹
(z.e.sahin@modelta.nl)

KEYWORDS: Modelling, CFD, phenomenological, LCA, social-LCA

Introduction

Modelling is a fundamental support to fill the gap between research and industrialization. In the MEASURED project, modelling is a multi-scale activity that spans from the material study for membrane development up to module description, process design, and lifetime assessments. This work showcases the modelling activities performed to date for MEASURED's three main membrane lines: gas separation, membrane distillation, and pervaporation.

Methods

An overview of the modelling activities in MEASURED is reported in Figure 1. Initially, nano-/micro-/meso-scales are investigated through DFT and MD techniques to study membrane properties and the interactions with permeating molecules. At the macro scale, membrane module relevant features (such as temperature and concentration polarization) are studied through CFD. Moreover, simplified phenomenological models are developed from these studied features, minimizing the required computational power while also including descriptions of relevant phenomena. These models are then integrated at the process scale for the definition of process design and techno-economic optimization. Environmental and social impacts are evaluated through LCA and social-LCA.

Results & Discussion

Each membrane process studied involves specific modelling activities. For the gas separation process (for H₂ and CO₂ removal from methanation products), CFD analysis studies the effect of concentration polarization due to permeation through carbon membranes. In membrane distillation (for water purification), temperature polarization is studied. In pervaporation, the membrane is integrated into a reactor, and the

¹Eindhoven University of Technology, The Netherlands

effect on an esterification reaction is examined. From the system point of view, a harmonization procedure has been developed to coordinate the different analyses (techno-economic, environmental, social, and circularity) to ensure uniformity of the complementary results.

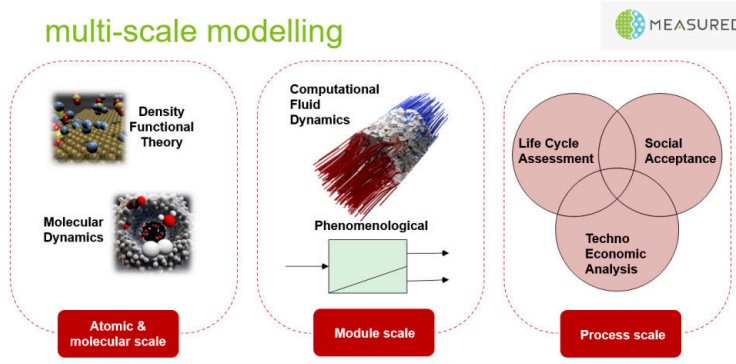


Figure 1: Overview of the modelling activities in MEASURED

Multi-scale Modeling of Membrane Distillation Demonstrators for Wastewater Treatment and Desalination in MEloDIZER Project

Aamer Ali, Matteo Fasano¹ (matteo.fasano@polito.it), Oliver Gloth, Joachim Koschikowski, Matteo Morciano, Roberto Raffaele Meo, Alba Ruiz-Aguirre, Alberto Tiraferri, Carlos Plana Turmo, Daniel Winter, Guillermo Zaragoza

KEYWORDS: Membrane distillation, Wastewater recovery, Desalination, Multi-scale modelling, Solar energy, Computational fluid dynamics

Abstract

The MEloDIZER project implements high-performance membranes and modules in strategic applications of membrane distillation (MD) aiming to advance MD technology significantly. Two prototypes (2-5 m³/day, 0.5-2 m³/day) are demonstrated in industrial facilities (textile, beverage, chemical industries) to reuse wastewater (70-90%), thereby reducing

¹Politecnico di Torino, Italy

water footprint, approaching zero waste, and recovering valuable nutrients from aquaculture wastewater. Additionally, two prototypes (50-100 L/day, 10-20 L/day) serve as low-cost passive decentralized units for delivering drinking water from saline and challenging sources at the community and family levels. These prototypes operate with 90-100% sustainable energy from waste heat and/or solar energy, with designs maximizing membrane and system performance.

Optimized modules focus on hydrodynamics and energy recovery improvements supported by multi-scale modelling to enhance membrane properties, customize module geometry, and increase system efficiency. Simulations clarify how membrane architecture and parameters affect performance for various media (e.g., acidic streams, salt brines, nutrient-rich waste streams) [1]. Fluid dynamics simulations address heat and mass transport, pressure drop, and fouling behavior influenced by channel and spacer geometry and flow regime. These simulations inform the design of optimized modules and spacers, leading to lower pressure drop, better mixing, optimal operating conditions, and brine recirculation [2]. Flat-sheet or hollow-fiber module geometry and arrangement simulations determine the optimal module topology using specific thermal energy consumption (STEC) as an objective function, with variables including membrane properties, operative conditions, spacers' characteristics, and feed water composition. The arrangement of modules in series and parallel will be optimized with a focus on maximizing the number of distillation stages for given thermal power and feed water quality. Energy device design and system thermodynamics performance are optimized through energy and exergy analyses at component, module, and system scales, identifying the most efficient configurations tailored to end-user conditions. Strategies for improving energy efficiency will be compared to balance efficiency and capital cost. Process control optimization focuses on minimizing idle time due to transients, energy supply intermittency, and maintenance.

Data from both modelling and testing will calculate energy performance and extrapolate technical performance for various feed waters and installation sites, supporting LCC, business case, and market entry analyses. The MEloDIZER project's comprehensive modelling approach will advance the demonstration of MD technology for wastewater treatment and desalination.

Acknowledgments

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nor the granting authority can be held responsible for them.

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Development & Scale-up of Advanced Membranes for Membrane Distillation, Gas Separation, and Pervaporation

A. Criscuoli, M. Raimondo, F. Russo, A. Corozzi, M.C. Carnevale, A. Gordano, F. Galiano, A. Figoli, E. Curcio, M. Aquino, P. Argurio, D. Venturi, M.A. Llosa Tanco, S. Agnolin, D.A. Pacheco Tanaka, M. Nikbakht Fini, M. van Tuel, Y.C. van Delft¹ (yvonne.vandelft@tno.nl)

KEYWORDS: PVDF sustainable membranes, direct contact membrane distillation, Hybrid Silica (HybSi) membrane, Pervaporation, Carbon molecular sieve membranes, gas separation

Introduction

Despite the strong progress in recent years, currently developed membranes do not fully meet the permeation selectivity and chemical / mechanical resistance required by the market. While novel nanomaterials and processing technologies have demonstrated superior performance at the laboratory scale, their Technology Readiness Level (TRL) remains low, presenting a challenge for scaling and deployment in industrial settings. The MEASURED project identifies three membrane processes—membrane distillation, gas separation, and pervaporation—targeted for scale-up to TRL7 in the chemical industry.

¹TNO, The Netherlands

Experimental/Methodology

For industrial wastewater treatment by Membrane Distillation (MD), PVDF membranes, prepared with greener solvents by CNR-ITM and supplied by GVS, were coated using procedures developed by CNR-ISSMC. Characterization included SEM, AFM, pore size determination, and surface energy measurements. Additionally, contact angles were measured with real industrial wastewater mixtures. Composite alumina-carbon molecular sieve membranes (Al-CMSM) for CH_4 polishing were prepared by the dip-dry-carbonization method. Two hybrid silica (HybSi) membrane variants, a standard and an acid-resistant type, were prepared for pervaporation using ceramic supports for dehydration of an acrylic ester mixture.

Results and Discussion

Coated PVDF membranes showed improved hydrophobicity and repellency, though a decrease in permeate flux was noted for smaller pore sizes. Gas permeation of Al-CMSM showed better performance at lower carbonization temperatures due to higher porosity. The standard HybSi membrane exhibited higher permeation flux than the acid-resistant HybSi, but stability was lower in acidic environments, with the standard membrane losing selectivity after a month.

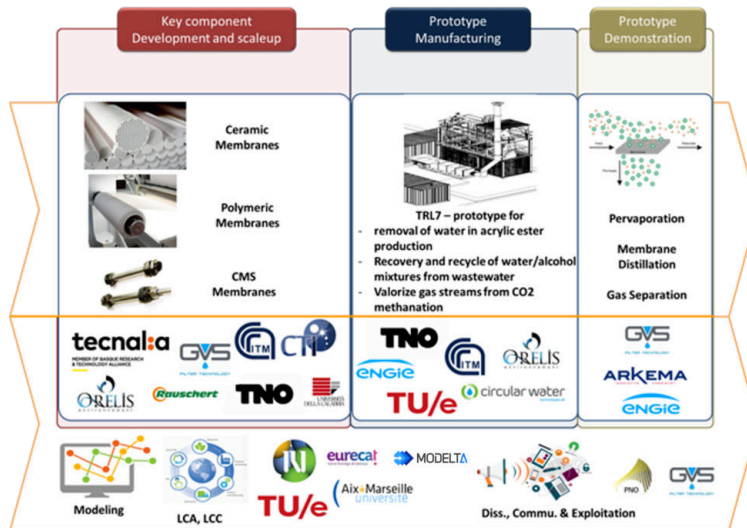


Figure 1: The MEASURED project value chain

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Session 14

Desalination II

Versatile fit-for-purpose membranes and processes

From water, wine and beer to milk & whey filtration and gas separation

Dr.-Ing. Jens Potreck¹ (jens.potreck@pentair.com)

Abstract

1. Ultrafiltration XF75 (and XF53) Development

Innovating the core, that was the starting point for the development of the new X-Flow XF75 and XF53 Membrane Element. With a fundamentally new UF membrane, a new internal design, and outer dimensions identical to those of the 55 and 64, the XF75 delivers a performance boost in a small package. The new X-Flow XF75 Membrane Element looks exactly the same as the well-known Pentair X-Flow XF64 and XF55 (also known as Pentair X-Flow Aquaflex 64 or Pentair X-Flow Xiga 64) Elements. However, the new membrane element is only the same on the outside. The firm's researchers and product developers have created a stronger membrane material that enables the fiber's inner and outer diameters to be modified, resulting in an impressive extension to 75 m² of active membrane surface area.

2. Smart S8 and Pentair Q

The BMF +Flux Smart S8 consists of a master controller and multiple membrane units. The core of the design is that the membrane units are able to filter and clean independently of each other, making it a self-foreseeing beer filter. Even when the quality of resources has fluctuations, the smart steering controller ensures the system runs as efficiently as possible. Furthermore, the smart steering controller decides which unit has to run, at which flow rate, and which unit can perform a cleaning. This implies a reduction of downtime to zero and a non-stop beer flow!

Pentair's Pentair Q is the brewer's virtual assistant to help excel in day-to-day business. The smart cloud-based software helps optimize the Beer Membrane Filtration process. Pentair Q's various dashboards extract data and turn them into valuable process insights, making manual data processing a thing of the past. The daily routines of operators and brewing managers are made easier, so brewers can focus on brewing a great natural product.

3. Milk & Whey Filtration

Available membrane technologies for the purification, fractionation, and

¹Pentair, The Netherlands

concentration of proteins in the dairy industry are largely based on spiral-wound and ceramic membranes. They both have advantages but also challenges impacting operation limits, consumptions, and costs.

In a recent work, our team investigated the use of hollow fiber membranes for the fractionation and concentration of milk proteins. Pentair's HF membranes meet industry quality standards with respect to casein content in permeate, showing consistent integrity during the tests. They have higher fluxes than commercial polymeric SW membranes and superior transmission of proteins, providing more than 2X the capacity of SW under identical process conditions.

Our hollow-fiber membrane modules are easy to drain, optimized for removing products at the end of runs. They allow for easy control of flux and retention and offer backpressure and backflush opportunities. They can operate with high levels of suspended solids and highly viscous solutions at high flux rates, providing customers with sustainable, low-operating cost solutions, with improved yield and selectivity compared to alternative membrane technologies.

4. Gas Separation

Improving energy efficiency in gas separation processes is today a major driver for competitiveness in our portfolio of Biogas and Carbon capture solutions. Most of the energy consumption in gas separation originates from compression systems that need to bring the different gaseous streams to the given pressure to meet product specifications. In the case of biogas, separation of CO₂ from methane is key to reaching gas specifications required by end users, such as gas grids, vehicles, fuels, etc. The work carried out by our teams consists of matching the best materials to the optimal process configurations to reduce power consumption, while considering the harsh reality of pollutant effects on membrane performance, either by clogging or permanent damage.

Experimental Investigation of Transport Properties of Ion-Exchange Membranes Equilibrated with Highly Concentrated Multi-Ionic Solutions

Giorgio Purpura¹, Valeria Guenther², Andrea Culcasi¹,
Alessandro Tamburini¹, Andrea Cipollina¹, Christian J.
Linnartz², Matthias Wessling², Giorgio Micale¹

KEYWORDS: Ion exchange membranes, Ionic diffusivities, Ionic conductivity, Multi-ionic solutions

Abstract

Ion-exchange membranes (IEMs) are a specific class of dense polymeric membranes widely adopted in all electromembrane processes for the sustainable production of water, energy, and chemicals, and for the valorization of waste brines. In recent years, the pressing need for more efficient separation processes has pushed forward the development of highly selective membranes to recover specific trace elements from seawater or salt-lake water brines [1]. A deep and thorough knowledge of ionic transport properties of IEMs and how they are affected by IEM's chemical structure and composition is required to produce tailor-made membranes. Recent works on the topic highlighted that at low external salt concentrations, species selectivity is mainly dominated by long-range electrostatic interactions between mobile ions and fixed charged groups in the polymeric matrix of the membranes. Therefore, by considering these interactions, thermodynamic and transport models have been proposed to predict partitioning coefficients and ionic diffusivities in the membrane phase [2]. However, most of these works focused on IEMs equilibrated with diluted and moderately concentrated single-salt solutions. Consequently, a fundamental understanding of how the transport properties of IEMs are affected by the system composition is still missing. This lack of fundamental knowledge is even more significant when dealing with highly concentrated brines. In such systems, long-range interactions among the charged species are screened greatly because of the high ionic strength, and the transport properties are likely to be mainly influenced by species-specific local interactions [3][4]. Therefore,

¹Dipartimento di Ingegneria, Università degli Studi di Palermo (UNIPA), Viale delle Scienze Ed 6, 90128 Palermo, Italy

²Chemical Process Engineering AVT.CVT, RWTH Aachen University, Forckenbeckstraße 51, 52074 Aachen, Germany

our research aims to investigate thoroughly the effects of concentration and composition on IEM transport properties. An extensive experimental campaign was carried out to determine partitioning coefficients, ionic diffusivities, and membrane conductivity of a commercial homogeneous cation-exchange membrane equilibrated with multi-ionic solutions over a broad concentration range, including Na^+ , K^+ , Ca^{2+} , Mg^{2+} , and Cl^- . These results provide valuable insights into the behavior of different ions in IEMs, elucidate the effect of concentration and composition on IEM transport properties, and through this understanding, pave the way for advancements in IEM design and modeling.

Acknowledgments

This work was supported by the EU within SEArcularMINE (Circular Processing of Seawater Brines from Saltworks for Recovery of Valuable Raw Materials) project – Horizon 2020 programme, Grant Agreement No. 869467. This output reflects only the author’s view. The European Health and Digital Executive Agency (HaDEA) and the European Commission cannot be held responsible for any use that may be made of the information contained therein.

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Advances in Two-Compartment Electrodialysis Configurations with Bipolar Membranes for Chemicals Production

A. Filingeri¹ (antonia.filingeri@unipa.it), F. Vicari¹, A. Culcasi¹, A. Tamburini^{1,2}, G. Micale¹, A. Cipollina¹

KEYWORDS: Brine valorization, performance indicators, electro-membrane

Introduction

Electrodialysis with bipolar membranes (EDBM) is a process characterized by repeating units, named triplets, consisting of three membranes (anion-, cation-, and bipolar) and three channels (acid, base, and saline). EDBM can be integrated within circular treatment chains to produce in-situ acid and base reagents to be used by the other technologies. However, bottlenecks to the widespread EDBM implementation include the high membrane cost and energy/water consumption. This work aimed to explore the performance of EDBM in new configurations with two and three compartments to reduce membrane area and water footprint.

Materials and Methods

A lab unit, assembled with 5 repeating units, was tested using 2.0 mol/L NaCl solution fed to either i) the saline compartment or ii) the saline and base ones, at current densities in the range of 200-400 A/m². In addition, the two-compartment configuration was implemented by combining the salt and basic channels into one. In both configurations, a 0.05 mol/L solution of HCl was fed to the acid channel. The two configurations were tested under similar operating conditions and compared in terms of current efficiency (CE), specific productivity (SP), and specific energy consumption (SEC).

Results and Discussion

Results showed that the two- and three-compartment configurations reached similar SEC, in the range of 1.2-1.5 kWh/kg NaOH. Although fewer membranes in the two-compartment configuration reduced the applied voltage, this effect was offset by a 12% reduction in CE. Conversely,

¹Dipartimento di Ingegneria, Università di Palermo, Viale delle Scienze Ed. 6, 90128 Palermo, Italy

²ResourSEAs SrL, Palermo 90128, Italy

in the two-channel configuration, the alkaline SP increased by 25% at 300 A/m² and water consumption was reduced by 50%. This study revealed new potential configurations of interest for applications of EDBM in circular approaches to produce chemicals.

Acknowledgments

This work was supported by the EU within the SEARcularMINE (Circular Processing of Seawater Brines from Saltworks for Recovery of Valuable Raw Materials) project – Horizon 2020 programme, Grant Agreement No. 869467. This output reflects only the author’s view. The European Health and Digital Executive Agency (HaDEA) and the European Commission cannot be held responsible for any use that may be made of the information contained therein.

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Posters

Water treatment

Real-Time Fluid Dynamics Analysis for Surface-Patterned Thin-Film Composite Membranes in Spacer-Filled Channel using Particle Image Velocimetry

M. Patel¹ (maharshi.patel@uni-due.de), A. Mitranescu², J.E. Drewes², S. Panglisch¹, I.M.A. ElSherbiny¹ (ibrahim.elsherbiny@uni-due.de)

KEYWORDS: Surface-patterning, thin-film composite membranes, fluid dynamics, feed spacer

Introduction

Surface patterning of membranes is a promising approach to overcome limitations due to feed spacers in spiral-wound modules (i.e., biofoulants accumulation, increased pressure drop) and promote their performance in water treatment applications. Simulations and lab-scale experiments, conducted in spacer-free channels, revealed that surface-patterned membranes can modify fluid characteristics in direct membrane vicinity, and accordingly, decrease both membrane fouling and concentration polarization [1][2]. Nevertheless, there is a knowledge-gap regarding fluid characteristics atop surface-patterned membranes in spacer-filled channels. Additionally, these effects are decisively influenced by surface pattern design, feed spacer characteristics, and operating conditions. Several mechanistic explanations (e.g., vortex shielding, secondary flow generation, shear-induced back-diffusion) were proposed in literature [3]; however, they were rather case-specific, and sometimes contradictory. To date, there is no solid experimental evidence concerning the improved fluid characteristics in the immediate vicinity of surface-structured membranes. The current study aims at sufficient understanding of the effect(s) of topographical membrane surface modification on fluid characteristics and examines reliable mechanisms for particles (or foulants) deposition on surface-patterned membranes in spacer-filled channels by means of real-time fluid dynamics analysis using Particle Image Velocimetry (PIV). Additionally, the outputs are compared with the results of parallel computational fluid dynamic (CFD) modeling efforts simulating the hydrodynamics on surface-patterned membranes in spacer-free and spacer-filled channels.

¹Chair for Mechanical Process Engineering / Water Technology, University of Duisburg-Essen, Duisburg, Germany

²Chair of Urban Water Systems Engineering, Technical University of Munich, Garching, Germany

Experimental

Surface-patterned TFC membranes were prepared by direct patterning of commercial FilmTec™ LC LE-4040 membranes via hot embossing micro-imprinting lithography using a brass mold with regular lines-and-grooves pattern (line width: 20 μm , line-to-line distance: 20 μm , groove depth: 10 μm). The flow in the membrane fouling simulator was set at a constant crossflow velocity of 0.15 m/s. Tracer particles with different characteristics were employed. Shake-the-Box (STB, Lagrangian Particle tracking) PIV with instantaneous measurement of three velocity components in a complete 3D measurement volume was employed. Different operating conditions (with/without permeation, with/without feed spacer) were examined.

Results and Discussion

Fluid dynamics measurement using STB in spacer-free channels at non-permeation and permeation conditions revealed that surface-patterning can indeed modify fluid characteristics in the feed-retentate channel. This was consistent with our earlier measurements using planar 2D-PIV [4], where vector velocity profiles showed substantially improved fluid characteristics for surface-patterned membranes compared to flat-sheet membranes. Using the volume flow 3D reconstruction function, fluid characteristics at different Z-distances from the surface-patterned membrane surface in spacer-filled channels are measured to investigate the joint effects of surface patterning and feed spacer design on hydrodynamics and tracer particles deacceleration patterns. These results are used to validate CFD modeling results suggesting that the hydrodynamics effects (e.g., velocity field, wall shear stress) in the vicinity of surface-patterned membranes in spacer-filled and spacer-free feed channels differ substantially.

Acknowledgments

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Ion Exchange Behavior in Polyelectrolyte Multilayer Nanofiltration Membranes: Impact on Salt Rejection Measurements

Áron Bóna¹ (bona.aron@pen.uni-pannon.hu), Ildikó Galambos, Nándor Nemestóthy

KEYWORDS: nanofiltration, polyelectrolyte multilayer, ion exchange, salt rejection

Abstract

The Layer-by-Layer assembly method is widely utilized to produce highly selective experimental and commercial polyelectrolyte multilayer nanofiltration membranes [1]. During this process, the oppositely charged polyelectrolytes bind together, forming intrinsic ion pairs and creating a thin polyelectrolyte complex active layer. However, some ionic functional groups of the polyelectrolytes remain unbound, forming extrinsic ion pairs with counter ions (most commonly Na^+ and Cl^-) found in the surrounding solution [2]. These act as dynamically changing ion exchange sites, significantly affecting salt rejection measurements under certain conditions. Divalent salt rejection is a key parameter for fine-tuning the Layer-by-Layer process parameters, highlighting the importance of identifying and addressing this error source for future membrane development efforts.

The effects of various pure water washing and annealing protocols on the amount of extrinsic ion pairs and salt rejection measurements are presented. Ion chromatography and conductivity measurement techniques are compared, pointing out that even selective ion chromatography results can yield inaccurate results when salt impurities remain bound in the polyelectrolyte complex. The effect of various polyelectrolytes on the ion exchange reaction rates is also investigated. Finally, we suggest principles and practical protocols for achieving accurate salt rejection values.

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¹Gebze Technical University, Türkiye

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Biofouling visualization in spacer-filled membrane filtration channels using OCT and CT

Kees Theo Huisman¹ (khuisman@hbku.edu.qa), Luca Fortunato, Johannes S. Vrouwenvelder, Peter Desmond, Bastiaan Blankert

KEYWORDS: Reverse osmosis, biofouling, spiral wound element, feed spacer, optical coherence tomography, automated image processing

Abstract

Spiral wound membrane elements are widely applied in reverse osmosis and nanofiltration, yet they are prone to biofouling [1]. Optical coherence tomography (OCT) has become a key technology for non-invasive monitoring of biofouling, but imaging challenges arise due to the complexity of detecting and visualizing feed spacers [2, 3, 4, 5]. This study presents an automated 3D OCT image processing method that utilizes a computed tomography (CT) scan of the feed spacer to generate a template, which is then overlaid onto the OCT image. This approach corrects distortions, reduces background noise, and enables precise visualization and quantification of fouling with reference to the feed spacer and membrane. By reproducible image alignment, this method allows

¹Hamad Bin Khalifa University (HBKU), Qatar

systematic comparison between datasets, and significantly enhances the ability to study and analyze biofouling in RO systems.

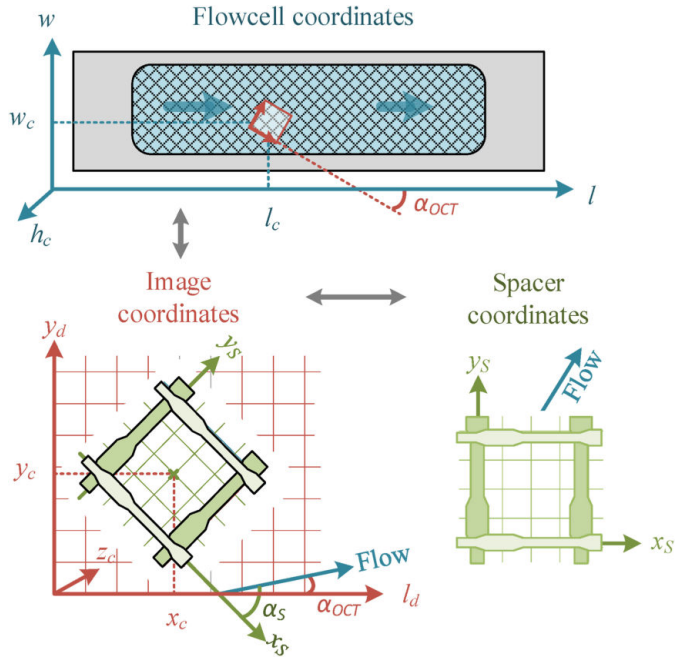


Figure 1: Three coordinate systems are used in this study: flow cell coordinates, image coordinates, and spacer coordinates.

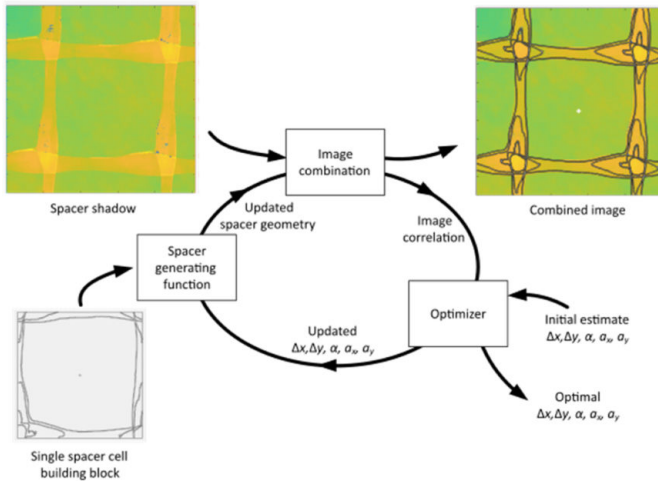


Figure 2: Fitting procedure to combine an OCT-scan with the spacer geometry using a MATLAB algorithm.

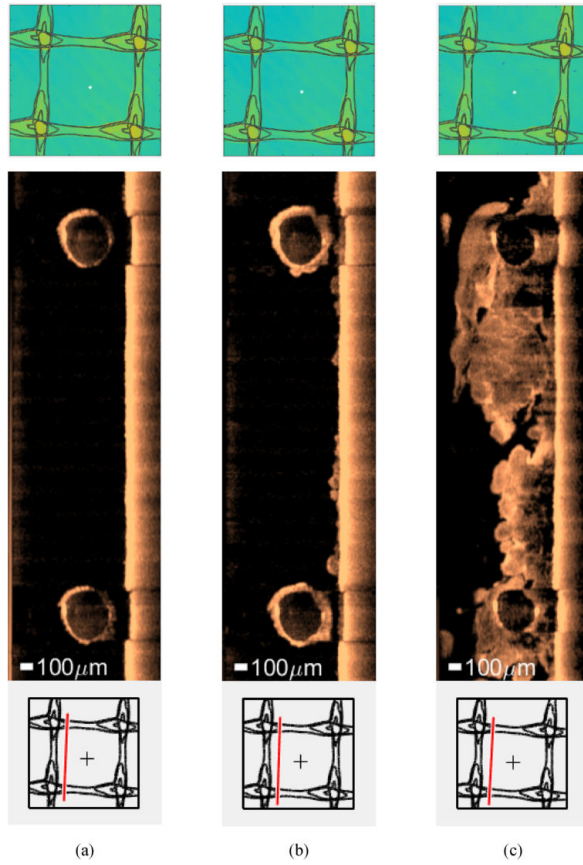


Figure 3: Examples of the fit between the feed spacer geometry and the spacer shadow for different fouling situations.

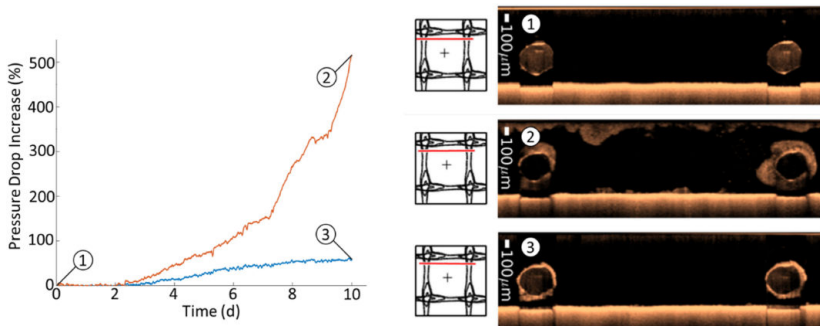
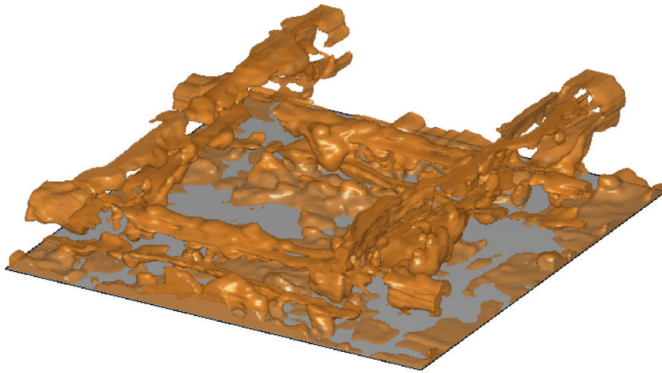
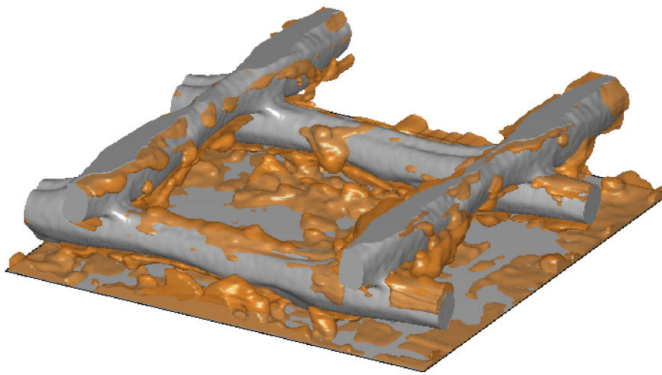


Figure 4: 2D OCT image slices from two different flow cells at different moments at different feed channel pressure drop.



(a)



(b)

Figure 5: 3D visualization of fouling on the membrane and feed spacer by fitting the spacer geometry onto the OCT image.

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A three-step water remediation scheme for contaminant removal and recovery of valuable species

Subhash Kumar¹ (subhashkumar@iisc.ac.in), Yagnaseni Roy¹

KEYWORDS: Arsenic remediation, Resource recovery, Zero waste, Sustainability, Sludge management

Introduction

Water contamination is a global issue, with groundwater arsenic contamination being a significant concern that affects over 230 million people worldwide [1]. In response, a three-step water remediation scheme has been developed, as depicted in Figure 1. Although designed to address

¹Centre for Sustainable Technologies, Indian Institute of Science, Bengaluru, India

arsenic contamination, the scheme is versatile and can readily apply to contaminants such as fluoride, lithium, and various metals and non-metals. Moreover, it has industrial applications for recovering valuable metals or products, promoting circularity. The scheme comprises three components: an adsorption-desorption unit, a membrane unit, and a biodigester or a precipitation unit. Contaminants such as arsenic or fluoride are removed through adsorption. Once the adsorbent reaches saturation, it is regenerated using either a NaOH or HCl wash solution, depending on the charge of the species. The regeneration process produces a NaOH/HCl solution rich in arsenic or fluoride, which is then fed to the membrane unit. In the membrane unit, the arsenic or fluoride is concentrated in the retentate, while the NaOH/HCl solution is recovered from the permeate and reused for further regeneration. The arsenic or fluoride retentate is then fed in either a biodigester or a precipitation unit for bioremediation or recovery in its salt form. This study focuses on the membrane part where arsenic study is carried out.

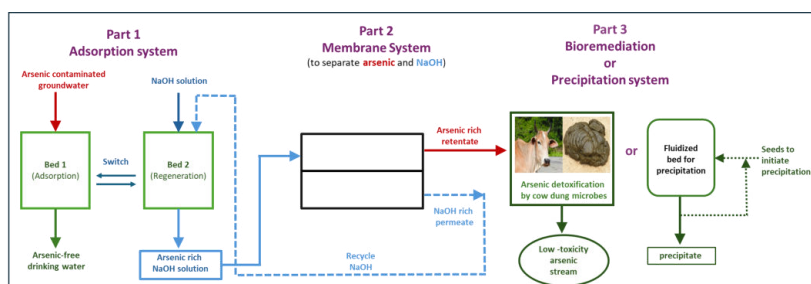


Figure 1: Schematic diagram of the three-step water remediation scheme.

Methodology

Experiments to verify that arsenic separation from the sodium hydroxide wash is possible using the membrane were carried out using a stirred cell dead-end setup with Hydracore70pHT membrane. In each experiment, the dead-end cell was filled with a high-pH arsenic solution, and the sodium hydroxide concentration was fixed based on the desired pH. The desired pressure was achieved using N_2 gas, regulated by a pressure regulator. The concentration of arsenic and NaOH is measured for permeate and retentate samples. Based on the experimental results, system

scale modeling is done to calculate the mass recovery of each component in the permeate and retentate side.

Results and discussion

For the proposed scheme, it is desirable to get a low concentration of As, a high concentration of NaOH, and high water recovery in the permeate. Experimental results with Hydracore70pHT membrane show 85% As(III) and 95% As(V) rejection at 30 bar with a feed concentration of 500 ppb at pH 11, indicating low As concentration in permeate. Based on the experimental results, system-level modeling predicts 82% As(III) and 94% As(V) mass recovery in retentate. However, the experimental NaOH rejection is 65%, with a predicted mass recovery of 24%. Ongoing work focuses on reducing NaOH rejection and enhancing mass recovery. The findings will be presented at the conference. The modeling results obtained for arsenic and water are in good agreement with the desired outcomes of the scheme.

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Extraction of curcumin and its concentration using organic solvent nanofiltration

Milana M. Mavinkurve¹, Yagnaseni Roy¹ (yroy@iisc.ac.in)

KEYWORDS: Organic solvent nanofiltration, Curcumin, Solvent recovery, Second law efficiency

Introduction

Curcumin is a polyphenol derived from the turmeric plant and an essential bioactive component in pharmaceutical products. It has multiple medicinal properties, such as anti-inflammatory, anti-cancerous, anti-viral, anti-microbial, and antiseptic properties [1]. With an annual growth rate of 16%, the global market value of curcumin is expected to reach 191.89 million USD in 2028 [2]. However, extraction of curcumin is challenging due to its low solubility and heat degradation. Curcumin

¹Center for Sustainable Technologies, Indian Institute of Science (IISc), Bengaluru, Karnataka, India

is extracted from turmeric by a solvent that dissolves it and removes it from the plant material. The resulting dilute solution of curcumin is traditionally concentrated using evaporation, which requires high temperature and energy. Pressure-driven membrane technology, such as nanofiltration, is a promising alternative to evaporation, as it avoids phase transition, significantly reduces energy requirements, and lowers operating temperatures to prevent thermal degradation of curcumin. A patent has been applied for the same (Indian Patent No: 202441000534).

Experimental/methodology

To devise an advantageous nanofiltration system to isolate high-quality curcumin, efforts must be made to optimize the system's energy requirement while maintaining a high solvent recovery ratio and curcumin separation efficiency. Computational modeling was carried out to calculate the curcumin extract concentration using nanofiltration at varying operating conditions. First, a transport model was developed based on the hindered transport theory (HTT) to evaluate curcumin rejection [3]. The developed transport model was extended to the system scale to evaluate the curcumin extract concentration along the nanofiltration unit. The second law (thermodynamic) efficiency, system sizing, solvent recovery, and curcumin yield of the concentration process in nanofiltration were calculated and compared with multi-effect evaporation at various operating conditions.

Results and discussion

Nanofiltration was found to have better thermodynamic efficiency, twice the efficiency of evaporation for the same final concentration of concentrated curcumin. Although the active curcumin flow rate in multi-effect evaporation (MEE) is higher than in nanofiltration, most of the curcumin in MEE is degraded and wasted due to heating, while it is not in nanofiltration and can be recycled. The amount of solvent recovered in nanofiltration is higher than the solvent recovered in MEE. The set of most advantageous operating conditions for concentrating curcumin-ethanol solution to its saturation point using nanofiltration is achieved. The developed model, coupled with lab-scale nanofiltration results, targets optimizing solvent selection and membrane choice for curcumin isolation, enhancing overall system performance.

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Preparation and characterization of metal organic frameworks/graphene oxide composite membranes for water purification

Gülcihan Ozdem¹, Ilkay Hilal Gubbuk¹, Ozlem Altintas Yildirim¹, Hakan Erer¹, Mustafa Ersoz¹ (ersozm@gmail.com)

KEYWORDS: Membrane, electrospinning, graphene, ZIF-8

Abstract

Polymeric membranes are used in water filtration in two ways: adsorption and size-based exclusion. Depending on the pore size of the membrane, different types of impurities can be removed from the aqueous solution. Microfiltration (MF) membranes have pore sizes in the 0.1-5 mm range and remove particles such as protozoa and bacteria. Nanofiltration (NF) membranes remove particles of approximately 0.001-0.01 mm, and reverse osmosis (RO) membranes remove particles of 0.001-0.0001 mm.

The development of polymeric nanofibrous membranes with versatile properties such as high flux, selectivity, antibacterial and antifouling characteristics is a growing field of research. In the presented study, MOF (ZIF-8) and GO-doped PAN nanofiber membranes were prepared and characterized.

Introduction

The demand for nanostructured membranes exhibiting high selectivity and efficiency has increased the interest in membrane design studies for

¹Selçuk University, Türkiye

a sustainable membrane production based on the obtainment of controlled pore size and homogeneous distribution of these pores in the membrane structure. Therefore, the integration of advanced technology nanoscale processes with each other is very important for the production, modification, and functionalization and structuring of membranes [1]. Membrane modification is a versatile technique for the production of novel electrospun membranes with numerous properties. In this study, the preparation of graphene/MOF hybrid membranes with controlled pore size and a certain morphology, the establishment of a reproducible production process and detailed characterization were aimed.

Experimental Section

The components of the polymer solutions prepared for producing fibers by electrospinning include DMF as the solvent, PAN, PAN/GO, PAN/ZIF-8, and PAN/GO+ZIF-8 polymer solutions for nanofibers. A 10% PAN solution and PAN/GO were added to DMF as powder and mixed at 800 rpm for 2 hours at 50°C. For GO and ZIF-8 addition, a pre-dispersion process with sonication was used instead of direct mixing, ensuring homogeneous dispersion in DMF. For nanofiber membrane preparation, the solution was taken into a syringe, ensuring no air bubbles remained, and placed in the automatic pump of the device. The flow rate and distance between the needle tip and collector plate were adjusted, and after setting the voltage, the device was started.

Results and Discussion

The prepared membranes were characterized by thermal analysis (TA), infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and EDX measurements [2]. All measurements are explained in detail and compared with references.

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Posters

Membranes

Fabrication and Characterization of Anti-Biofouling Membranes Immobilized with Live Quorum-Quenching Bacteria Using Polymers

Jinwoo Kim¹ (rlawlsdn521@gmail.com), Eunkyung Bae, Hyeona Park, Hyeonuk Yeo, Jongho Jeon, Kibaek Lee, Kwang-Ho Choo

KEYWORDS: Biofilm, Quorum quenching, Anti-Biofouling, Immobilizing bacteria

abstract

Biofouling is a persistent problem in the operation of membrane bioreactors. When signal molecule concentrations in the mixed liquor exceed a threshold, it initiates DNA transcription, forming biofilms (biofouling) on the membrane [1]. Quorum quenching (QQ) has been investigated to combat biofouling. QQ involves using QQ bacteria that enzymatically degrade signal molecules, either by directly injecting the bacteria into the reactor or by adding encapsulated biomedica [2]. More recently, immobilizing QQ bacteria onto the membrane surface has shown promise in delaying fouling [3]. However, this method initially impaired membrane permeability (50 L/m²-h-bar), highlighting the need for further improvements. Therefore, we aimed to fabricate a highly permeable and anti-fouling QQ membrane using various polymers. QQ activity was evaluated by measuring the degradation rate of a signal molecule (10 nM C8-HSL) over time. When the QQ membrane was coated with polymer A, the QQ activity was 0.37 h⁻¹. Polymer B increased activity 2.3 times to 0.85 h⁻¹. Furthermore, polymer C enhanced QQ activity by 4.2 times to 1.56 h⁻¹, achieving a substantially high water permeability of 281 L/m²-h-bar [3]. Polymer C appeared to retain the QQ bacteria more effectively than polymers A and B. These results demonstrate the effective immobilization of QQ bacteria on the membrane using polymers while improving membrane permeability.

¹Kyungpook National University, South Korea

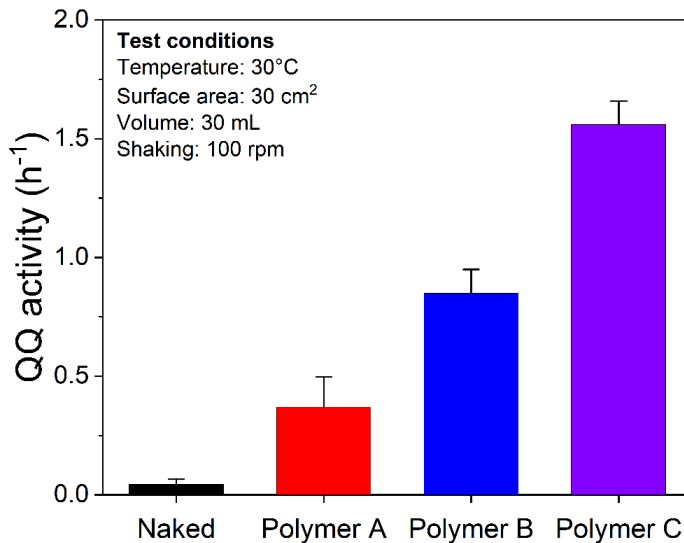


Figure 1: QQ activity of anti-biofouling membranes prepared with different polymers.

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Recycled Polycarbonate as Source Material for Membrane Preparation via NIPS

Daniel Breite¹ (daniel.breite@iom-leipzig.de), Marco Went, Tim Schreiber, Yasaman Zhale, Mathias Kühnert, Andrea Prager, Agnes Schulze

KEYWORDS: Membrane preparation, PC, NIPS, Recycling

Abstract

Membrane filtration is applied in many industrial fields like water purification, wastewater treatment, food and beverage industries, as well as chemical industries, and is an important separation technique. Due to the isothermal condition, membrane filtration is advantageous compared to other separation techniques. Especially polymer-based membranes have many fields of application. Unfortunately, the required material is often based on fossil resources, which is a point of concern. Reuse and recycling of membranes at the end of their lifetime as well as membranes based on recycled polymers are emerging fields of research. In this study, polymer membranes were prepared based on recycled polycarbonate (PC).

Recycled PC is purified by first dissolving the polymer flakes in N-methyl pyrrolidone (NMP) followed by precipitation in water or ethanol under rigorous stirring. The precipitated polymer is kept stirring and is subsequently dried overnight. Membranes are prepared from recycled polymers using poly(ethylene glycol) or poly(vinylpyrrolidone) as pore-forming agents and NMP as solvent in the non-solvent induced phase separation (NIPS) process. The concentrations of the components and preparation parameters are varied according to a design of experiment approach. After preparation, all membranes are washed and dried. Characterization is carried out using scanning electron microscopy (SEM), permeance measurements, water contact angle measurements, and tensile testing. Further investigations are carried out using Hg porosimetry, X-ray photoelectron spectroscopy, and zeta potential measurements for selected samples.

PC membranes could be prepared with a permeance in the range of 300 – 3500 L m⁻² h⁻¹ bar⁻¹ while the tensile strength varies in the range of 0.8 – 2.0 N mm⁻² (membranes without support and a thickness of 120 μm). The water contact angle results range from 60 to 80° depending on the preparation parameters. Different types of recycling-based PC

¹Leibniz-Institut für Oberflächenmodifizierung, Germany

are investigated to prepare membranes. It is possible to prepare membranes from all types of investigated PC sources, including e.g., PC from car parts or substitute glass sheets. While the latter has low amounts of impurities, the former was blended with another polymer. Nevertheless, membrane preparation is possible in both cases, resulting in highly permeable filters.

Nutrient Recovery Using Ceramic Membrane Contactors

Sarah Trepte¹ (sarah.trepte@ikts.fraunhofer.de), Christian Pflieger², Marcus Weyd², Hannes Richter², Yana Shopova¹, André Wufka¹, Burkhardt Faßauer¹, Marc Lincke¹

KEYWORDS: Nutrient recovery, nitrogen, membrane extraction, ceramic membrane, contactor, transmembrane chemisorption

Abstract

Motivation and Potential

Membrane extraction or transmembrane chemisorption using ceramic, temperature-stable, chemically resistant, and abrasion-proof membrane contactors is a promising key technology for recovering ammoniacal nitrogen (NH_4/NH_3) from aqueous residues. Nitrogen is a valuable material used in a variety of ways, and as a nutrient, it is a cornerstone of high-yield agriculture. In terms of saving resources and environmental protection, the recovery of nitrogen from residues represents a cost-effective and sustainable alternative to mineral fertilizers and offers the possibility of efficiently closing nutrient cycles. Additionally, there are further potential applications in the field of sustainable energy supply, among others.

Development and Production of Ceramic Membrane Contactors

In the BMBF-funded project abonocare®, Fraunhofer IKTS pursued the goal of developing and testing tubular ceramic membrane contactors for nutrient fractionation integrated into an agricultural fermentation process (FKZ 03WKD13C, final report available online). For this purpose, membrane samples based on single-channel and multi-channel tubular

¹Fraunhofer Institute for Ceramic Technologies and Systems IKTS, Winterbergstrasse 28, 01277 Dresden, Germany

²Fraunhofer Institute for Ceramic Technologies and Systems IKTS, Michael-Faraday-Strasse 1, 07629 Hermsdorf, Germany

membranes, up to capillary bundles made of aluminum oxide (Al_2O_3), were developed and manufactured.

By setting a partial pressure gradient, the gaseous ammonia in the aqueous residue desorbs and passes through the porous membrane. The ammonia is redissolved in a sulfuric acid solution, producing ammonium sulfate solution (ASL), a valuable liquid and approved fertilizer. A permanently water-repellent (hydrophobic) membrane surface is essential for the process to ensure a stable gas phase in the membrane pores. Inorganic oxidic membrane materials such as Al_2O_3 are intrinsically hydrophilic. Ceramic membranes were therefore hydrophobized, with silane proving to be the most promising hydrophobizing agent.

Performance for Nitrogen Recovery

In addition to basic material characterizations, the determination of the transfer coefficient was used for the process engineering evaluation. Clear dependencies on material coating, pore size, geometry, and process parameters were found. The most important factor for the process is ensuring a stable phase boundary with low diffusion resistance. Therefore, membrane coating with a fine porous layer on both sides as well as thin membrane wall thicknesses are favored. The best results were achieved with ceramic capillaries (transfer coefficient: 0.016 m/h). The contactors were tested for the extraction of ammoniacal nitrogen from aqueous residues provided by project partners. Ammonium sulfate solutions with up to 6% (by mass) were produced in the first approach.

Overall, the results are positive and confirm the further development of the process and the ceramic membrane contactors. The next steps include evaluating the most economical operating conditions, scaling up the technology, determining long-term performance, and assessing product quality. The next research project on this topic will start in autumn 2024.

Effect of crosslinking on the properties of polystyrene-co-vinylbenzyl chloride copolymer based anion exchange membrane for fuel cells

Züleyha Sarac¹ (zsarac@gtu.edu.tr), Çiğdem Taşdelen-Yücedağ¹

KEYWORDS: Cross-linked anion exchange membranes, fuel cells, mechanical properties, poly(vinylbenzyl chloride)

Introduction

Fuel cells are widely acknowledged as a renewable energy source for the 21st century. Anion exchange membrane (AEM) fuel cells have gained popularity due to their advantages over proton exchange membrane fuel cells, such as less fuel crossover and high efficiency [1]. In this work, an anion exchange membrane was designed using poly(styrene-co-vinylbenzyl chloride) copolymer (P(St-co-VBC)) as the polymer backbone and triethylamine (TEA) as quaternization agent. In order to increase the mechanical strength of membranes, crosslinking was employed by adding different amounts of diethylene glycol (DEG) into the reaction medium.

Experimental

Poly(St-co-VBC) copolymer was synthesized via free radical copolymerization of styrene (St) and vinylbenzyl chloride (VBC) in the presence of 2,2'-azobis(2-methylpropionitrile) (AIBN) initiator at 90°C for 24 hours. Copolymer structures were identified by ¹H-NMR and GPC. Then, the copolymers were turned into positively charged form through a quaternization reaction. Crosslinking was utilized to enhance the chemical stability and mechanical durability in alkaline media. Afterwards, membranes were manufactured from crosslinked positively charged polymer matrix.

Results and discussion

The chemical structure of synthesized poly(St-co-VBC) was identified by ¹H-NMR spectra. Fig. 1 depicts in detail all protons in the chemical structure of the produced copolymer, as well as their peaks and the molar ratio of the two monomers in the copolymer structure. As the crosslinker group concentration increased, the membranes' IEC values steadily dropped, from 4.03 to 1.73 mmol/g. The QM_highCross membrane exhibited the highest tensile strength value of 5 MPa, whereas

¹Gebze Technical University, Chemical Engineering, Kocaeli, Türkiye

the QM_zeroCross membrane's tensile strength value was measured at approximately 2 MPa (Fig. 1). These results suggest that the crosslinking reaction can effectively enhance the mechanical properties of the membranes. The produced AEMs were evaluated by structural analysis, thermal analysis, water uptake capacity, swelling ratio, ionic conductivity, and ion-exchange capacity measurements.

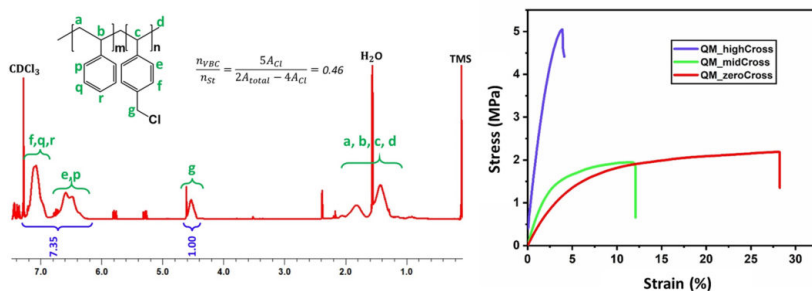


Figure 1: (a) ¹H-NMR spectra of the polymer, (b) stress-strain curve of membranes at different degrees of crosslinking.

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Effect of quaternization agent on the ionic conductivity of polysulfone/MXene nanocomposite anion exchange membranes

Selinnur Kıvanç¹, Çiğdem Taşdelen-Yücedağ¹
(cigdem@gtu.edu.tr)

KEYWORDS: nanocomposite anion exchange membrane, MXene, fuel cell, polysulfone

Introduction

Due to the escalating energy demand all over the world, fuel cells (FCs) are promising alternatives for clean and sustainable energy production in the future. Anion exchange membrane fuel cells (AEMFCs) predominate over proton exchange membrane fuel cells with their superior properties, such as no need for precious metal catalysts and faster electrochemical kinetics under alkaline conditions [1]. However, AEMs still require more research to enhance the mechanical properties and long-term stability in alkaline media. Preparing composite AEMs by the integration of two-dimensional (2D) nanoparticles into the membrane matrixes is a beneficial strategy that has gained interest in the past decade [2]. The motivation of this study was to develop MXene-containing polysulfone (PSU)-based membranes with high conductivity, high chemical and mechanical stability, and investigate their utilization as AEMs for FCs.

Experimental

For this purpose, polysulfone (PSU, Udel-P1700, mw: 65,000 – 75,000 Da, Solvay, Germany) was chloromethylated to introduce functional groups onto the polymer backbone [3]. The structure of the polymer was investigated via ¹H-NMR. MXene was synthesized from MAX phase (Nanografi, Türkiye) by etching aluminum through in-situ HF method [4]. The structure of MXene was identified by Raman Spectroscopy, XRD, XPS, and SEM-EDX analyses. Three different quaternization agents, triethylamine (TEA, Carlo Erba), 1-methyl imidazole (MIm, Aldrich), and 1,4-diazabicyclo[2.2.2]octane (DABCO, Aldrich), were employed for the quaternization of chloromethylated PSU (C-PSU) through active ends, hence three different AEMs were synthesized. For the preparation of nanocomposites, 3% w/w (of polymer) MXene was added into the C-PSU solution and sonicated for 15 min. Afterwards, the quaternization agent was added to the mixture and stirred at 60°C for 24 hours. All the membranes were prepared by the solution casting technique.

¹Gebze Technical University, Chemical Engineering, Kocaeli, Türkiye

Results and discussion

After the 24-hour chloromethylation reaction of the inert pristine PSU, the degree of substitution was calculated as 0.25 from $^1\text{H-NMR}$ using the integration areas of the signals of the $-\text{CH}_3$ and $-\text{CH}_2\text{Cl}$ protons [5]. The structure of MXene synthesized from MAX precursor was verified via Raman Spectroscopy, XRD, XPS, and SEM. The results showed that the incorporation of MXene into the membrane matrix led to an increase in ionic conductivities of all the samples prepared with different quaternization agents. The membrane samples of MIm and DABCO agents containing MXene demonstrated an increment in ionic conductivity with surface functional groups of MXene as compared to the samples having no MXene. The water uptake, swelling ratio, and ion exchange capacity of the membranes were also investigated.

Acknowledgments

The authors are grateful to Solvay Advanced Polymers, Germany for providing polysulfone (Udel-P1700).

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Formation and Characterization of Controlled Porous Membranes Using Block Copolymers

Didem Aydin¹ (didemaydin___@hotmail.com), Ilkay Hilal Gubbuk¹, Mustafa Ersoz¹

KEYWORDS: Block copolymer, Controlled pore size, Membrane

Abstract

Block copolymer (BCP) membranes with controlled pore structures are crucial for advanced separation technologies due to their tunable nanoscale features. In this study, we aimed to fabricate BCP membranes with precise control over pore size and distribution through systematic stages. Initially, phase-separated BCP films (PS-*b*-P2VP, PS-*b*-PEO) were prepared using spin-coating and solvent vapor annealing techniques. Following this, metal oxides were introduced to functionalize the BCP templates. Etching methods, including plasma and UV-O treatment, were employed to remove BCP domains, resulting in nanoporous structures selectively. These membranes were thoroughly characterized using SEM, AFM, and XRD to analyze their morphology and functionality. This study demonstrates the importance of controlled pore formation in BCP membranes, showcasing their potential for applications in filtration and separation.

Introduction

Block copolymers (BCPs) are increasingly used for creating advanced nanoporous materials due to their ability to undergo microphase separation, resulting in well-defined and tunable nanostructures. The distinct blocks in BCPs, such as polystyrene (PS), poly(2-vinylpyridine) (P2VP), or poly(ethylene oxide) (PEO), exhibit different affinities for solvents and other materials, enabling the formation of periodic domains like lamellae, gyroids, or cylindrical structures. (1) This natural self-assembly process allows for the precise control of pore size, shape, and distribution, which is critical for applications in filtration, catalysis, and sensors. Additionally, the ability to fine-tune BCP compositions and phase behavior makes them ideal candidates for high-performance membrane development.

Experimental Section

BCP solutions (1.5% w/w in 1:4 THF/toluene) were prepared by stirring for 24 hours. Silicon wafers and ITO substrates were cleaned using

¹Selçuk University, Türkiye

ultrasonic baths with solvents like acetone and ethanol. BCP films were spin-coated at various speeds and thermally annealed using chloroform vapor to induce microphase separation. Metal nitrate solutions in 1-butanol were deposited on the BCP films, followed by UV-O exposure for 3 hours to remove the polymer and create a metal oxide mask. Additional heat treatment at 400°C was applied when necessary to ensure complete polymer removal. SEM and AFM were used to analyze the phase separation and pore formation [2].

Results and Discussion

The BCP films displayed effective microphase separation and pore formation, as observed through SEM and AFM imaging. Solvent choice, particularly chloroform, was critical in achieving proper phase separation. UV-O exposure successfully removed the polymer, allowing the metal oxide mask to form on the surface. AFM images showed the development of periodic nanopores after UV-O treatment, confirming the success of the etching process.

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Transport of methylene blue dye into graphene-based polymeric membrane

Gülcihan Ozdem¹, Ilkay Hilal Gubbuk¹ (ihilal@gmail.com), Ozlem Altintas Yildirim¹, Hakan Erer¹, Mustafa Ersoz¹

KEYWORDS: Metal-Organic Frameworks, ZIF-8, MB, dye removal, transport mechanism

Abstract

More than 700,000 tons of paint are produced worldwide each year, and it is well known how serious and global health and environmental problems are caused by the release of unrefined organic paints and their by-products into nature. According to the World Health Organization (WHO), environmental pollutants found in soil, air, and water are responsible for 25% of diseases that impair human health.

This study aims to develop nanoscale applications for forming membranes from porous materials, integrating material science and membrane technology. The study focuses on water purification processes, specifically targeting methylene blue (MB), a toxic and carcinogenic component, through the use of PAN nanofiber membranes.

Introduction

Methylene blue (MB) is an organic dye with a cationic (basic) structure and is widely used in the textile, printing, leather, paper, plastic, food, cosmetics and pharmaceutical [1]. MB, a cationic dye, is widely used to dye cotton, wool and silk. It is also used in the treatment of some diseases because it is an antiseptic. MB accumulates despite being highly soluble in water. Its persistent aromatic structure is the most important reason for this problem because it is not biodegradable and remains in water for a long time. Therefore, before wastewater is discharged, it must be subjected to environmental remediation processes that will reduce the rate of pollutants in water. Among the persistent pollutants in water such as heavy metals, polycyclic aromatic hydrocarbons, herbicides and organic dyes, dyes are the most dangerous type for water resources because they are toxic, mutagenic and carcinogenic [2]. Colored wastewater containing dyes and their by-products negatively affects aquatic plants, photosynthetic bacteria and the ecology of water.

¹Selçuk University, Türkiye

Experimental Section

The transport process of MB was carried out using a specially designed laboratory device consisting of two Teflon chambers. The nanofiber membrane was placed between these chambers with the help of a gasket in a leak-proof manner. The volume of each cell (feed and receiver phases) was 40 ml. In the experiment, an aqueous solution of MB was used as the feed phase, placed on the left side of the permeation cell, while deionized water was used as the receiver phase on the right side. Both phases were stirred using a magnetic stirrer to prevent boundary layer formation.

Results and Discussion

MB removal studies were conducted by mixing a 5 ppm dye solution with 2 x 8 cm nanofiber membranes in a beaker at 100 rpm at room temperature. The absorbance of samples taken at the start and end of 30-minute intervals was measured at 664 nm using a UV spectrophotometer, and concentrations were calculated using a previously created calibration curve.

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Posters

Electrodialysis

Circular Chemical Use: Producing Acid and Base with Bipolar Electrodialysis from IEX Regenerate

Nienke Koeman¹, Timon Rijnaarts¹
(timon.rijnaarts@kwrwater.nl), Bas Pol¹

KEYWORDS: Bipolar Electrodialysis, Brine treatment, Circular process

Abstract

Ultrapure water is produced using ion exchange (IEX) columns. These columns need to be regenerated using acid and base and produce a salt effluent stream. When this effluent is treated by electrodialysis with bipolar membranes (EDBM), the salts are separated and transferred back into their associated acid and base components. These can then be used again to regenerate the IEX columns. This enables the circular use of chemicals and saves water as well. Moreover, it will reduce logistics and storage of concentrated chemicals.

In this research, we have compared two types of membranes (from different manufacturers) for their performance. The electrical efficiency of Weifang membranes was 77% at 200 A/m² for base production compared to 65% for Mega membranes using a NaCl brine. Also, the effect of other ions present in the IEX regenerate on the electrical efficiency was investigated using a synthetic stream.

Experimental results were used to evaluate product (acid and base) quality, recovery efficiency, and energy efficiency of the EDBM process. In addition to studying pure NaCl brine streams, a stream from a chemical plant (with ions other than NaCl) was evaluated as well.

The results of this research enable the circular use of chemicals, leading to a smaller water and chemical footprint in the industry.

¹KWR Water Research Institute, Groningenhaven 7 3433 PE Nieuwegein, The Netherlands

An Ultra-High Pressure Osmotic Membrane Module and Apparatus for Osmotic Energy and Desalination of Hypersaline Brine: Design, Construction, and Evaluation

Haofei Guo¹ (hgo@saltpower.net), Jan Eric Thorsen, Jørgen Mads Clausen, Lars Storm Pedersen

KEYWORDS: Osmotic energy, Ultra-high pressure apparatus, Hypersaline, Pressure Retarded Osmosis (PRO), Membrane cassette

Introduction

An ultra-high pressure apparatus including a membrane module was designed and constructed for both osmotic energy production based on the Pressure Retarded Osmosis (PRO) process and energy-efficient desalination based on the Reverse Osmosis (RO) process, where hypersaline brine is used. The test rig can operate at pressures up to 200 bar. The pressure-resistant membrane cassette comprises 3 layers and 4 ports. It can be applied for both PRO and RO by controlling the inlet valve. To our knowledge, this is the first 200 bar multi-function osmotic membrane facility worldwide. The article focuses on the function of the apparatus and evaluation of the membrane cassette concept. Further, the pressure resistance property and performance of polymeric membranes at pressures up to 200 bar were investigated.

Materials & Methods

Pharmaceutical grade NaCl was used as the salt solute (Dansk Salt A/S), with a NaCl concentration of 310 g/L. Water was from RO permeate with a conductivity of 10 $\mu\text{S}/\text{cm}$. The commercial polyamide RO membrane and a lab version non-backing polymeric membrane were tested. The open loop osmotic membrane system is shown in Figure 1. The high-pressure pump was applied on the brine side, with the active membrane surface facing towards the high-pressure side in the membrane module. In the RO mode, the low-pressure pump was closed, and water was supplied to the high-pressure side by opening an auto valve. In this case, the draw (out) tank was used for feed rejection, while the feed (out) tank was used for permeate. In PRO mode, saturated brine was used.

¹SaltPower ApS, Denmark

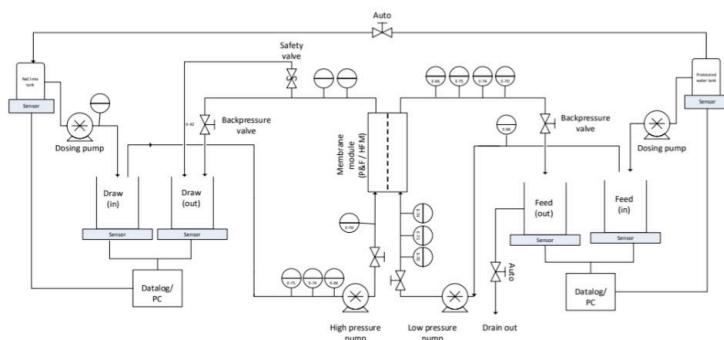


Figure 1: Schematic diagram of open loop osmotic membrane system for RO/FO/PRO up to 200 bar.

Results & Discussion

The pressure was controlled stably at 200 bar with constant and limited deviation in an osmotic membrane process, as shown in Figure 2. The cassette function was indicated by RO mode testing, where RO water was channeled to the active surface of the membrane at 150 bar and further collected in rejection and permeate tanks respectively. The mean weight gradient per minute into the feed rejection tank was calculated as 0.530 kg from the difference between feed inlet and permeate, while the experimental record was 0.526 kg, as shown in Figure 3. The difference between the record and calculation was less than 1%, indicating that the cassette functioned properly as the flow could entirely distribute along the permeate channel. All above results were based on a composite membrane, and it was verified that the standard RO membrane withstands 200 bar and still produces permeate at high pressure. However, for the non-backing membrane, it was damaged when pressure was raised to 100 bar without a spacer underneath the membrane, as shown in Figure 4.

Acknowledgements

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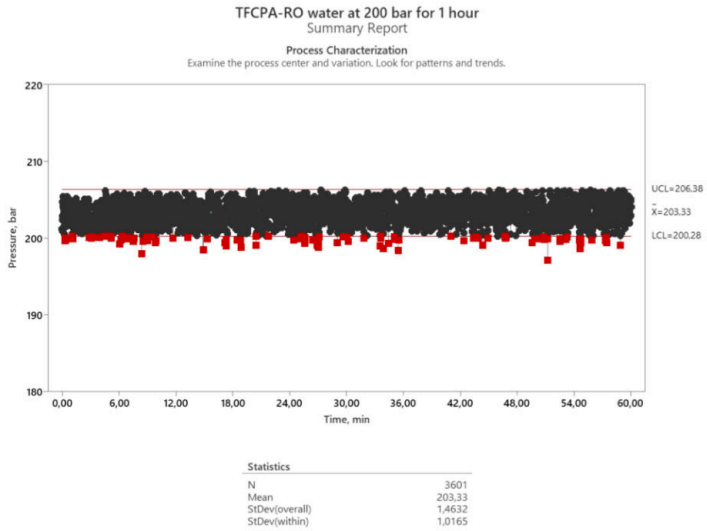


Figure 2: Statistical analysis of pressure stability in RO mode at 200 bar for 1 hour.

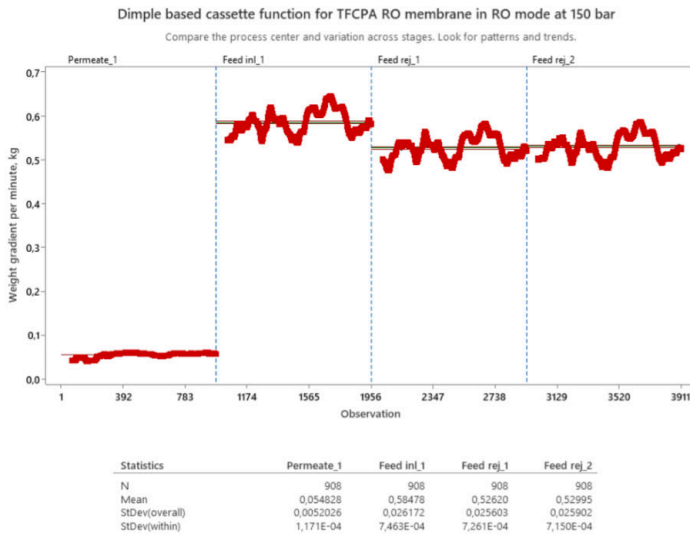


Figure 3: Mass balance analysis in RO mode using TFCPA RO membrane at 150 bar.

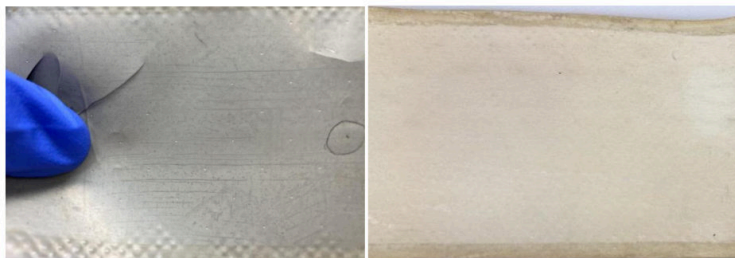


Figure 4: Membrane appearance after applying pressure from top up to 200 bar. Left: non-backing membrane, max. 100 bar; Right: TFCPA RO membrane, 200 bar.

Testing of Electrodialysis with Bipolar Membrane at Semi-Industrial Scale for In-Situ Reactant Production

A. Filingeri¹ (antonia.filingeri@unipa.it), A. Culcasi¹, C. Casaro¹, M. Nanfara¹, A. Tamburini^{1,2}, G. Micale¹, A. Cipollina¹

KEYWORDS: Brine valorization, chemical generation, electro-membrane process

Introduction

Electrodialysis with bipolar membranes (EDBM) has been gaining attention for the production of chemicals, such as acids and bases, for direct use in situ within circular and integrated processes, thus avoiding transport and handling of highly concentrated and dangerous reagents. Although this process has been explored in recent years at the laboratory scale, few studies have aimed at the development of pilot-scale units. This work aims to fill this gap by scaling up and testing an EDBM unit at a semi-industrial scale.

Materials and Methods

The pilot unit consists of 50 triplets with a total active membrane area of 0.32 m². The setup includes measurement instruments to monitor flow rates, conductivity, pressure, etc. Tests were performed in batch mode with synthetic solutions at an initial concentration of 1.0 mol/L NaCl in the saline channels and 0.05 mol/L HCl and NaOH in the acid and base channels, respectively. The effect of different initial volumes in the compartments was investigated by comparing performance indicators such as current efficiency (CE), specific energy consumption (SEC), and specific productivity (SP).

Results and Discussion

Results showed that, when doubling the volume of acid, a SEC of 1.05 kWh/kg NaOH and CE of 95% were reached at 0.5 mol/L of OH⁻. Moreover, OH⁻ concentration above 1.0 mol/L could also be reached (with corresponding SEC and CE of 1.05 kWh/kg NaOH and 76%, respectively). Lowering the acid concentration (by increasing the acid tank

¹Dipartimento di Ingegneria, Università di Palermo, Viale delle Scienze Ed. 6, 90128 Palermo, Italy

²ResourSEAs SrL, Palermo 90128, Italy

volume) reduced the undesired effect of diffusion towards the saline channel and partial neutralization of acid and base. This work demonstrated the scalability of the EDBM process and highlighted some practical operating conditions to optimize the EDBM performance indicators.

Acknowledgments

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Determination of selectivity coefficients of ion exchange membranes at various process conditions

Karin Lovrantová¹, Jan Tomek¹, Natália Václavíková¹
(natalia.vaclavikova@membrain.cz)

KEYWORDS: Ion selectivity, selectivity coefficient, ion exchange membrane, electrodialysis, desalination

Introduction

To enhance the performance of electrodialysis, it is essential to explore new approaches and techniques aimed at better describing membrane properties and predicting the desalination process. Selectivity is a valuable tool for examining the transport of ions through ion exchange membranes, which allows forecasting the rate of reduction in ion concentration during desalination. Current research focuses on methods for determining the selectivity coefficient between monovalent and divalent inorganic ions using both homogeneous and heterogeneous membranes. The main goals were to validate the applicability of the suggested method and to assess the selectivity coefficients of different membranes.

Materials & Methods

A series of desalination batch experiments was conducted using a laboratory electrodialysis module with ten cell pairs. Feed solutions con-

¹MemBrain s.r.o., Czech Republic

tained various monovalent and divalent ions. A constant voltage of 1V/cp was maintained across the membrane stack, and to examine the validity range of the selectivity coefficients, different experimental conditions were tested, such as temperature, voltage, ion concentrations, or the presence of organic non-dissociating compounds. Samples of the feed were collected during each batch and analyzed to determine the concentrations of ions.

Results & Discussion

The evaluation revealed that the suggested method can be used for both homogeneous and heterogeneous ion exchange membranes under a broad spectrum of operating conditions. It was found that heterogeneous membranes Ralex[®] exhibit only insignificant selectivity between mono- and divalent ions, and in the case of CEM, they are even more selective for divalent ions. The monovalent selectivity of Selemion ASVM membranes was confirmed. However, it was shown that membranes fumasep[®] FAB-PK-130 with hydroxyl-blocking properties have a much higher selectivity coefficient ($\text{Cl}^- / \text{SO}_4^{2-}$).

Acknowledgements

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The use of membrane processes for lithium carbonate production – a comparative study between electrodialysis and membrane crystallisation

Veronika Kúdelová¹ (veronika.kudelova@membrain.cz), Jakub Fehér¹

KEYWORDS: Lithium, lithium carbonate, electrodialysis metathesis, membrane crystallisation, ion-exchange membranes, scale-up

Introduction

In recent years, the demand for lithium has dramatically increased, largely driven by the growth of electromobility and the corresponding

¹MemBrain s.r.o., Czech Republic

need for lithium batteries. The global assumption is that the demand for lithium batteries in 2030 will be more than 11 times higher than in 2022. With limited access to primary lithium sources and lithium listed in the critical metal list in 2020, lithium's recovery from secondary sources becomes more important. Several technologies have been investigated for this purpose, such as ion exchange, electro dialysis, and membrane crystallisation, each with challenges that necessitate optimisation. Ongoing research and development are vital for enhancing electro dialysis and other membrane processes and integrating different technologies to improve lithium recovery initiatives.

Materials & Methods

In this work, the recovery of lithium carbonate was studied using two technologies. The first technology used was electro dialysis metathesis with a module made of polypropylene ion-exchange membranes followed by crystallisation. The second technology applied was membrane crystallisation using a module with anion-exchange hollow fibres. Final crystal purities and process parameters were studied and compared.

Results & Discussion

Both technologies are promising for lithium carbonate recovery. Each technology has advantages – the advantage of electro dialysis lies in the intermediate product's high purity and applicability to different solution compositions. The advantage of membrane crystallisation is the low pressure and energy consumption required for operation. However, there are disadvantages in both technologies – high energy consumption during electro dialysis and the required higher purity of the raw material during membrane crystallisation. Both technologies require further development, and the technology choice depends on the feed solution's quality.

Acknowledgements

The work was carried out within the framework of the project “A long-term concept for the development of the research organization” supported by the Ministry of Industry and Trade of the Czech Republic, using the Membrane Innovation Centre infrastructure.

Posters

Energy applications

Thin-Film Nafion-Polyethylene Composite Separator Membranes for Zinc Polyiodide Flow Battery

Anupam Das¹ (anupam.das@uni-due.de), Lukas Siefert², Falko Mahlendorf², Mathias Ulbricht¹

KEYWORDS: Zinc polyiodide flow battery, Composite membranes, Nafion, Polyethylene, Energy storage

Abstract

The Zinc polyiodide flow battery (ZIFB) is one of the most promising technologies for grid-scale energy storage due to its attractive features of high energy density, high safety, and being environmentally friendly. A dedicated design of a separator membrane with low resistance (high conductivity) and minimized anion permeation (high ion selectivity) is necessary for high efficiency and cycling stability of ZIFB.

For this work, we have chosen different types of porous polyolefin (PE) membranes (thickness down to 11 μm) as support and coated them with thin Nafion layers (thickness down to 1 μm) to obtain Nafion-PE composite membranes. Scanning electron microscopy (SEM) analysis confirmed homogeneous coating of Nafion on the PE surface and was also used to determine Nafion film thickness. For one prototype, pure water permeance of the Nafion-PE composite [33 L/m².h.bar] was much lower compared to that of the porous PE support [317 L/m².h.bar]. Contact angle (CA) of the Nafion-PE composite decreased compared to PE due to the amphiphilic Nafion coating on the hydrophobic PE surface.

Area-specific resistance (ASR) of the Nafion-PE composite membrane obtained from impedance spectroscopy increased compared to porous PE. However, the ASR of the Nafion-PE composites was observed to decrease with decreasing Nafion layer thickness, and it was significantly lower than that of conventional thick Nafion membranes. Compared to free diffusion, the ratio of effective diffusion coefficients for Zn²⁺ versus I⁻ was approximately 4-fold increased, confirming the ion selectivity of the Nafion film in the composite membranes. Further optimization of the fabrication and analyses are underway with the aim to fully unlock the potential of the newly developed ion-conducting thin-film Nafion-PE composite membranes as separators for ZIFB.

¹Lehrstuhl für Technische Chemie II, Universität Duisburg-Essen, Essen, Germany

²Lehrstuhl Energietechnik, Universität Duisburg-Essen, Duisburg, Germany

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The Roles of the Membrane in Alkaline Water Electrolysis in Interplay with Water Components

Ankitkumar Kansara¹ (ankitkumar.kansara@uni-due.de), Ignacio Sanjuán², Anam Asghar³, Corina Andronescu², Mathias Ulbricht¹

KEYWORDS: Water electrolysis, Anion exchange membranes, Membrane degradation

Abstract

The utilization of advanced anion exchange membranes (AEMs) has revolutionized the field of water electrolysis and fuel cells. This study investigates the roles of commercially available AEMs such as Fumasep Aemion and Sustainion in the process of converting water to green hydrogen via electrolysis under alkaline conditions, focusing on their interaction with various extrinsic (from the feed) and intrinsic (produced in the electrolyser) water components and the consequences for efficiency and stability of hydrogen production. The work comprises the detailed analysis of AEMs to comprehend degradation mechanisms including hydrolysis, substitution, Hofmann elimination, and radical reactions. Key properties such as ion exchange capacity, membrane conductivity, water uptake, membrane swelling, and gas permeability are evaluated. Atomic force microscopy, scanning electron microscopy, and small-angle X-ray scattering are employed to study membrane morphology; chemical structure is assessed using ATR-IR, Raman, and X-ray photoelectron spectroscopy. Thermal and mechanical properties are determined through thermogravimetric analysis coupled with mass spectrometry, differential scanning calorimetry, and tensile strength measurements. Furthermore, the study investigates the release of organic degradation products into water via liquid chromatography coupled with high-resolution mass spectrometry and total organic carbon analysis. Ex situ (membranes just

¹Lehrstuhl für Technische Chemie II, Universität Duisburg-Essen, Essen, Germany

²Technische Chemie III, Universität Duisburg-Essen, Duisburg, Germany

³Instrumentelle Analytische Chemie, Universität Duisburg-Essen, Essen, Germany

under alkaline conditions at high temperature) and in situ (membrane as separator during electrolysis) experiments are conducted over a reasonable timeframe to observe relevant degradation effects. The research aims to provide insights into membrane degradation mechanisms and their impact on water quality and system performance in AEM-enabled alkaline water electrolysis.

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The Ultimate Project: A Case Study on Resource Valorization with Electro-Chemical Membrane Processes from Greenhouse Wastewater

Tavishi Guleria¹ (tavishi.guleria@kwrwater.nl), Joep van den Broeke, Ides Platteau, Timon Rijnaarts, Abdulsalam Alhadidi, Leonardo Gutierrez, Emile Cornelissen

KEYWORDS: Capacitive Electrodialysis, Irrigation Water Reuse, Greenhouse Wastewater, Pilot Scale

Abstract

As food production dominates global freshwater consumption and nutrient discharge regulations tighten specifically for the greenhouse horticulture sector, the performance of novel technologies at an applied scale needs to be studied to optimize water use and minimize environmental impact. Water reuse and nutrient recovery from greenhouse wastewater were assessed using one-pass capacitive electrodialysis (CED) at a pilot

¹KWR Water, The Netherlands

scale (19.32 m² membrane area, 1-4 m³/day capacity) employing carbon-based electrodes. CED successfully achieved the target irrigation water quality for reuse and good nutrient concentration for recovery with significantly lower specific energy consumption (SEC) than reverse osmosis (RO) and similar electrodialysis (ED) studies, suggesting it is a promising technology for the greenhouse horticulture sector aiding the move toward zero liquid discharge.

Materials & Methods

Two synthetic wastewaters (mimicking individual and collective greenhouse wastewater) using KH₂PO₄, NaHCO₃, (NH₄)HCO₃, NaNO₃, MgSO₄, KNO₃, and CaCl₂ at a final conductivity of 1.7 mS/cm, and real feed from a collective greenhouse consortium in the Netherlands (2.5 mS/cm conductivity) were tested. These streams were tested on a CED pilot setup. The CED stack consisted of 4 hydraulic stages, 150 membrane pairs (FujiFilm type 10), active area of electrodes: 400 cm², and was operated at cross-flow velocities of 5.12 cm/s and 7.72 cm/s in one-pass mode. The voltage, current, pressure drop, conductivity, flow rate, and pH of the feed, concentrate, and diluate were logged every 5 seconds. Specific energy consumption (kWh/m³), current efficiency of nutrients compared to strong electrolytes (Na and Cl), water recovery (%), concentration factor, and losses (%) were calculated. The target was to meet Dutch Greenhouse Water Quality guidelines [1].

Results & Discussion

Diluate water quality and nutrient fate

All feed compositions achieved the highest-quality irrigation water for greenhouses (conductivity <0.2 mS/cm, Na⁺ and Cl⁻ <0.1 mmol/l) (Figure 1). Interestingly, monovalent ions, particularly Na⁺ and K⁺, remained at relatively higher concentrations in the diluate compared to other ions even at low conductivity levels (<0.2 mS/cm). The removal efficiency for ions followed the order: Ca²⁺ > Mg²⁺ > K⁺ > Na⁺ for cations, and PO₄³⁻ > SO₄²⁻ > NO₃⁻ > Cl⁻ > HCO₃⁻ for anions. This selective removal was more pronounced at low current densities and shifted toward enhanced monovalent ion transport at higher voltages. In addition to desalinating water for reuse, the CED system concentrated ions for nutrient recovery from greenhouse wastewater. Concentration factors for ions showed a preference for divalent over monovalent ions, aligning with diluate quality findings. The CED concentrate displayed potential for recovering multiple ions, notably K⁺ (2.4, 2.3), Ca²⁺ (4.6, 4.1), Mg²⁺ (3.8, 3.6), NO₃⁻ (2.7, 2.1), HCO₃⁻ (2.5, 2.3), and SO₄²⁻ (1, 1.9) at 90% water recoveries for collective and individual greenhouse wastewater.

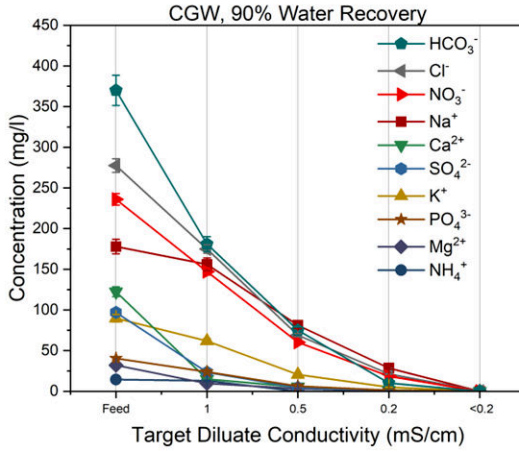


Figure 1: Cation and anion removal from collective greenhouse WW (CGW) at 90% water recoveries

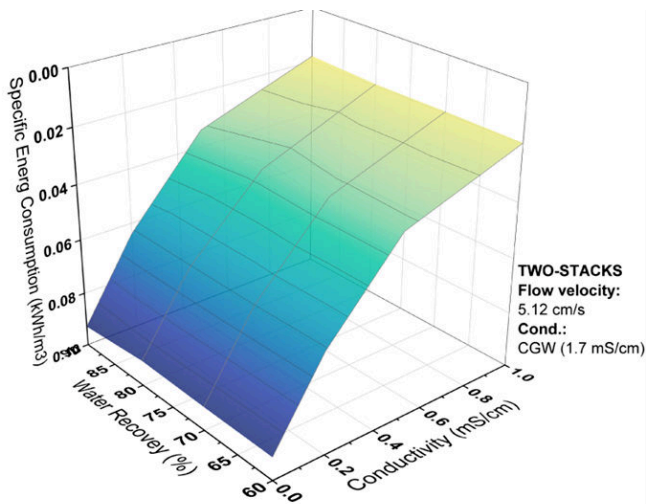


Figure 2: Specific energy consumption (SEC) in kWh/m³ for desalinating greenhouse wastewater (CGW feed composition). The color gradient from light green to dark blue represents an increase in SEC corresponding to target conductivities (1 to <math><0.2</math> mS/cm), achieved over water recoveries of 60–90%

Capacitive electro dialysis (CED) energy consumption

For achieving the highest target irrigation water quality (<0.2 mS/cm conductivity), specific energy consumption (SEC) was estimated at 0.02-0.14 kWh/m³ for the CED stack for all feeds tested at 90% water recovery (Figure 2). Pumping SEC varied between 0.043 and 0.120 kWh/m³ (pressure drops of 0.35-0.7 bar and 80% efficiency). The total energy consumption, excluding treatment losses, was estimated at 0.14-0.24 kWh/m³. This study demonstrated lower SEC for greenhouse wastewater treatment compared to reported values for ED (minimum 0.29 kWh/m³) and RO (minimum 0.36 kWh/m³), highlighting CED's energy efficiency and economic promise for producing fit-for-use irrigation water and recovering nutrients from greenhouse wastewater at an applied scale.

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Posters

Health

Novel Membrane Oxygenator with Combined Kidney Support – From Membrane Bundle Parameters to Device Development

Ana Martins Costa¹ (a.f.martinscosta@utwente.nl), Jan-Niklas Thiel², Frank Halfwerk^{1,3}, Bettina Wiegmann^{4,5,6}, Michael Neidlin², Jutta Arens¹

KEYWORDS: Extracorporeal Membrane Oxygenation, Lung Support, Kidney Support, In-vitro Test, Hollow Fibers

Introduction

A new artificial lung and kidney support device (RenOx) is being developed combining gas exchange and dialysis hollow fibers in a single bundle. This device aims to provide sufficient gas exchange with the blood, while simultaneously, blood toxins and excess fluid are removed. The RenOx combined bundle is required to 1) provide lung support, 2) deliver kidney support, and 3) allow for a compact device size comparable to state-of-the-art oxygenators. Therefore, for the RenOx device development, first, parameters for a novel integrated membrane need to be derived. Second, user specifications and optimal blood flow distribution need to be considered for device design.

Objectives

First, our work derived parameters for the development of a compact and integrated membrane bundle able to provide sufficient pulmonary and renal support. For that, we analyzed in which proportion and arrangement gas exchange fiber layers and dialysis fiber layers could be combined maintaining high gas transfer efficiency. After, we evaluated the effect of utilizing dialysis fibers in a novel approach outside-in (blood flow outside the fibers) on their capacity to clear toxins and filtrate blood. Second, steps on the development of the RenOx comprising design and optimization of its housing and blood inlet and outlet geometry are described.

¹Engineering Organ Support Technologies, University of Twente, Netherlands

²Department of Cardiovascular Engineering, Institute of Applied Medical Engineering, RWTH Aachen University, Germany

³Thorax Centrum Twente, Medisch Spectrum Twente, Netherlands

⁴Hannover Medical School, Germany

⁵Implant Research and Development (NIFE), Germany

⁶German Center for Lung Research, Germany

Methods

First, we evaluated the effect of replacing part of gas exchange fibers by dialysis fibers. For this, membrane oxygenators with fiber layers arranged at 90° and/or 24° angle, with a different number of gas exchange fiber open (100%, 75%, 67%, 50%) were tested for gas transfer capacity following the ISO 7199:2017. In a follow-up study, the performance of commercial dialyzers applied outside-in and in traditional inside-out mode was compared during in-vitro blood tests adapting the ISO 8637:2016. Second, we conducted interviews with users (e.g., intensivists, ECMO specialists) to derive design requirements. RenOx design concepts were derived considering requirements and bundle parameters. The blood flow path of the RenOx was designed considering existing patented blood geometries, and optimized for flow distribution by computational fluid dynamics (CFD) simulations.

Results

Our first study showed that 25% of gas exchange fibers could be replaced keeping 90-100% of the gas exchange efficiency of our membrane oxygenator [1]. This could result from the contribution of blood mixing to oxygen exchange. Our second study demonstrated that hemodialyzer's fibers utilized outside-in achieved similar levels of urea and creatinine clearance as conventional inside-out dialysis fibers. However, filtration capacity was reduced, and resulting ultrafiltration coefficient for outside-in mode (11.7 mL/h/mmHg) was 4 times lower than for inside-out [2]. Regarding RenOx design, a device with surface area of 2 m² is required to support 80 kg patients (75% gas exchange fibers = 1.5 m², and 25% of dialysis fibers = 0.5 m²). In this sense, we selected a hexagonal fiber bundle arrangement, since this would allow us to replace 1 in every 4 gas exchange fiber layers by a dialysis fiber layer, Figure 1. A novel flow path was designed and CFD simulations showed that a cap with guiding angle of 30° or 10° resulted in low relative stagnation zone 0.002%, and low pressure drop in the cap of 3 mmHg. The design of RenOx housing accounted for sealing of compartments for gas, blood, and dialysate and the possibility for easier membrane bundle removal for posterior analysis.

Conclusions

A novel membrane oxygenator with combined kidney support function (RenOx) is under development. Our results indicate that an integrated hollow fiber bundle composed of 75% gas exchange fibers and 25% of dialysis fibers utilized outside-in could provide required lung support and blood clearance. RenOx prototypes with optimized blood flow distribution are being manufactured for future in-vitro and in-vivo tests.

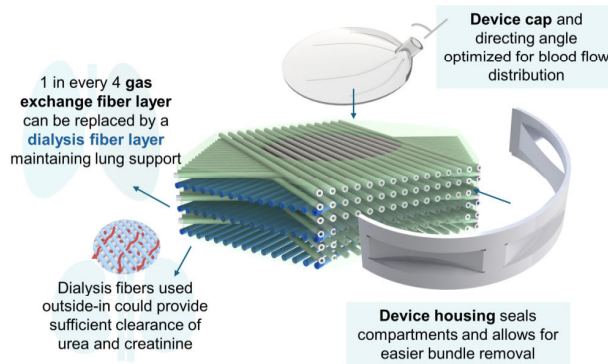


Figure 1: Development of a new artificial lung and kidney assist device with a fiber bundle that integrates gas exchange and dialysis hollow fibers.

Acknowledgement

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Mechanical Stability of Microporous Membranes for Liquid Atomization and Dosing

Rene Pfister¹ (rene.pfister@ost.ch), Yves Mermoud¹, Chantal Stolz¹, Claudio Wolfer², Cord Henrik Surberg¹, Samuel Huber¹

KEYWORDS: Microporous membranes, mechanical testing, membrane design, membrane stability

Introduction

Microdroplets and aerosols are essential in a wide range of applications such as spray drying, microprinting, or healthcare. Their generation can be achieved with nozzles, i.e., thin membranes with well-defined orifices in combination with either pressure or membrane actuation [1]. Therefore, mechanical properties are essential for design, operation, and reliability of membranes. This study aims at investigating a novel method for determining the micromechanical properties of thin, microporous membranes with different porosities. A nanoindentation instrument is used to determine the membrane's intrinsic stress at room temperature, elastic modulus, and the maximum load before rupture.

Materials & Methods

SiNX microporous membranes with square shapes of 184 μm and 170 μm side length with porosities $k = 0.1\%$ and $k = 13.5\%$, respectively, were fabricated at wafer scale by standard dry etching microfabrication techniques. A low-stress 300-nm-thin SiNX layer deposited by Low Pressure Chemical Vapor Deposition (LPCVD) on 4-inch Si wafers is used as a substrate material in which nozzles of various sizes and arrangements are lithographically defined (Figure 1a). A nanoindentation instrument (FemtoTools model FT-I04) with a spherical glass tip of diameter $D = 50 \mu\text{m}$ is used to obtain force-deflection readings [2] (Figure 1b-c). The relationship between applied controlled membrane deflection w and the corresponding force F is expressed as the sum of three main contributions, i.e., bending, stretching, and pre-stress related [3][4]:

$$F = A \cdot \frac{Eh^3}{r^2}w + B \cdot \frac{Eh}{r^2}w^3 + C\sigma_0w,$$

¹Institute for Microtechnology and Photonics, OST – Eastern Switzerland University of Applied Sciences, Buchs (SG), Switzerland

²Institute for Computational Engineering, OST – Eastern Switzerland University of Applied Sciences, Buchs (SG), Switzerland

where E , σ_0 , h , r denote Young's modulus, pre-stress, membrane thickness, and size, respectively. A , B , C incorporate Poisson's ratio and adapt for the non-circular shape. Therefore, the method allows extraction of the membrane layer's Young's modulus and pre-stress as well as their virtual change as a function of the porosity. Moreover, when further increasing deflection, membranes can be loaded until their point of rupture, leading to measurements of maximum force F_{\max} and maximum deflection w_{\max} .

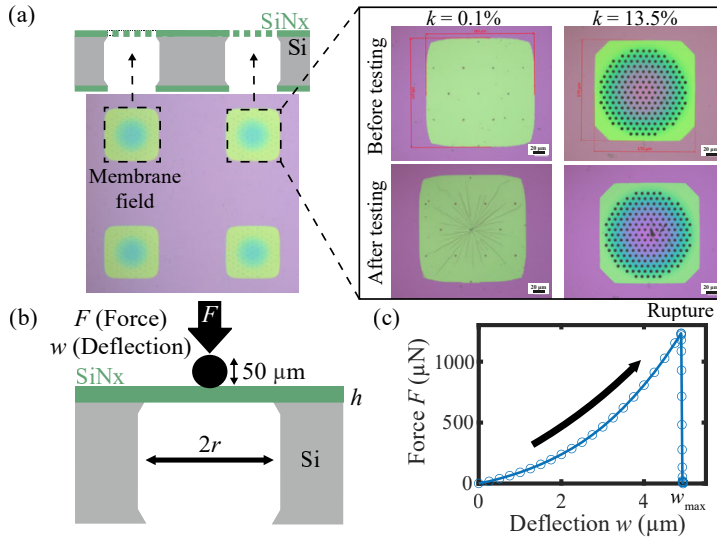


Figure 1: (a) Schematic cross-section and micrograph of four membrane fields on a fabricated membrane wafer, along with a micrograph (before and after testing) of characterised 300-nm-thin membranes with porosities of 0.1% and 13.5% and sizes of $184\ \mu\text{m}$ and $170\ \mu\text{m}$, respectively. (b) Schematic cross-section of the characterisation method applying controlled deflection and measuring force based on a FemtoTools nanoindentation instrument with a spherical glass tip with a diameter of $50\ \mu\text{m}$. (c) Example of a force-deflection graph obtained from the membrane characterisation. Membrane deflection leads to a force increase until membrane rupture occurs at w_{\max} , F_{\max} .

Results & Discussion

Force-deflection measurements were performed with 33 membranes M#A with $k = 0.1\%$ and 21 membranes M#B with $k = 13.5\%$ in the range of up to $12\ \mu\text{m}$ deflection and $12\ \text{mN}$ force. The responses of the devices were found to be largely identical for the loads before rupture. In

P 5

contrast, the point of rupture (F_{\max}, w_{\max}) was found to show much larger spread, as shown for $k = 13.5\%$ in Figure 2. Following the model (1), the Young's modulus and pre-stress values for the two types of membranes were extracted from the measurements. M#A revealed $E_A = 270$ GPa and $\sigma_{0,A} = 136$ MPa, whereas for M#B $E_B = 140$ GPa and $\sigma_{0,B} = 80$ MPa were extracted, with both types of membranes realized with the same LPCVD SiN layer. In comparison, the result obtained with 0.1% porosity is in line with literature value $E = 290$ GPa and confirms increasing membrane flexibility with increasing porosity [5]. Subsequently, the points of rupture were analyzed, revealing median values of $F_{\max,m,A} = 8.3$ mN, $w_{\max,m,A} = 9.1$ μm and $F_{\max,m,B} = 4.4$ mN, $w_{\max,m,B} = 8.7$ μm for the two types of membranes, respectively.

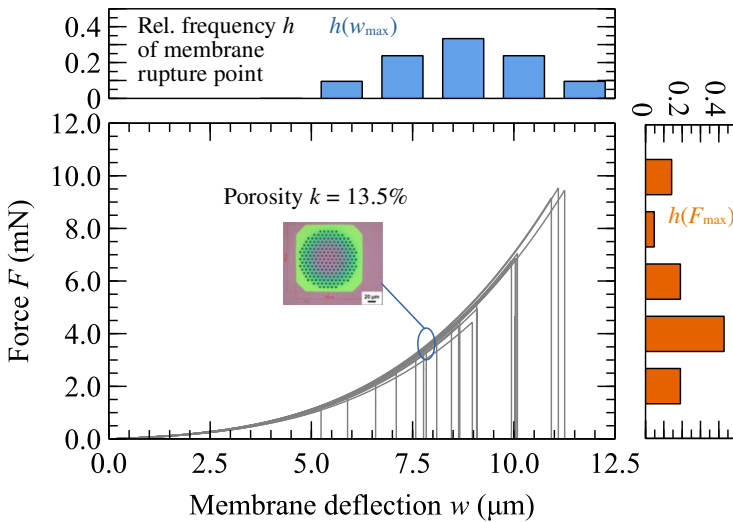


Figure 2: Force-deflection measurements of 21 membranes with porosity $k = 13.5\%$. Distributions of measured points of rupture are indicated with their relative frequencies $h(F_{\max})$ and $h(w_{\max})$.

Conclusion and Outlook

Our work demonstrates a method to extract mechanical properties of porous micromembranes. These properties are essential for membrane design, operation, and reliability in their target applications, e.g., filtration/sieving, liquid atomization, inkjet nozzles. Currently, the focus is on quasistatic load. In view of the various applications with dynamic operation, we envisage extending the study to dynamic load profiles. Furthermore, we intend to advance the modeling of membranes towards predictability and reliability.

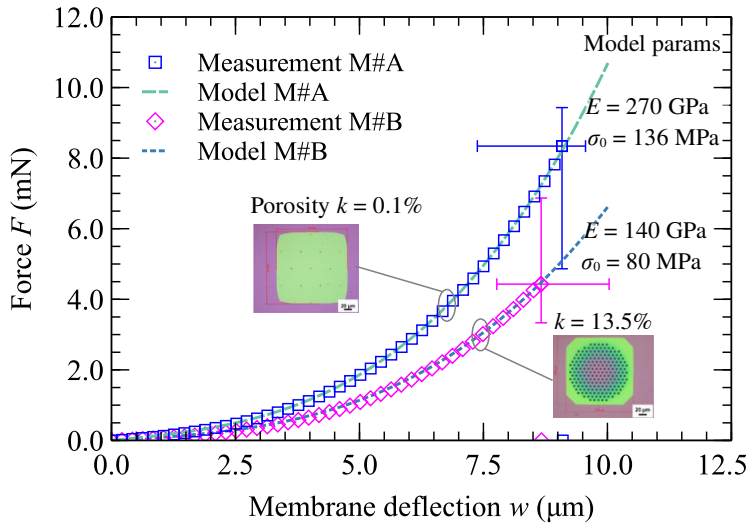


Figure 3: Average values of force-deflection measurements for 33 devices M#A and 21 devices M#B. Through the fit of (1), the corresponding model parameters, i.e., Young's modulus and pre-stress, were obtained. The points of rupture are indicated as median values with bars representing the first and third quartiles.

Acknowledgements

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3D printing of a microfluidic oxygenator

Wiebke Wiessner¹ (wiebke.wiessner@avt.rwth-aachen.de),
Lukas Hirschwald¹, Matthias Geiger¹, Sebastian Brosch¹,
Florian Neuhaus¹, John Linkhorst¹, Matthias Wessling¹

KEYWORDS: Microfluidics, artificial lung, Triply periodic minimal surface structures, direct laser writing

Abstract

Current extracorporeal membrane oxygenators use hollow fiber membranes to exchange oxygen and carbon dioxide with the surrounding bloodstream but cannot fully replicate the gas exchange capabilities of the human lung. The gas exchange rate is limited by the low surface-to-volume ratio and the long diffusion distances over laminar boundary layers on the blood side.

Higher surface-to-volume ratios and shorter diffusion pathways can be realized in microfluidic channels. However, microfluidic devices made with soft lithography are limited to 2.5D structures, leading to unfavorable flow conditions, where the majority of the channel surface does not

¹RWTH Aachen University, Germany

take part in gas exchange. 3D-structured membranes, such as triply periodic minimal surface (TPMS) structures, offer more membrane surface and induce mixing with their flow conditions. Macroscopic TPMS oxygenators have already shown improved gas transfer rates and reduced clogging due to enhanced flow conditions.

In this work, we use direct laser writing to 3D-print a microfluidic oxygenator with a TPMS-structured membrane. The membrane was printed from PDMS and features blood and gas channels with diameters of 20 μm , comparable to those of the capillaries in the lung. The combination of short diffusion pathways, mixing induced by channel geometries, and large membrane area-to-volume ratios is expected to greatly increase gas transfer rates and hemocompatibility for the extracorporeal membrane oxygenator.

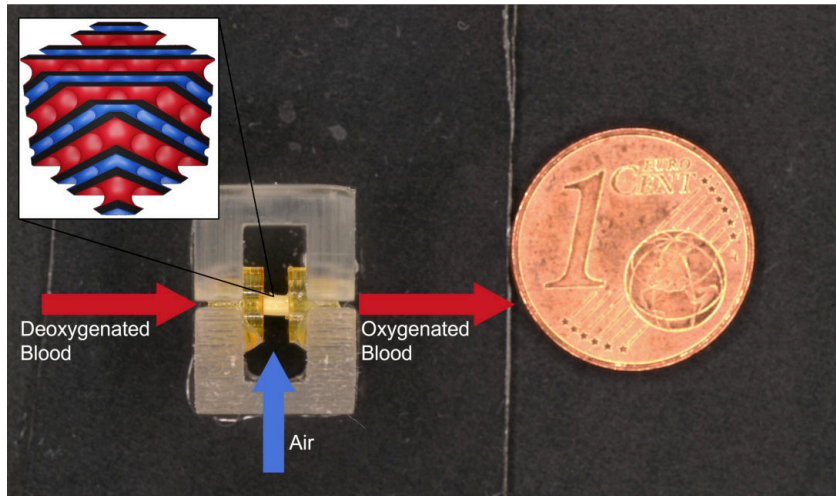


Figure 1: 3D printed microfluidic oxygenator with TPMS-structured membrane.

Posters

Biotechnology

Inoculation Techniques of Single Capillary Membrane with *Streptomyces coelicolor*

Piroska Lajtai-Szabó¹ (lajtai-szabo.piroska@mk.uni-pannon.hu),
Éva Hülber-Beyer, Nándor Nemestóthy

KEYWORDS: membrane gradostat reactor, biofilm formation, *Streptomyces coelicolor*

Introduction

The *Streptomyces* genus is of high importance in producing bioactive compounds, e.g., antibiotics and antitumorals. Secondary metabolite production usually occurs during the late growth phase, triggered by environmental stress factors [1]. However, the requisite limiting circumstances cannot be maintained in the long term. Therefore, secondary metabolites are generally produced in batch mode. A novel device called Membrane Gradostat Reactor (MGR) provides an alternative to accomplish continuous production. In this device, microbes form a biofilm on the outer surface of the membrane. Nutrients are circulated in the lumen, from which they diffuse to the outer surface. The biofilm layer closest to the membrane gains enough nutrients to grow, while cells further away suffer limitations, thus secondary metabolite production and biofilm renewal occur simultaneously [1]. One of the challenges of the MGR's practical implementations is appropriate inoculation, which results in a stable biofilm covering the entire membrane surface. Therefore, this study focuses on the effect of different inoculation techniques on biofilm formation of *S. coelicolor*.

Materials & Methods

Hydrophobic single capillary hollow fiber membranes were inoculated with *S. coelicolor* spore suspension. The inoculum contained different adhesives — agarose, starch, or carboxymethyl cellulose — and the effect of the type and concentration of them on biofilm formation and actinorhodin production was investigated with mass measurement, as well as visual and microscopic analysis.

Results & Discussion

Capillary membranes were successfully inoculated, although there were some differences in biofilm thickness and stability depending on the ad-

¹University of Pannonia, Hungary

hesive. Actinorhodin was also produced in cases where the system could be maintained for a sufficiently long time.

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Analysis of Impeller-Induced Shear Stress Distribution on a Membrane Surface in a Circular Test Cell for Porous Membranes

M. Haghshenasfard¹ (masoud.haghshenasfard@tu-dresden.de), A. Leon¹, K. Kerst¹, A. Lerch¹

KEYWORDS: Shear stress, CFD, Membrane, Rotating impeller, MBR

Abstract

Fouling is a common issue in membrane bioreactors, significantly reducing their separation efficiency. Increasing surface shear force is an effective strategy for mitigating or reducing fouling [1]. This study employed a shear-enhanced lab-scale membrane bioreactor, featuring a stationary circular membrane and a rotating impeller with a diameter of 35 mm, housed within a cylindrical cell with a diameter of 56 mm. Flow field simulations inside the membrane module were performed using the commercial Computational Fluid Dynamics (CFD) software, ANSYS Fluent. The influence of different rotational velocities on shear stress distribution was analysed, and the CFD predictions were compared with theoretical models. Shear stress contours at a rotation speed of 250 rpm, and the radial distribution of shear stress on the membrane under various rotation speeds are shown in Fig. 1a and b, respectively. The results indicate that increasing impeller speed leads to a corresponding increase in shear stress at the membrane surface, driven by a higher velocity gradient between the impeller and membrane. For instance, the maximum shear stress at 1000 rpm was 94%, 84%, and 47.5% higher than that observed at 100 rpm, 250 rpm, and 500 rpm, respectively. Shear stress increases with radial distance due to the rise in tangential velocity generated by the rotating impeller. Maximum velocity is observed near the

¹TU Dresden, Germany

rim of the impeller, while the minimum is at the cell center. Consequently, shear stress is lowest at the center of the cell and highest near the impeller rim ($r = 0.0175$ m). As illustrated in Fig. 1a, the shear stress on the membrane surface decreases steadily from $r = 0.0175$ m towards the wall, due to the wall's obstruction. Furthermore, because of the impeller's short length, the region between the impeller rim and the housing wall experiences less exposure to the high shear forces generated by the impeller.

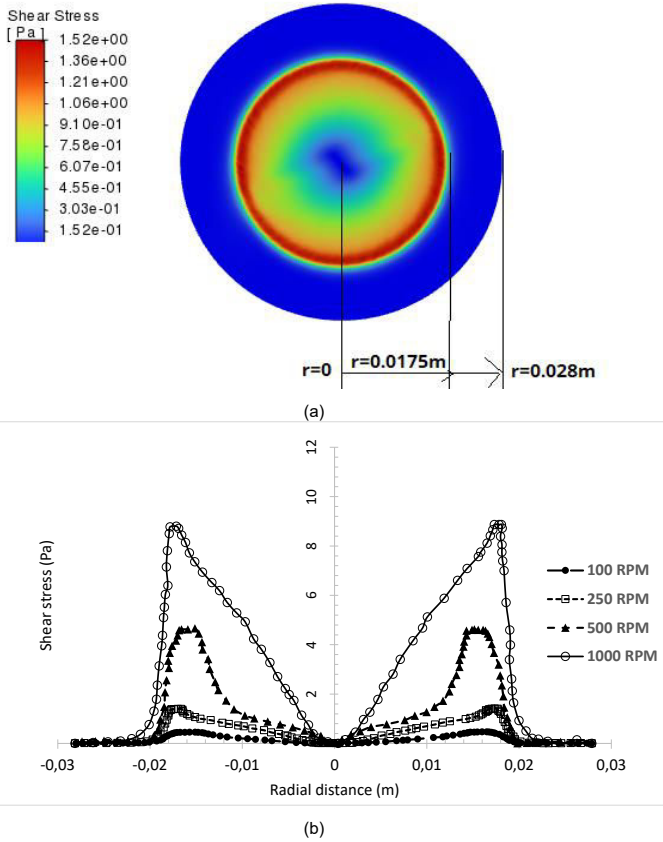


Figure 1. (a) Shear stress contours at a rotation speed of 250 rpm. (b) Radial distribution of shear stress on the membrane at various rotation speeds.

Figure 1: Radial distribution of shear stress on the membrane surface under various rotation speeds.

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Posters

Process Engineering

Hybrid Separations for Solvent Recovery: Distillation Versus Pervaporation – An Economic Comparison

Elmar Boorsma¹ (elmar.boorsma@pervatech.nl), Ilse Lammerink¹, Rob de Lange¹, Mohammed Nazeer Khan², Pieter Vandezande², Anita Buekenhoudt²

KEYWORDS: Pervaporation, Economic analysis, CAPEX/OPEX, Energy reduction, Solvent recovery, Solvent dehydration

Introduction

The chemical industry relies extensively on thermal-based separation processes, such as distillation, to achieve high-purity materials. These processes are energy-intensive, primarily utilizing steam generated from the combustion of natural gas. In recent years, the cost of steam has escalated significantly, with further increases anticipated due to diminishing natural gas supplies and the 2050 climate goals. This social and economic pressure necessitates that companies engaged in solvent recovery not only enhance the efficiencies of existing unit operations but also explore alternative separation techniques, such as hybrid systems. One notable example of such a hybrid system is the combination of distillation and pervaporation. In this configuration, a simple, conventional distillation column is employed to concentrate the solvent near azeotropic concentrations, followed by pervaporation to remove the residual water. Breaking of azeotropes via pervaporation was also explored by Baik et al. [1], specifically on the pressure dependent azeotrope between methanol and dimethyl carbonate. The conventional process uses a high and low pressure distillation column. Integrating a simple pervaporation system between these columns allows for the pressure in the HP column to be reduced, resulting in significant CAPEX and OPEX reductions of 46% and 21%, respectively.

This paper presents a techno-economic assessment of these hybrid separation systems. The analysis revisits the methodology and case studies originally presented by Van Hoof et al. [2] two decades ago, contextualizing them within the current economic landscape where costs have surged dramatically. Additionally, membrane development has sky-rocketed, and new generations of pervaporation membranes are examined for their

¹Pervatech, Heliumstraat 11, 7463 PL, Rijssen, The Netherlands

²Vito (Flemish Institute for Technological Research), Boeretang 200, B-2400 Mol, Belgium

impact on process efficiencies. The study includes a detailed examination of several solvents, such as N-methyl-2-pyrrolidone, isopropanol, 2-methyltetrahydrofuran, acetic acid, and acetonitrile, assessing their behavior and recovery efficiency in hybrid separation processes.

Materials & Methods

Simulations for the distillation processes were performed in Aspen Plus®, developed by AspenTech. The results were compared to the benchmark process of each solvent; for isopropanol, this is azeotropic distillation using benzene as the entrainer. For both the benchmark and the hybrid process, a feed flow rate of 1000 kg/h at 50:50 wt% composition and a final product purity of 99.5 wt% isopropanol were used. Pervaporation experiments were performed at Pervatech BV, using a lab-scale pervaporation set-up equipped with a 0.04 m² PERVATECH HybSi® AR membrane. Expected membrane replacement every 5 years is considered as OPEX. Membrane costs for the first 5 years are considered as CAPEX.

Results & Discussion

Separation costs (€ / ton product) using the hybrid distillation-pervaporation system were 39% less than the benchmark azeotropic distillation. CAPEX reduction in the pervaporation case was due to the elimination of expensive distillation columns. OPEX reduction in the pervaporation case was due to the lower steam and cooling water requirement.

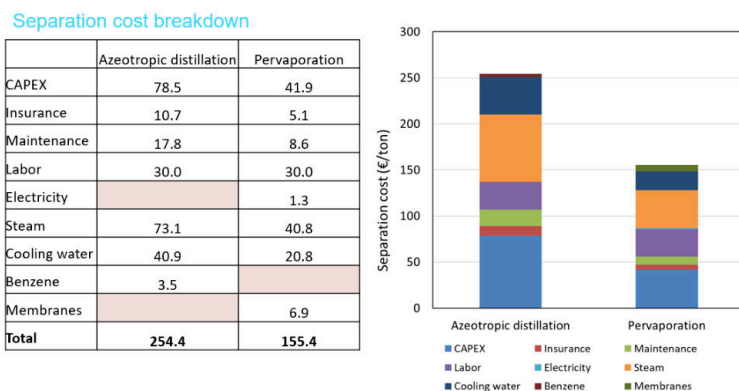


Figure 1: Techno-economic assessment for the dehydration of isopropanol via the azeotropic distillation process and the hybrid distillation-pervaporation process.

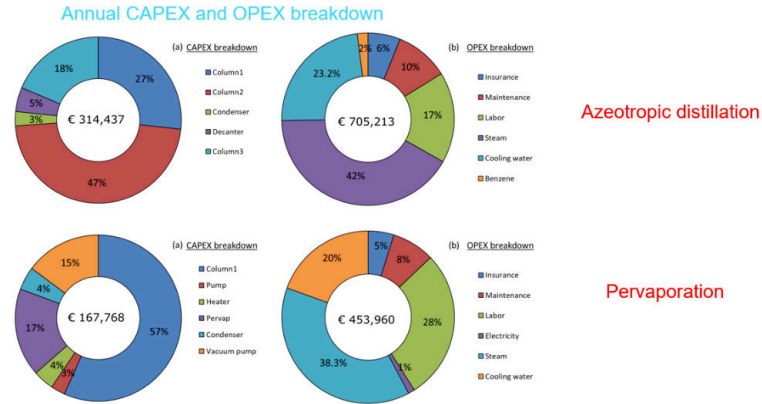


Figure 2: Breakdown of the annual CAPEX and OPEX for the azeotropic distillation process and distillation-pervaporation process.

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Organic Solvent Nanofiltration and Data-Driven Approaches

Pieter-Jan Piccard¹²³ (pieterjan.piccard@uhasselt.be), Pedro Borges³, Bart Cleuren¹, Jef Hooyberghs¹², Anita Buekenhoudt³

KEYWORDS: Organic Solvent Nanofiltration, Data-driven techniques, Ceramic membranes, Predictive models

Abstract

Industrial acceptance of organic solvent nanofiltration (OSN) as a separation technique is hampered by the slow process of membrane screening based on trial and error for each solute-solvent couple. Such extensive experimental screening is still necessary due to limitations of predictive models which are generally based on separations in water. The complexity and variety of competing interactions of solute-solvent-membrane affinities challenge our understanding and accuracy of predictions of the underlying separation mechanism.

Recently, data-driven techniques have shown their potential to enhance predictability in the field of OSN. We explore the use of data-driven techniques specifically for ceramic membranes, both native and functionalized, which have been one of VITO's technological focus points in OSN activities for many years. More specifically, we explore the dataspace of ceramic membranes to determine the key physico-chemical properties to track, as well as the least important features to discard. This aids the optimal development of data-driven models to predict OSN performance by reducing the data dimension and, at the same time, provides much-needed physical insight into the correlation between descriptors and the separation process.

The non-swelling property of ceramic membranes reduces the complexity of the separation process and consequently that of the data-driven prediction models required. However, challenges remain, including the amount of experimental data needed to represent the chemical space of interest, the dimensionality of input data, and the quality and completeness of the data. We believe data-driven models can provide the predictive power needed in OSN, while also helping to unravel the complex transport processes involved. This approach may ultimately lead to

¹Theory Lab, Faculty of Sciences, UHasselt—Hasselt University, Agoralaan, 3590 Diepenbeek, Belgium

²Data Science Institute, Faculty of Sciences, UHasselt—Hasselt University, Agoralaan, 3590 Diepenbeek, Belgium

³Unit Separation and Conversion Technology, VITO N.V.—Flemish Institute of Technological Research, Boeretang 200, 2400 Mol, Belgium

well-understood designs and leverage the acceptance of the technology in sustainable chemistry.

Predicting Protein Rejection In Membrane Separations Using Machine Learning Techniques

Gbenga Daniels¹ (gdani11@lsu.edu), Kofi S.S. Christie¹

KEYWORDS: Protein Rejection, Membrane Separation, Machine Learning, Gradient Boosting, XGBoost, Artificial Neural Networks, Random Forest, Ultrafiltration, Microfiltration, Cross-validation, Mean Squared Error, Coefficient of Determination, Ensemble decision-tree

Abstract

Protein rejection is a critical parameter in many applications of membrane separations because protein fouling can lead to reductions in both permeability and selectivity of a membrane. In this study, we leverage four machine learning (ML) models (Random Forest, XGBoost, Gradient Boosting, and Artificial Neural Networks) to predict protein rejection rates during ultrafiltration and microfiltration with varied operational conditions, membrane properties, and protein concentrations. Using a database of 505 data points derived from 83 peer-reviewed articles spanning 30 years, the models were trained and optimized through cross-validation and hyperparameter tuning to minimize mean squared error (MSE) and maximize the coefficient of determination (R^2). The Gradient Boosting model was the most effective, achieving an R^2 of 0.69, indicating its ability to explain 69% of the variance in unseen data, and an MSE of 0.033, demonstrating high prediction accuracy under varied operational conditions.

Our results demonstrate the superior predictive power of ensemble decision-tree ML models over traditional approaches and offer valuable insights into the design and operation of future membrane processes. The Shapley additive explanation method, rooted in cooperative game theory, was employed to reveal the impacts of different protein, operational, and membrane properties on the model predictions. This analysis demonstrated that the ML models can effectively capture the significant effects of size exclusion in regulating membrane separation. By predicting

¹Department of Civil and Environmental Engineering, Louisiana State University, Baton Rouge, LA, USA

the performance of yet-to-be-fabricated membranes, our findings can enhance industrial applications by enabling the creation of more effective and cost-efficient membrane configurations.

Membrane contactors and potassium-based solvents for biogas upgrade and direct CO₂ utilization

D. Koutsonikolas¹ (dkoutson@certh.gr), A. Asimakopoulou, N. Lazaridou, T. Grekou, G. Pantoleontos, G. Karagiannakis

KEYWORDS: Biogas upgrade, membrane contactors, CO₂ capture, gas-liquid contact membranes

Introduction

Biogas, a renewable energy source, is a gas mixture typically consisting of CH₄ (50-60%), CO₂ (40-50%) and other trace gas species (e.g., H₂S <1%). Removing CO₂ from biogas is needed to increase the CH₄ content of the fuel and thus its heating value or for direct injection into the existing natural gas grid. Existing CO₂ capture technologies are primarily based on solvent absorption, such as amines, in packed columns. Such applications have significant energy consumption and require the installation of voluminous equipment, resulting in reduced process efficiency and increased capital and operating expenses. CO₂ removal using membrane-based gas absorption is gaining attention as an alternative to industrial well-established gas separation and reaction equipment due to membrane modules' high mass transfer area (known a priori), modular design, and easy scale-up. Moreover, employing potassium-based solvents instead of amines can offer economic and environmental benefits and also the potential for the direct electrochemical conversion of CO₂ to potassium formate, which is considered a very promising energy carrier suitable for long-term energy storage [1, 2].

Materials & Methods

In the current work, the CO₂ capture process from simulated biogas streams is experimentally studied using commercial 3M™ Liqui-Cel membrane contactors and potassium-based solvents. The gas and liquid flows, as well as the CO₂ concentrations upstream and downstream of the membrane module, are continuously monitored to parametrize the

¹Centre for Research & Technology Hellas - CERTH, Greece

process and assess the performance of the proposed technology under different operating conditions.

Results & Discussion

The experimental results indicated that the CO₂ capture rate increases with liquid flow rate and decreases with gas flow rate, while CO₂ capture rates up to 100% can be achieved for selected process conditions. On the other hand, CO₂ flux increases with both liquid and gas flow rates. Generally, the results revealed the high efficiency and flexibility of the process.

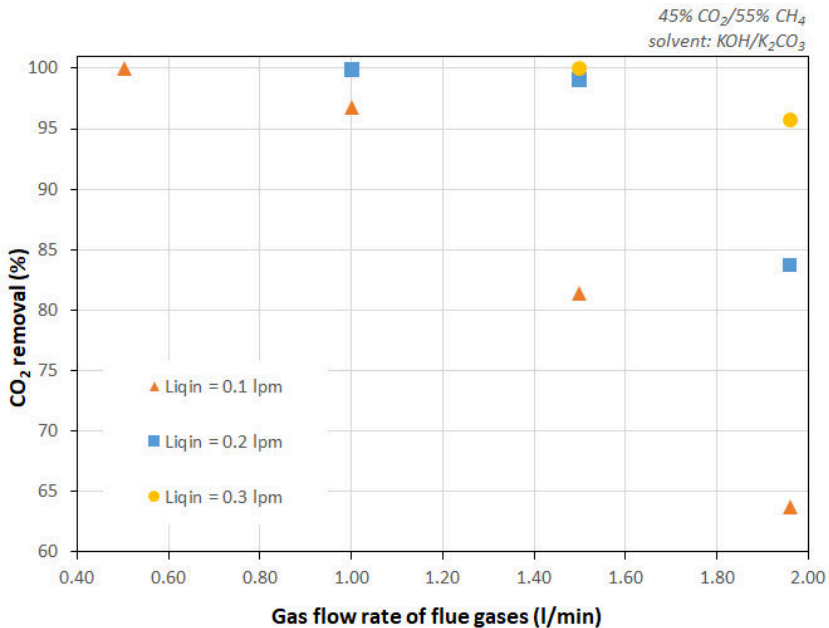


Figure 1: CO₂ removal with gas flow rate for different liquid flow rates.

Acknowledgments

The work has received financial support through the project FRESH - Formate for Renewable Energy Storage, funded through HORIZON EUROPE (GA No. 101069605).

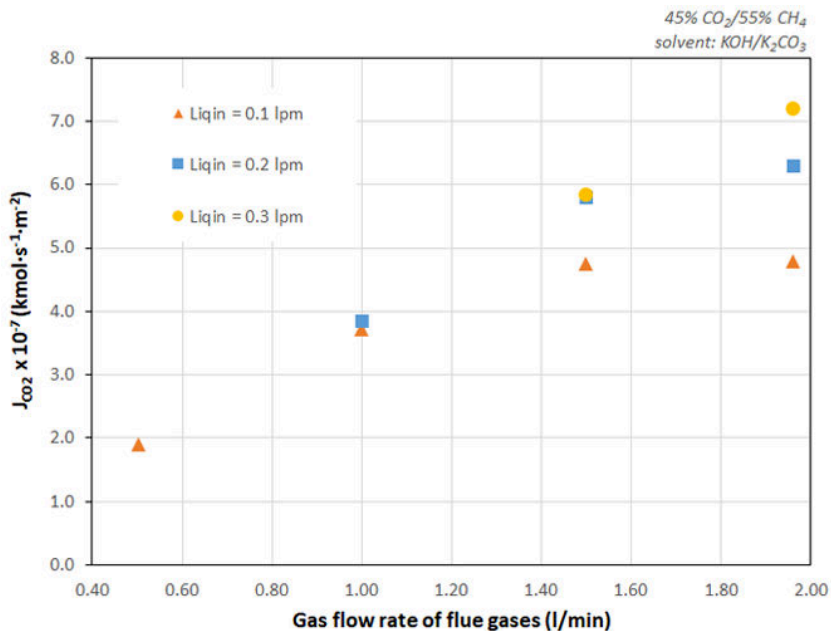


Figure 2: CO₂ flux with gas flow rate for different liquid flow rates.

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INNOMEM – Open Innovation Test Bed For Nano-Enabled Membranes

Kristianne Tempelman (k.tempelmann@emi-twente.nl)¹

KEYWORDS: OITB, Single Entry Point (SEP), R&D services, innovation

Abstract

INNOMEM provides a single entry point for all membrane related questions. INNOMEM is a collaboration between 8 parties, each with their own specialty in the membrane field. The consortium 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. Therefore SME's can contact the OITB for both the acquisition of technical and non-technical services. The success of INNOMEM has been demonstrated by several demonstration cases.

¹EMI Twente, Germany

The Open Membrane Database: a data hub for membrane technology

R. Verbeke, S. Van Buggenhout, H. Roth, S. Herrmann¹ (s.herrmann@utwente.nl), G. Ignacz, G. Szekely, M. Elimelech, G. Ramon, C. Tang, T. Stassin, and I. Vankelecom

KEYWORDS: Membrane database, RO membranes, SRNF membranes, HF membranes, Gas separation membranes, FAIR and open data

Abstract

The open membrane database (OMD) aims to make membrane data from academia and industry findable, accessible, interoperable, reproducible, and open (FAIR/O). FAIR/O data is crucial to accelerate research, understand fundamentals, and solve essential challenges, e.g., related to the United Nations Sustainable Development Goals, as efficiently as possible. [1]

The OMD started to collect data for reverse osmosis (RO) membranes [2] in 2021 and was recently extended to solvent-resistant nanofiltration (SRNF), also called organic solvent nanofiltration (OSN) [3]. All data points in the OMD are peer-reviewed and validated by the OMD community. The OMD offers the opportunity to add membrane data for everyone, as long as the data set is already published, in a standardized way using an online submission tool. The database is open to the public and can be visualized, filtered, and searched on the OMD website for extensive data exploration. Single data points are directly linked to the respective publication to ensure the findability of the original data.

The OMD already contains a comprehensive dataset on the membrane materials, synthesis parameters, operational conditions, physicochemical properties, and performance of OSN/SRNF and RO membranes.

The next extension of the OMD focuses on hollow fiber (HF) membranes, especially complemented by spinning parameters and gas separation (GS) membranes. The backend is currently being set up, and data collection in the GS and HF membrane communities will start within the next few months.

The OMD thereby paves the road for FAIR/O data in the field of RO, OSN/SRNF, GS, and HF membranes to increase transparency, enable more accurate data analysis, and foster collaboration and innovation.

www.openmembranedatabase.org

¹University of Twente, The Netherlands

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Posters

Gas and Vapor Separation

Development of a Hollow Fiber Membrane Based Enthalpy Exchanger

Lisa Krüger¹ (lisa.krueger@eonerc.rwth-aachen.de), Wiebke Wiessner¹, Martin Kremer¹, Dirk Müller¹, Christian Linnartz¹, Matthias Wessling¹

KEYWORDS: Membrane Enthalpy Exchanger, Hollow Fiber Membranes, Tubular Membranes

Abstract

Temperature and humidity management in buildings significantly contributes to high energy consumption. Air handling units (AHUs) with latent and sensible heat recovery, therefore, offer substantial saving potential. Common enthalpy exchangers rely on flat sheet membranes for heat and mass transfer. However, hollow fiber membranes and tubular membranes present a promising alternative due to their ease of manufacturing and self-supporting characteristics. Additionally, a high fiber packing density increases the active membrane surface area per unit volume, potentially enhancing the efficiency of enthalpy exchangers or reducing the component's dimensions.

We introduce a research project that combines the development, characterization, and simulation of a hollow fiber membrane enthalpy exchanger with reduced energy demand and operating costs.

At the beginning of the project, we fabricate water vapor permeable tubular membranes and characterize their permeance and selectivity experimentally. We examine their integration into the enthalpy exchangers using computational fluid dynamics (CFD). Parameter studies will be conducted to determine optimal membrane arrangements and geometric boundary conditions, followed by the construction of a functional prototype for experimental evaluation in an AHU. Concurrently, a dynamic model will be developed based on the obtained data, enabling year-round simulation of the system.

The project aims to enhance the performance of enthalpy exchangers, thereby contributing to more sustainable building ventilation systems.

¹RWTH Aachen University, Germany

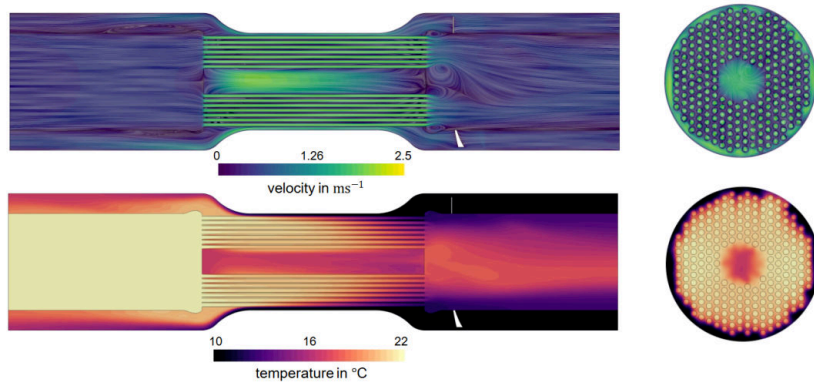


Figure 1: Flow field and temperature field of the enthalpy exchanger module.

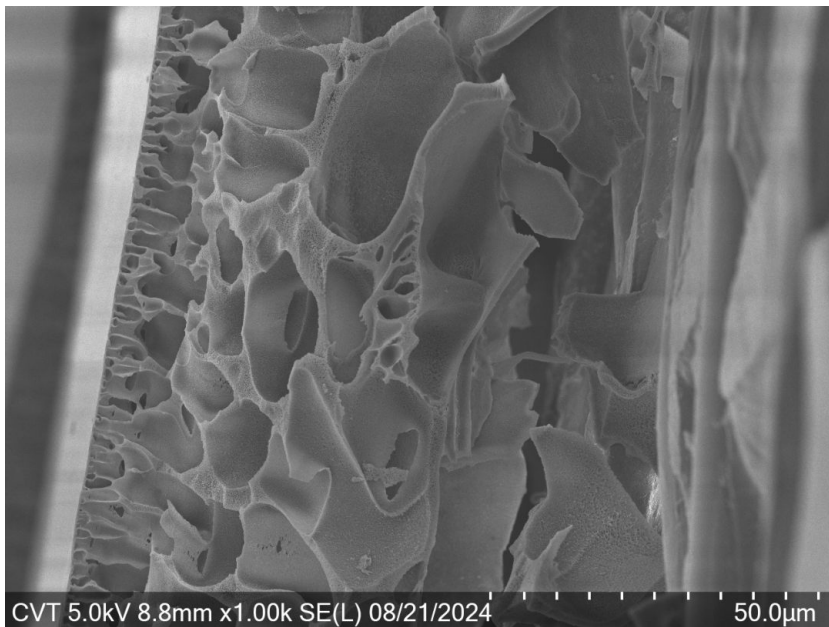


Figure 2: Electron microscope image of the membrane.

Polymer Blending for Advanced Gas Separation Membrane Materials

R. Válek¹ (robert.valek@membrain.cz), O. Horký¹

KEYWORDS: Polymer blend, gas separation, hollow fiber membranes, phase inversion, DOE

Abstract

Polymers blends could be promised materials for membranes. In this work, we use polymer blend to increase permeance of the polyetherimide membranes. Polyetherimide, whose typical representative is Ultem 1000, is a suitable membrane material with a favorable price, useful selectivity and a low tendency to plasticize. Its disadvantage can be low gas permeability. Ultem 1000 was used as the majority polymer component of the membrane material. It was combined with Matrimid 5218 polyimide or another polyimide equivalent made in China. Matrimide-type polyimides show good selectivity and excellent permeability. An obstacle to the wider use of this material can be its very high price and tendency to plasticize with carbon dioxide at higher pressures. These disadvantages can be eliminated by using a polymer blend as membrane material.

The permeation of gases through membranes based on polymer blend does not show a simple mechanism, and the resulting permeability strongly depends on the achieved microstructure of the polymer blend.

Hollow fibers were formed from the polymer solution by the phase inversion method. Advanced statistical methods of experiment design (DOE) were used to optimize the transport and separation properties of the membranes. Hollow fiber membranes were tested in small laboratory modules as well as in larger ones with a membrane area of around 5 m². Permeance was measured on single gases as well as on gas mixtures. The microstructure of the membranes was studied by light and electron microscopy and DSC.

By spinning a solution containing two types of polymers, it was possible to prepare hollow fiber membranes. The best membranes showed an air separation selectivity of 8 at an oxygen permeance of 12 GPU. Furthermore, these membranes showed an ideal selectivity for hydrogen and methane separation of 200 at a hydrogen permeance of 130 GPU. DSC measurements showed the presence of two glass transition temperatures, which proves the two-phase structure of the membrane. Temperatures of glass transition allow to estimate the proportion of polymer components in the individual phases of the polymer blend.

¹MemBrain, Stráž pod Ralskem, Czechia

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The work was carried out within the framework of the project “A long-term concept for the development of the research organization,” supported by the Ministry of Industry and Trade of the Czech Republic, using the Membrane Innovation Centre infrastructure.

Appendix

Index of Exhibitors

Index of Exhibitors

Atech innovations GmbH

Contact: **Dipl. -Ing. Erich Kossin**
Address: **Am Wiesenbusch 26**
45966 Gladbeck
Germany
Tel.: **+49 (0) 2043 9434 0**
E-mail: **info@atech-innovations.com**
Web: **www.atech-innovations.com**

AVT.CVT - Chemical Process Engineering

Contact: **Dr. Robert Keller**
Address: **RWTH Aachen University**
Forckenbeckstraße 51
52074 Aachen
Germany
Tel.: **+49 241 80-47817**
E-mail: **secretary.cvt@avt.rwth-aachen.de**
Web: **www.avt.rwth-aachen.de**

Blue Foot Membranes

Contact: **Steffen Richter**
Address: **Josink Kolkweg 23**
Benzstraße 5
48369 Saerbeck
Germany
Tel.: **+49 (2574) 8875 8259**
E-mail: **info@bluefootmembranes.com**
Web: **www.bluefootmembranes.com**

Blue Foot Membranes NV

Contact: **Steffen Richter**
Address: **Gerard Mercatorstraat 31**
B- 3920 Lommel
Belgium
Tel.: **+32 (0) 11 73 66 96**
E-mail: **srichter@bluefootmembranes.com**
Web: **www.bluefootmembranes.com**

Demcon Convergence

Contact: **Hassein Alinia**
Address: **Institutenweg 21**
7521 PH Enschede
Netherlands
Tel.: **+31 88 215000**
E-mail: **hassein.alinia@demcon.com**
Web: **www.demcon.com**

EMI Twente B.V.

Contact: **Dr. ir. Patrick de Wit**
Address: **Drienerlolaan 5**
75722 NB Enschede
Netherlands
Tel.: **+31 (0)53 7400 642**
E-mail: **info@emi-twente.nl**
Web: **www.emi-twente.nl**

Evonik Industries AG

Contact: **Dr. Goetz Baumgarten**
Address: **Paul-Baumann-Straße 1**
45772 Marl
Germany
Tel.: **+49 (0) 2365 4901**
E-mail: **sepuran@evonik.com**
Web: **www.membrane-separation.com**

FTRJ GmbH

Contact: **Alexander Felix**
Address: **Chemnitzer Straße 65**
09669 Frankenberg
Germany
Tel.: **+49 37206 573688**
E-mail: **a.felix@ft-rj.de**
Web: **www.ft-rj.de**

Linde GmbH, Linde Engineering

Contact: **Christian Voss**
Address: **Dr.-Carl-von-Linde-Straße 6-14**
82049 Pullach
Germany
Tel.: **+49 (0) 89 7445 2215**
Web: **www.engineering.linde.com**
/hiselect-for-hydrogen

PVF Mesh & Screen Technology GmbH

Contact: **Florian Weidenbach**
Address: **Adalbert-Stifter-Weg 30**
85570 Markt Schwaben
Germany
Tel.: **+49 (0) 8121 47840**
E-mail: **info@pvfgmbh.de**
Web: **www.pvfgmbh.de**

SIMA-tec GmbH

Contact: **Siegfried Tuchborn**
Address: **Schier 12 c**
41366 Schwalmtal
Germany
Tel.: **+49 (0) 21366 349210**
E-mail: **siegfried.tuchborn @sima-tec.de**
Web: **www.sima-tec-gmbh.de**

Solventum

Contact: **Martin Ulbricht**
Address: **Edisonstraße 6**
59174 Kamen
Germany
Tel.: **+49 (0)2131/8819094**
E-mail: **martin.ulbricht@solventum.com**
Web: **www.solventum.com**

Start-Up Corner

Amovion

Contact: **Christian Linnartz**
Address: **Forckenbeckstraße 50**
52074 Aachen
Germany
Tel.: **+49 (0) 175 3884499**
E-mail: **christian.linnartz@amovion.de**
Web: **www.amovion.de**

flex-X-cell GbR

Contact: **Jonas Bäßler**
Address: **Forckenbeckstraße 50**
52074 Aachen
Germany
Tel.: **+49 (0) 157 89272554**
E-mail: **jonas.baessler@flex-x-cell.com**
Web: **www.flex-x-cell.com**

Naiontech

Contact: **Milan Abel**

Address: **Forckenbeckstraße 51**
52074 Aachen
Germany

Tel.: **+49 241 80 98095**

E-mail: **milan@naion.tech**

Web: **www.naion.tech**