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Membrane News Twente

News magazine of the Membrane Technology Group

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Membrane Process Technology

Walter van der Meer

Antoine Kemperman

On Thursday, March 27th, 2008, Walter van der Meer, part-time professor of Membrane Process Technology at the University of Twente, gave his inaugural lecture. The title of his presentation was 'Membranes: Performing under Pressure.' Membrane technology is a good alternative for many existing processes in drinking water production and waste water treatment. Membranes are modular, come in many commercially available varieties, their operation is simple, and they only require a limited amount of chemicals. However, depending on the membrane type (MF, UF, NF, or RO), there are several problems associated with the use of membranes in water treatment: organic and inorganic fouling, biofouling, energy use, cleaning the membranes, and disposing of the concentrate. The Membrane Process Technology (MPT) group aims to understand the complex interaction between the process conditions, the membrane, and the feed components shown schematically in Figure 1. MPT carries out research projects which envisage the role of these different actors in the membrane filtration process. The projects can be grouped into three different subjects: fouling, biofouling, and cleaning.

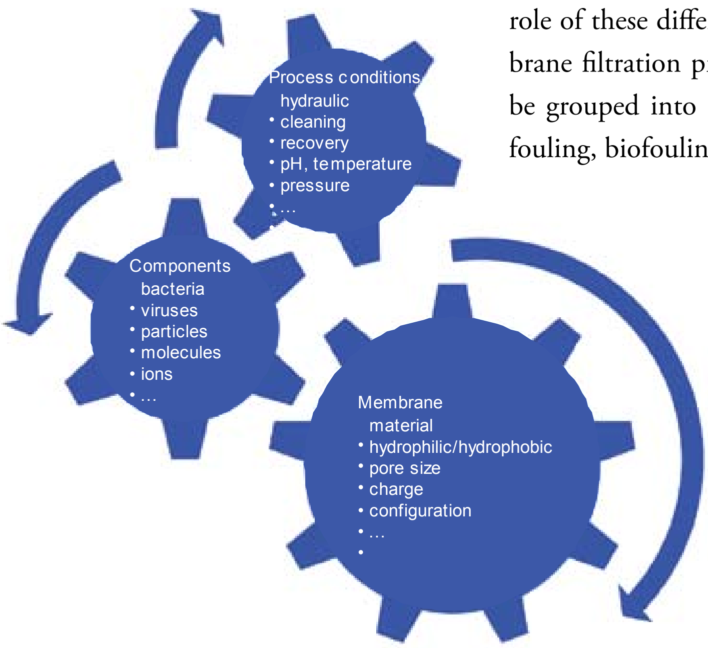


Figure 1: The three most important actors in membrane filtration.

Fouling

Membrane properties such as charge, hydrophilicity, pore size, and membrane and module configuration, combined with the applied process conditions, determine whether organic and inorganic fouling of the membrane can take place. Methods such as the silt density index (SDI), fouling index, and membrane filtration index (MFI) are often used to study the interaction between particles and membrane and spacer surfaces. MPT is investigating, on a fundamental level, why inconsistencies sometimes occur within and between SDI and MFI measurements. By investigating membrane properties (charge, surface roughness, hydrophobicity/hydrophilicity, etc.,) we try to gain a better understanding of these methods on a fundamental level, with the goal of improving them for practical use.

A disadvantage of these methods is the indirect determination of particle-membrane interaction. For that reason, one project focuses on the direct visualization of interactions between organic particles and membrane and spacer surfaces. In a microfluidic device (Figure 2), these interactions are visualized using a high-speed

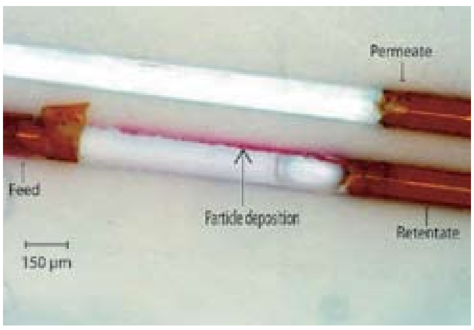


Figure 2: Microfluidic device for fouling research.

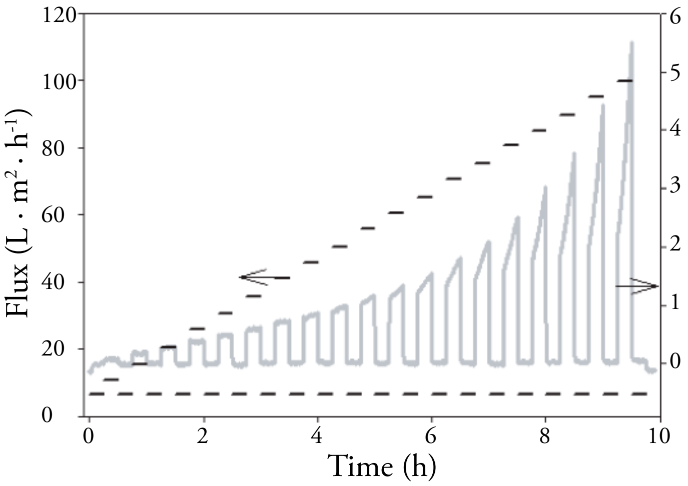


Figure 3: Improved flux step method with a relaxation step.

camera. The particles' trajectory in the feed channel is followed in time, giving us essential information on particle-membrane and particle-spacer interaction. Ultimately, this will result in a mathematical model providing us with a tool for upscaling, and giving us a method for predicting which particle-particle interactions are important and which are not.

Finally, a third research line focuses on mapping the factors playing a role in the possible interaction of larger organic molecules, such as alginates and humic acids, with the UF membrane surface. Since the presence of inorganic ions such as Ca^{2+} and K^{+} affects this interaction, this is also a point of attention. Ions change the structure of the foulant (alginate), and as a result the critical flux is different.

Biofouling

In biofouling, bacteria, fungi, viruses, and higher organisms adhere to the membrane and spacer surfaces. This causes an increase in the pump energy (and expense) required to maintain the desired flux. Biofouling is a very complex process, since biomass is able to reproduce and adapt to circumstances. To study biofilm formation in real time, instead of afterwards by membrane autopsy, we use special membrane modules: a high-pressure cell (with permeate production) and a membrane biofouling simulator (without permeate production). The influence of the process conditions on biofouling, as well as that of the type of membrane (e.g. material, surface roughness) and spacer is studied with the goal of finding operational parameters and materials for reducing biofilm formation. A second way to achieve this goal is to use an adapted biofouling monitor, in which biofouling of membrane and spacer materials in time can be tested under identical process conditions.

Biofouling is also studied in membrane bioreactors (MBRs). A high biomass concentration is desirable for optimum operation of an MBR, but this might cause severe biofouling on the membranes. An improved flux-step method was developed in order to find the most favorable biomass concentration (Figure 3). The strong point of this method is that we can easily distinguish between reversible and irreversible fouling. By applying this flux-step method, we investigate the influence of parameters such as membrane material, pore size, particle size, and process conditions on the effectiveness and critical flux of the applied MBR process.

Cleaning

Membrane fouling and biofouling always occur in practice, and physical, mechanical, or chemical cleaning is necessary. However, cleaning shortens the membrane lifetime, necessitates disposal streams, and reduces production capacity. We are investigating the optimization of chemical membrane cleaning, and the use of a two-phase (air-water) cleaning method. In the latter method, air is injected into spiral-wound NF and RO modules for cleaning. The first results are very promising, and we are therefore studying this method on a more fundamental level now. The project focuses on the influence of hydraulics, membrane and spacer configuration, and the presence of particles and bacteria. The air-water interface can be visualized using a high-speed camera in combination with a flat test cell (Figure 4). Such measurements will be used to formulate a mathematical model for two-phase cleaning.

For more information please visit our website (http://mtg.tnw.utwente.nl) or contact Dr. Antoine Kemperman (a.j.b.kemperman@utwente.nl).

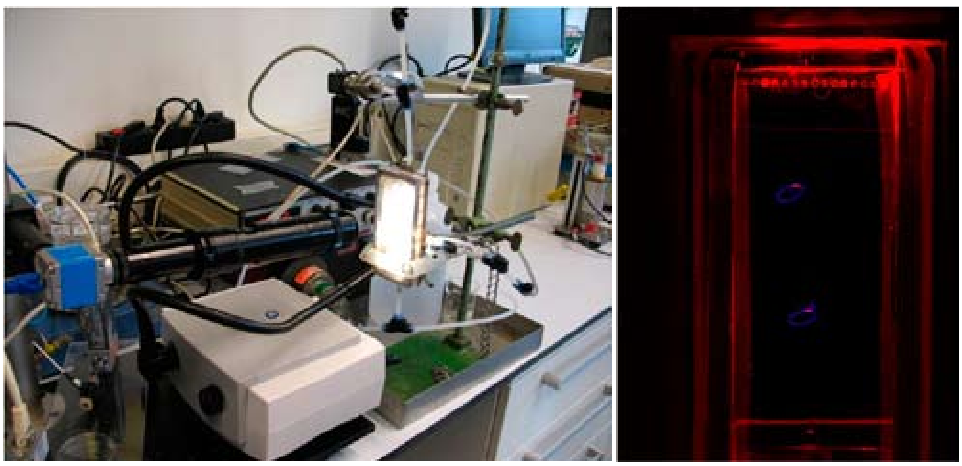


Figure 4: Setup with high-speed camera and flat test cell (left), and a digitally processed image of an air-water mixture in the cell (right).

Application of Membrane Technology in Microfluidic Devices

'Microfluidic' refers to the research field that develops methods and devices for controlling, manipulating, and analyzing flows in sub-millimeter dimensions. Heat and mass transfer in these systems can be predicted and controlled efficiently, due to well-defined flows and high surface-to-volume ratios. As a result, reactions can be pushed to higher yields, higher selectivities, and less waste. In analysis, the small dimensions enable efficient analysis of small sample amounts, with faster results and better resolution. Stacking multiple microdevices opens up the way for rapid screening of new chemical reactions and process conditions. Therefore, microfluidics is expected to revolutionize chemistry and biology, just as microelectronics revolutionized information technology in the previous century.

A current focus in the field is on the integration of multiple unit operations in a single device. Many of these operations, such as prefiltration, separation, or contacting, can be carried out using membranes. Additionally, in our membrane field, a lot of knowledge and experience is available on topics that are also covered by the field of microfluidics, including interaction of materials, materials processing, sealing, mass transport, and module design. The main aim of this explorative PhD project was to bridge both fields and discover new opportunities.

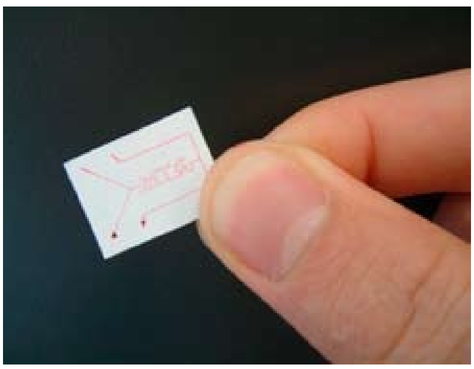


Figure 1: Porous microfluidic chip made by phase separation micromolding.

First, we discuss an overview of the state-of-the-art technology in the integration and application of membranes in microfluidic devices. We focus especially on devices made of polydimethylsiloxane (PDMS), which is currently one of the most popular materials in academic microfluidic research.

The first micro devices were prepared using phase separation micromolding (PSμM). This process results in a membrane with integrated microfluidic structures. After sealing to a cover slip, a porous microfluidic chip is obtained (Figure 1). The walls of the microchannels can be used for selective mass transport of gases, liquids, and/or solutes. A proof-of-principle was realized by visualizing CO_2 transport through the channel walls.

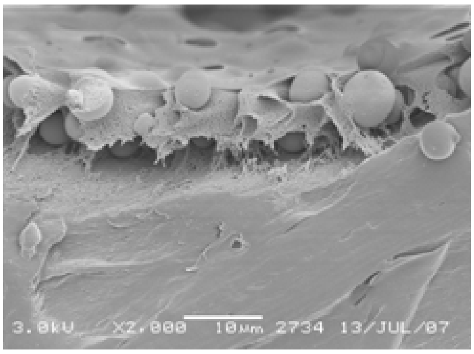


Figure 2: Porous coating in a microchannel with silica particles functionalized with platinum catalyst.

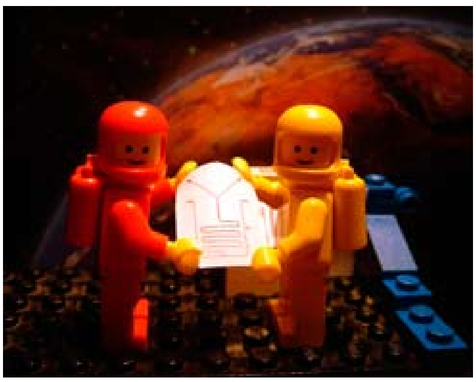
The phase separation principle can also be exploited for the preparation of thin porous coatings in microchannels. The morphology can be tuned and particles can be incorporated, leading to functionalized coatings (Figure 2).

A more straightforward method for integrating membrane functionality is to clamp membranes between substrates with microchannels (Figure 3). These channels can be made by micro milling, which enables rapid prototyping. Prepared devices have been tested in micro gas-liquid contacting. Results from absorption mea-

Figure 3: Switching pH gradients in microchannels.

surements of CO_2 in water show good agreement with a basic 2D COMSOL model, demonstrating the predictability of mass transport on the small scale. By using acidic and basic gases, such as CO_2 and NH_3, the pH of a liquid can be influenced locally. This approach was first exploited in explorative micro fermentation experiments. Subsequently, it was used to locally create stationary and moving concentration gradients (Figure 3). Two examples that broaden the concept further can be mentioned: local crystallization of NaCl using HCl vapor, and consecutive reactions of ammonia with copper (II) ions.

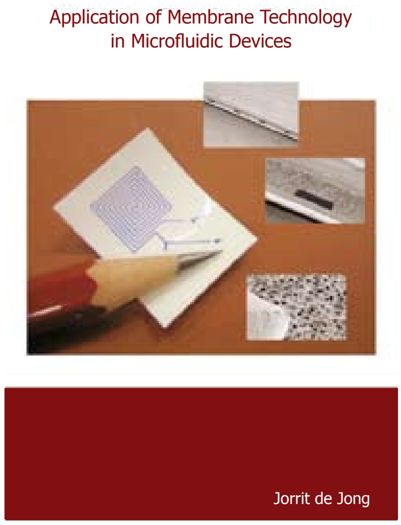
Finally, a special chapter in the thesis is dedicated to tips, tricks, and new concepts. Simple methods targeting the integration of membrane functionality in microfluidic devices include the use of hollow fibers and embossing micro structures in porous membranes. Furthermore, it has been demonstrated that hollow fiber membranes can be used as an



intermediate in the preparation of micro packed beds and monoliths. After filling, the porosity of the membrane is removed using heat treatment. This densification concept also enables the preparation of local optical windows in membrane systems, which can be exploited for studying flow and fouling behavior. The chapter ends with general guidelines for membrane integration.

Summarizing the thesis in a single sentence, it gives a nice overview of the field of microfluidics from the perspective of a membrane technologist. It can therefore serve as a good starting point for anyone interested in entering this exciting area.

Jorrit de Jong defended his thesis on April 18th, 2008. A digital copy can be obtained by sending an e-mail to info@membrane.nl. Jorrit currently works at the research department of Teijin Aramid in Arnhem, where he is involved in spinning aramid fibers. He can be contacted at jorrit_de_jong@yahoo.com.



OSMO: Optical Saving in Membrane Operation

One of the large challenges that engineers face in the coming century is supplying the world with clean drinking water. Due to climate change, increasing global population, and water demand, it is becoming increasingly difficult to prepare drinking water from surface water sources. Membrane technology has been recognized as the leading-edge technology for producing clean and safe drinking water, at competitive price levels.

Dead-end ultrafiltration can replace many of the traditional processes for producing drinking water from surface water. Compared to cross-flow filtration, dead-

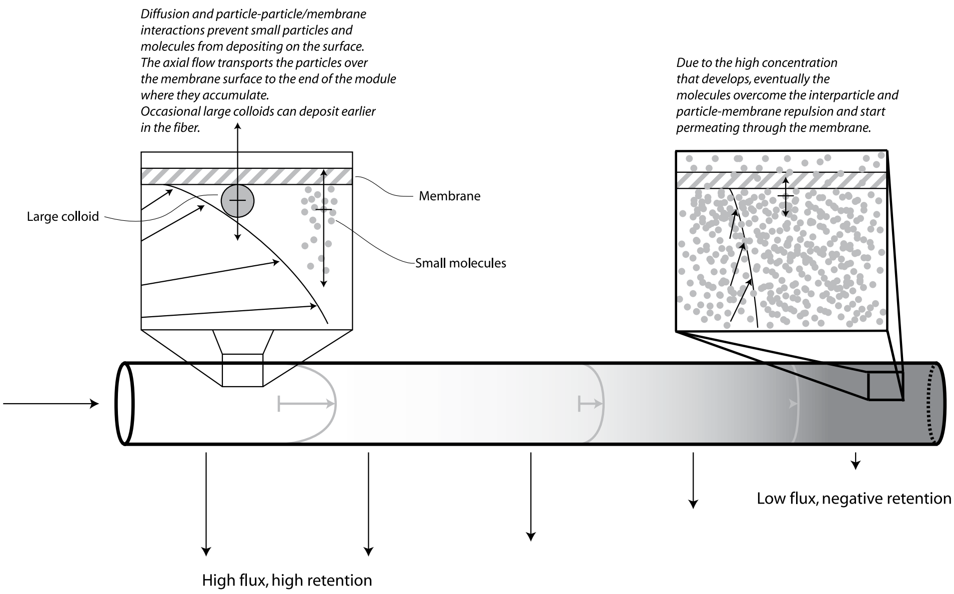


Figure 2: Localized fouling of dead-end hollow fiber membranes and the implications for the filtration process. The flux and the quality of the permeate will decrease over the length of the fiber.

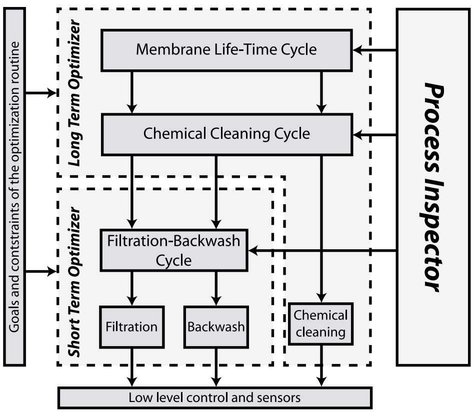


Figure 1: Scope of the OSMO project, showing the division of the membrane filtration life-cycle into short-term operation, long-term operation and the position of the process inspector.

end filtration is more energy-efficient. However, membrane fouling in dead-end filtration is more severe. In order to counter fouling, the modules are backwashed frequently. When backwashing alone is not sufficient to restore membrane performance, chemicals are used.

The OSMO project aimed to understand and optimize the process operation of dead-end ultrafiltration. The project was split into three smaller projects, each carried out by one PhD student. The first PhD focused on short-term (filtration and backwash) optimization, and the work of the second PhD was dedicated to long-term (chemical cleaning and aging) optimization. The third PhD, Wilbert van de Ven, developed an Inspector, which is a new method of determining the filterability of the feed water and the state of the process (Figure 1). The results of this work are the process control variables to be used as key parameters by the optimizers.

Wilbert van de Ven worked on the Process Inspector project in the Membrane Technology Group. His thesis describes two novel ways to characterize the filterability of feed water in combination with the membrane process used. The first method focuses on determining a critical flux in dead-end ultrafiltration, while the second method shows the extent of interaction that occurs between constituents of the feed water with the membrane material using flow-field flow fractionation (flow-FFF), a commercially available analysis technology.

Wilbert van de Ven successfully defended his thesis, 'Optimal Saving in Membrane Operation, the Development of Process Inspection and Feed Water Characterization Tools' on April 24th, 2008. He currently works as a water treatment expert at Vitens, the Netherlands' largest drinking water company. Water treatment experts at Vitens are responsible for the continuous supply of clean drinking water to the public, and study process improvements and innovative water cleaning technology (for more information: wilbert.vandeven@vitens.nl).

In addition to these two new characterization techniques, Wilbert's work describes a method for optimizing the backwash operation of dead-end ultrafiltration fiber membranes. The new backwash process was derived from the observation that membrane fouling, under specific conditions, is heavily dependent on the axial position in the fiber (Figure 2). In the newly-developed partial backwash concept, only the part of the fiber that is heavily fouled is hydraulically cleaned. The rest of the module does not require frequent backwashing (Figure 3). Experiments with various model solutions revealed that the partial backwash concept results in the same filtration characteristics as regular backwashing, at 80% lower backwash water consumption.

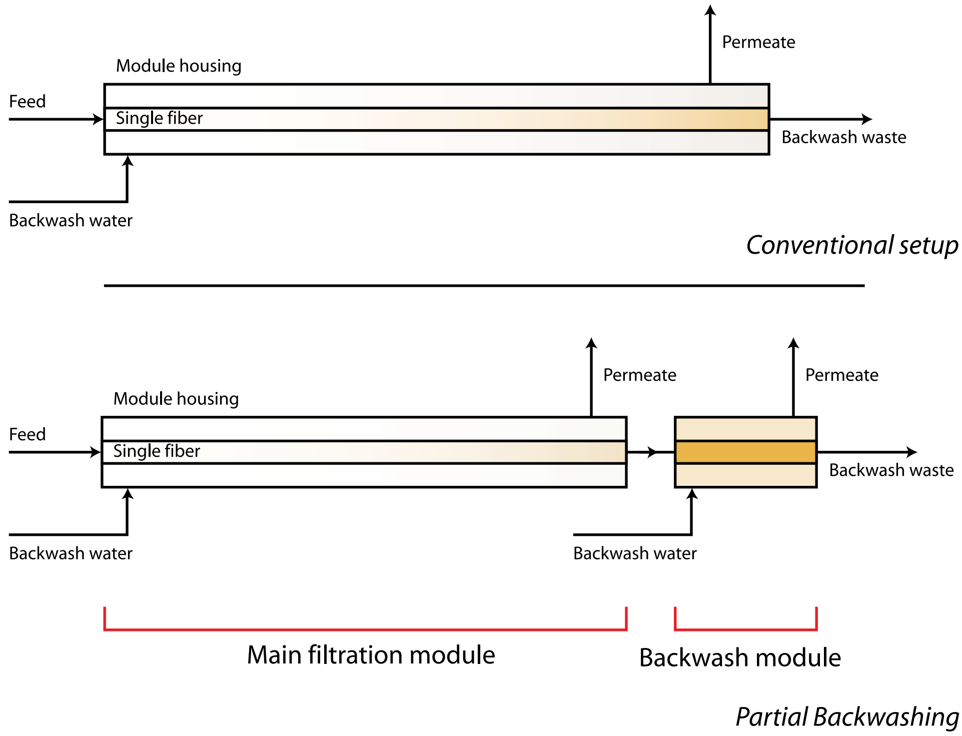
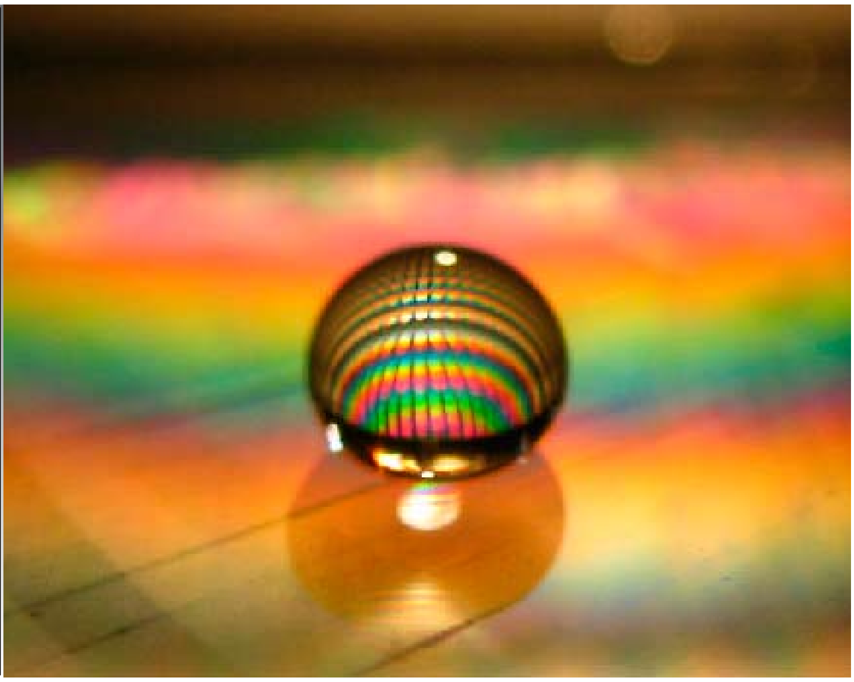


Figure 3: The concept of partial backwashing. Since membrane fouling primarily occurs at the end of the fiber, lower backwash water usage can be achieved by mainly backwashing the fouled part of the fiber.

Picture of the month, January 2008
Water droplet on a superhydrophobic surface



The picture shows a water droplet on a superhydrophobic (structured) surface. The substrate consists of cylindrical posts on a square lattice, made of SU-8 photoresist. The surface roughness causes diffuse reflection.

Picture by Gor Manukyan

Picture of the Month Competition

The picture of the month is chosen from the pictures taken in the MTG group during the past month. The winner receives free drinks for one night and gets to pick the winner from the next month's contributions. All the winners can be found on the website www.membrane.nl

Membrane Runners participate in Batavierenrace

Once again, the Membrane Technology Group participated in this year's Batavierenrace, which was organized on April 26th, 2008. The group was registered as the Membrane Runners, and all of the membrane participants put in an enormous effort. For many years, participation in the Batavierenrace has been a recurring event for the Membrane Technology Group.

The Batavierenrace is a 185-kilometer relay race from Nijmegen to Enschede, divided into 25 stages (17 men's and 8 women's stages) varying from 3.4 km to 11.9 km. More than 7,500 students (300 teams) participate

in this race. In 1972, a couple of students from University of Nijmegen came back from the SOLA relay run in Sweden with so much enthusiasm, that they decided to organize a similar event in the Netherlands. These students inspired academics and they chose the route of the Batavians, who sailed down the Rhine in 50 B.C. The first race followed the original route to Rotterdam, but the route was later (from 1974 onwards) redirected to Enschede because of infrastructural problems.

The Membrane Runners gave a big effort and performed very well on all of the 25 stages. Two runners were disqualified because of a little disorganization in the night team, although they ran their

distances. Even with these two disqualified stages the Membrane Runners still placed 253rd. If this unlucky miscommunication had not occurred, we would have placed 140th, which is a big improvement compared to previous years. The Membrane Runners will keep running in coming years and continue to show their competences in a field outside their daily research.

After the race, the team's accomplishment and the support of the rest of the membrane scientists were rewarded with a barbecue at Louis van de Ham's place, followed by the biggest student party in Europe on the Twente University campus.

This event would not have been possible without the aid of our sponsors. The Membrane Runners would like to express their gratitude to Bernd Krause from Gambro Dialysatoren GmbH, Walter van der Meer from Vitens, Sybrand Metz from Wetsus (Research Institute for Sustainable Water Technology) and Zandrie Borneman and Antoine Kemperman from the European Membrane Institute

Introducing...

New MTG staff

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On May 1st, Nieck joined the Membrane Technology Group as an assistant professor. His research activities in Membrane & Interface Science and Inorganic Membranes will focus on the physics and chemistry of thin sophisticated films. His interests include multi-component transport, biomimetic membranes, size and confinement dependence of materials properties, and synthesis and characterization of sol-gel derived inorganic membranes for high pressure and temperature applications. Previously, Nieck was an assistant professor in the Process Development Group at the Eindhoven University of Technology, and he worked in the Membrane Technology Group of DSM Reseach in Geleen. Nieck is an alumnus of the University of Twente, where he studied chemical engineering and obtained his doctorate from the Inorganic Materials Science group.

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Membranes for Flue Gas Dehydration

Coal-fired power plants produce electricity, and also large volume flows of flue gas, which are close to saturation with water vapor. In order to prevent condensation, part of the water vapor needs to be removed before emission into the atmosphere. Experiments with highly permeable and selective membranes developed in our group together with Kema (consulting company) prove that membrane technology is a viable and potentially interesting option for removing this water vapor from the flue gases. In addition to the potential energy savings, the removed water can be reused in the power plant for power generation. Long-term field tests in a real flue gas stream of a power plant are very promising and prove the stability of the developed membranes: even after 5300 hours, there is hardly any loss in membrane performance or membrane stability. The concept developed here also offers promising possibilities for the removal of CO_2 from flue gases.

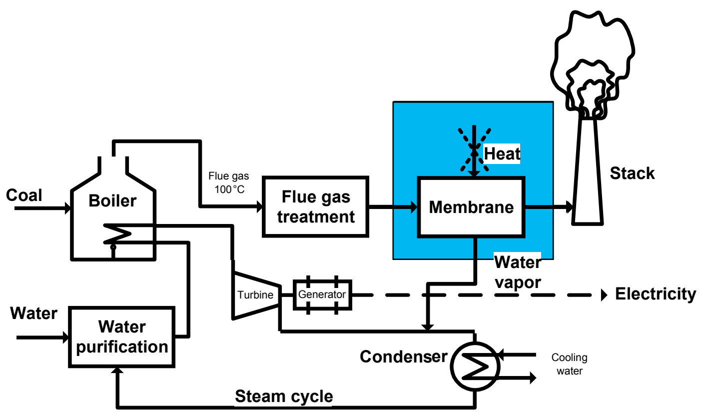

Figure 1: Process scheme of a power plant with a membrane unit for the dehydration of flue gases.

Flue gases from power plants mainly contain nitrogen, but also approximately 11 vol.% of water vapor. This seems only a small amount, but power plants produce tremendous amounts of flue gases (a relatively small power plant of 450 MW produces 1,500,000 m^3 of flue gas per hour), and thus emit large volume flows of water vapor as well. The flue gas stream that leaves the stack is almost fully saturated with water vapor. This can easily lead to condensation in the stack. Condensation causes corrosion problems due to locally high concentrations of trace amounts of aggressive components in the condensed droplets left in the flue gas after treatment. Currently, condensation has to be prevented by reheating the flue gas stream, resulting in extra energy consumption and additional costs. However, partial removal of the water vapor before emission would make reheating the flue gas stream redundant.

The application of membrane technology for this separation is attractive due to the additional energy savings and the possible reuse of water. The membrane unit can be positioned directly in the flue gas stream, after the flue gas treatment unit and before the stack, thereby replacing the reheater.

The membranes developed are composite hollow fiber membranes consisting of a porous hollow fiber support that

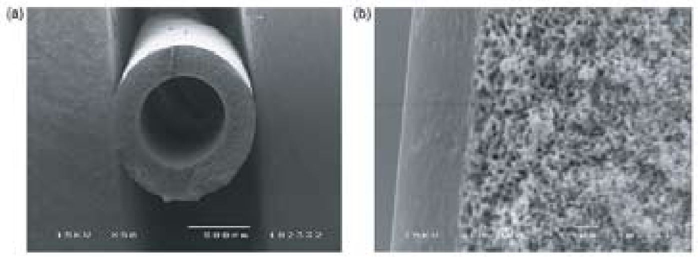

Figure 2: Cross-section of the composite hollow fibers developed (left: magnification 50×; right: magnification 10,000×). The dense separating layer of SPEEK is approximately 2 μm thick.

provides the mechanical stability and a dense separating layer of the effective membrane on top of that support. The effective membrane layer has a thickness of only 1 to 2 μm. Sulfonated poly (ether ether ketone) (SPEEK) is used as a selective membrane material. It combines a very high water vapor permeability with an extremely high water vapor over nitrogen selectivity. A vacuum on the inside of the hollow fibers provides the driving force for permeation. The vacuum's advantage is that water vapor easily condenses, which makes it attractive to use the overcapacity of the condenser system already present in the power plant. The membrane exhibits a very low permeability for the non-condensable gases in the flue gas (mainly nitrogen, about 70 vol.%), which results in extremely high selectivities.

Short and long-term testing of the membranes revealed good mass transport properties and resistance under flue gas conditions. Long-term field tests with the membranes directly positioned in the flue gas stream of a coal-fired power plant in Borssele (The Netherlands) prove that 0.2–0.5 liters of water

Figure 3: Each membrane module (left) consists of 19 hollow fibers, and 20 such modules are installed in the flue gas channel (right).

per m² of membrane area can be removed continuously during a period of over 5300 hours.

This study not only demonstrates the technical viability of flue gas dehydration, but also suggests that polymer membranes may be a viable option in directly removing CO_2 from flue gas. Within the large European project NanoGlowa, recently started in our group in collaboration with several membrane developers, membrane and module producers, end-users, and consultancy and diagnostic companies, we work on the development of suitable membranes and further exploration of this concept for the capture of CO_2.

For more information, please contact Dr. Kitty Nymeijer (d.c.nijmeijer@utwente.nl; phone: +31 (0)53 489 4185). The work 'Flue Gas Dehydration using Polymer Membranes' by Hylke Sybesma, Kitty Nymeijer, Rob van Marwijk, Rob Heijboer, Jens Potreck, and Matthias Wessling was published in the Journal of Membrane Sciences 313 (2008) 263–276.

Introducing...

Microporous Transition Metal Oxide Membranes for Gas Separation

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Martin Wolf was born in Schwäbisch Gmünd, Germany on November 9th, 1981. He finished his MSc in molecular science at the Friedrich-Alexander-University in Erlangen, Germany. He did his MSc thesis at the University of Wollongong, Australia on the topic: 'The Synthesis of 6,6′-biindoles as Potential New Anti-HIV Agents.'

Martin gained his experience in ceramics at Aaflow/Membraflow Filtersysteme GmbH (now GEA), and worked on various reasearch topics: flux measurements with different fluids through various ceramic filter elements and membranes; fabrication of different ceramic filter elements in all steps; and pressure stability experiments with these elements.

His PhD project focuses on synthesis and characterization of microporous membranes fabricated from transition metal oxides for gas separation membranes aiming for zero-emission fossil power plants. The targeted membranes should exhibit molecular sieving properties, and are to be employed in the separation of H_2 from CO_2, for example.

Membrane Characterization under Harsh Conditions

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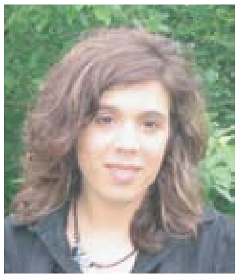
On June 1st, 2008, Hans joined the Membrane Technology Group as a PhD student. He did his MSc in chemical engineering and chemistry at the Eindhoven University of Technology. In September 2006 he started his PhD project at the Eindhoven University of Technology. He now continues his work on this project in Enschede.

The aim of his research project is to develop, characterize and apply stable membranes with properties tailored for harsh conditions, e.g. high pressure and temperature and extreme pH values.

Ceramic-Supported Polymer Nanofiltration Membranes



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Ana Pinheiro was born in Coimbra, Portugal on June 26th, 1982. She finished her chemistry degree in 2006 at the University of Coimbra, Portugal. During the last year of the degree she performed her curricular scientific training in the area of Catalysis and Organic Synthesis, which resulted in her dissertation thesis 'Synthesis of Porphyrins—Potential Oxygen Photosensitizes.' During the last year she worked in the Biomaterials, Biodegradables, and Biomimetics research group at the University of Minho, Portugal, on the 'Thermal Behavior of Biodegradable Polyesters for Medical Applications' project. She gained experience in thermal and mechanical characterization of several polyester films and polymeric samples along with synthesis of new polymeric materials.

In February 2008 she started her PhD in the Membrane Technology Group. The goal of her project is to develop ceramic-supported polymer nanofiltration membranes for bulk liquid applications under harsh conditions (extreme pH and/or solvent environments).

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The Membrane Technology Group

Multidisciplinary approach in membrane science and technology

The Membrane Technology Group focuses on the multi-disciplinary topic of membrane science and technology. We consider our expertise to be a multidisciplinary knowledge chain ranging from molecule to process. The knowledge chain comprises the following elements:

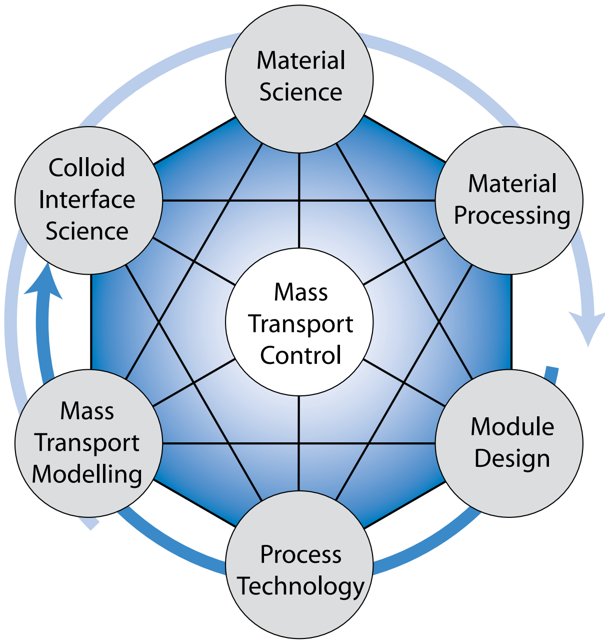
- Colloid and interface science
- Macroscopic mass transport characterization and modeling
- Material Science
- Material Processing
- Module and system design
- Process technology

The research team is assembled such that permanent staff members cover one or more of the disciplines involved.

The majority of the research deals with the separation of molecular mixtures and selective mass transport. Our research program is divided into four application clusters:

- Sustainable Membrane Processes
- Water
- Biomedical and Life Science
- Micro Systems Technology

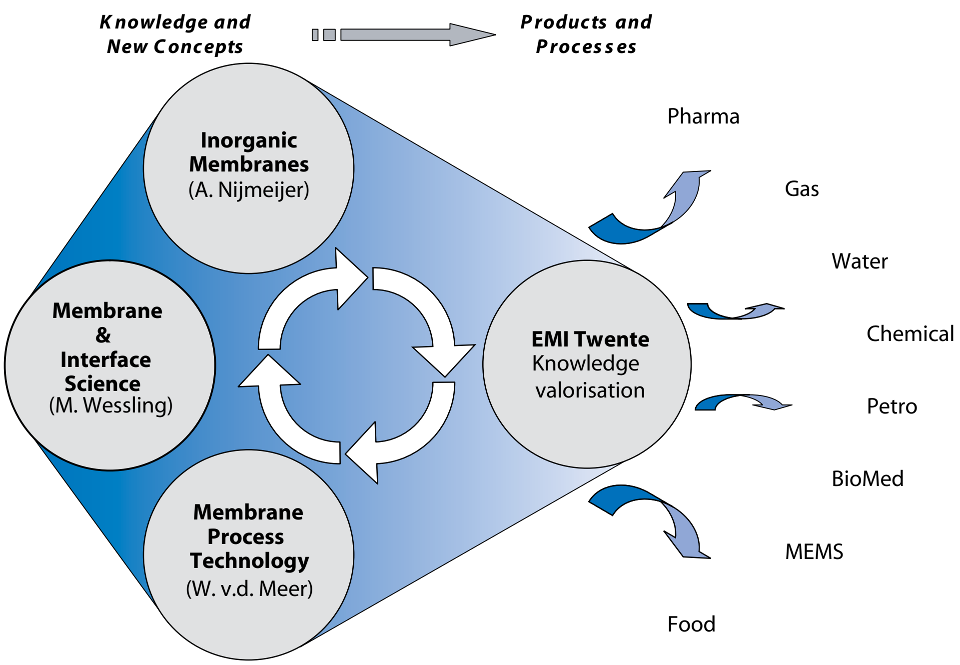
The research clusters are embedded in three research institutes, IMPACT (process technology), BMTI (biomedical) and MESA+ (nanotechnology).



Total mass transport control by a multidisciplinary approach. The Membrane Science and Technology group, together with the Membrane Process Technology and Inorganic Membranes groups, covers the full spectrum of mass transfer phenomena in membrane separation. From the very small scale to module and process design, the groups' combined knowledge can handle any membrane-related problem.

Knowledge transfer and utilization

In recent years, our group has focused especially on knowledge utilization and transfer. We have experienced that the time scales for research progress are quite different in industry and academia. Even within industry, there is a significant difference between the time-scales of small and medium enterprises and those of multinational cooperations. We have adjusted our organization structure such that we can distinguish between long-term scientific activities and short-term technology transfer. We have established the European Membrane Institute (EMI) Twente for this purpose. The EMI performs research and development work on new membrane products and processes. The work often focuses on the production of a tangible deliverable.



Bringing new knowledge and concepts to the market. The traditional discrepancy between the needs of the industry and the research performed within universities is bridged by the European Membrane Institute.

MNT Information

Membrane News Twente is published two times per year. Our aim is to inform the membrane community about the Membrane Technology Group's activities.

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