2011 winter

Membrane News Twente

News magazine of the Membrane Technology Group

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Membrane Science and Technology at UTwente

Vision

The research group Membrane Science and Technology of the University of Twente focuses on the multi-disciplinary topic of membrane science and technology to control mass transfer through interfaces (Figure 1).

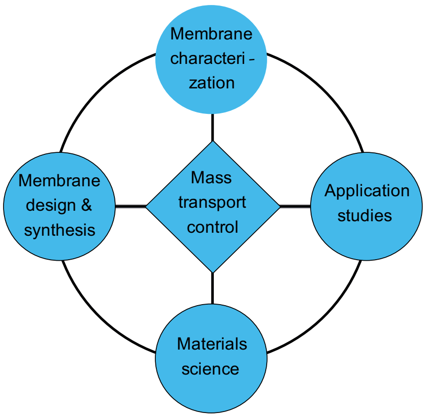


Figure 1 - Membrane Science & Technology.

Our research focuses on the separation of molecular mixtures and selective mass transport. We aim at tailoring membrane morphology and characteristics on a molecular level to control mass transport in macroscopic applications. We consider our expertise as a multidisciplinary knowledge chain ranging from molecule to process. We distinguish three application clusters, i.e. Energy, Water and Life Sciences (Figure 2).

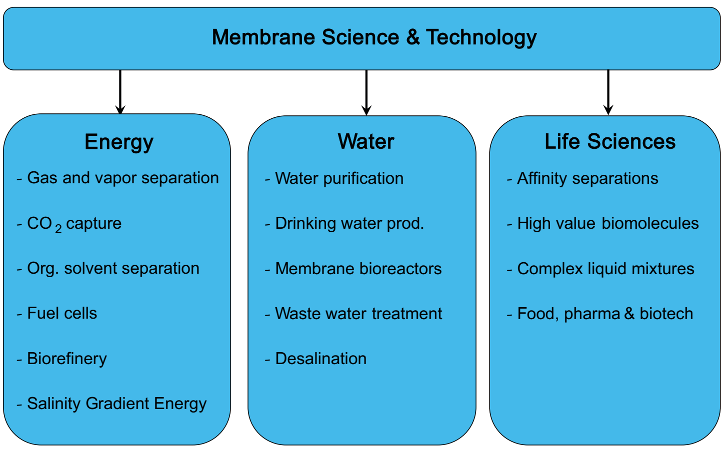


Figure 2 - Research themes Membrane Science & Technology.

Energy

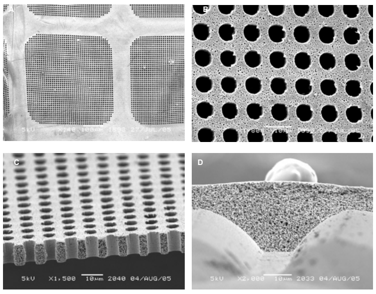
Research on Energy is dedicated to the molecular design and synthesis of polymer membranes for e.g. gas and vapor separations (CO_2 capture, olefin/paraffin separation, water vapor removal), biorefinery applications, fuel cells and the generation of energy from the mixing of salt and fresh water ('salinity gradient energy' or Blue Energy). Relevant research aspects are the control of structure-properties relationships, separation of complex, multi-component mixtures (binary, ternary systems, effect of impurities), interaction of the feed components with the membrane and performance evaluation.

Water

Within the application cluster Water, research addresses the development of membranes and the application of membrane technology for water treatment, e.g. water purification, desalination, membrane bioreactors and waste water treatment. In particular it investigates the relation between membrane design and membrane properties, spacer design and hydrodynamic conditions in relation to biofouling, fouling control and performance evaluation.

Life sciences

Within the application cluster Life Sciences, we focus on the design of porous membranes to separate complex multicomponent mixtures in pharmaceutical, food, beverage and biotech applications. Important subjects are the tuning of the material properties and structure (e.g. pore morphology and porosity), the development of functional materials (e.g. affinity separations of biomolecules) and the creation of new and/or improved processes (e.g. faster processes, higher yields,



less fouling, etc.). Also aspects related to process design and industrial implementation, such as scale-up of novel membrane fabrication methods, are investigated.

Knowledge valorization

Our group has decided to establish a significant effort in the valorization of its knowledge. The European Membrane Institute Twente (EMI Twente) was established in 1995 and performs confidential contract research directly with the industry. Students are not involved. Research is governed by questions out of industry. EMI Twente creates, transfers and translates (fundamental) scientific knowledge into products, processes and applications. Projects can last from only a few days up to several years. The EMI Twente acts as the interface between the academic research and the industrial needs.

Services:

The EMI Twente provides the following services:

- Membrane development
- Membrane characterization
- Desktop studies
- Consultancy
- Selling equipment (e.g. test cells for gas separation, UF and MF, casting knives, cloud point meters, hollow fiber spinning lines and spinnerets

More information

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MNT- Information

Membrane News Twente is published two times per year and aims to inform the membrane community about the activities of the Membrane Technology Group of the University of Twente.

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Gordon Research Conference

The preliminary program is available for the
Gordon Research Conference, Membranes: Materials & Processes

July 29 - August 3, 2012, Colby-Sawyer College, New London, NH, USA
http://www.grc.org/programs.aspx?year=2012&program=membmat
A more detailed program will be published soon in Science magazine.

This Gordon Research Conference apires to encourage communication and discussion of ideas and new unpublished results at the very frontier of a Membrane Science and Technology. The conference is officially "off-the-record", with no abstracts or proceedings published before, during, or after the conference, and ample time is provided between sessions for socializing and informal discussions with fellow attendees. A limited number of participants can still apply.

Solvent resistant nanofiltration membranes

The beginning of nanofiltration (NF), a membrane process capable of efficient separation of molecules in the range of 200-1000 g mol^{-1}, dates back to the late 1950s. Over the years NF proved useful in many applications such as water softening, removal of pesticides and micro-pollutants from ground water, treatment of textile wastewater, virus and bacteria removal, decontamination and recycling of industrial wastewater, as feed pretreatment for desalination and removal of heavy metal ions from ground water. The success of NF in aqueous systems has triggered expansion to organic solvents. In fact, in the late 1990s a new spin-off of NF, solvent resistant nanofiltration (SRNF) emerged. Low energy consumption, compared to traditional techniques like distillation, and ease of combining with already existing processes made SRNF particularly attractive for industrial applications. However, the use of the first generation of NF membranes, designed almost exclusively for aqueous systems, presented a lot of difficulties in organic solvents. Due to excessive swelling or even dissolution of membrane forming material, loss of selectivity was often observed. An urgent need for better membranes for SRNF arose. In this thesis the development of novel membranes, with target molecular weight cut-off lower than 500 g mol^{-1}, for organic solvent filtration (OSF) was presented. New membranes were prepared by either coating a PDMS layer on a ceramic capillary or hollow fiber support (I), or diisocyanate crosslinking of commercial polyamide-imide flat nanofiltration (NF) membranes (III) or spinning of polyimide hollow fibers (HF) using chemistry in a spinneret concept (IV). In all chapters, the membranes were characterized by solvent permeation and molecular weight cut-off (MWCO) studies. The importance of the careful selection of the solute and process conditions for the MWCO determination of the newly developed membranes is highlighted.

Objectives and scope of the project: The main aim was to develop membranes for solvent resistant nanofiltration (SRNF) with molecular weight cut-off below 500 g mol^{-1}. Several concepts to obtain SRNF membranes have been explored.

I. Capillary α-alumina / poly(dimethylsiloxane) (PDMS) composite membrane

As a support, a commercial α-alumina capillary, with pore size of 800 nm on the outside and 20 nm on the inside, was used. The best composite capillary membrane was prepared by coating on the inside of the capillary (Fig.1). This membrane has a 7 µm PDMS selective layer, stable performance for over 40 h in toluene, a toluene permeance of 1.6 l m^{-2}h^{-1}bar^{-1} and a MWCO of 500 g mol^{-1} (Fig.2).

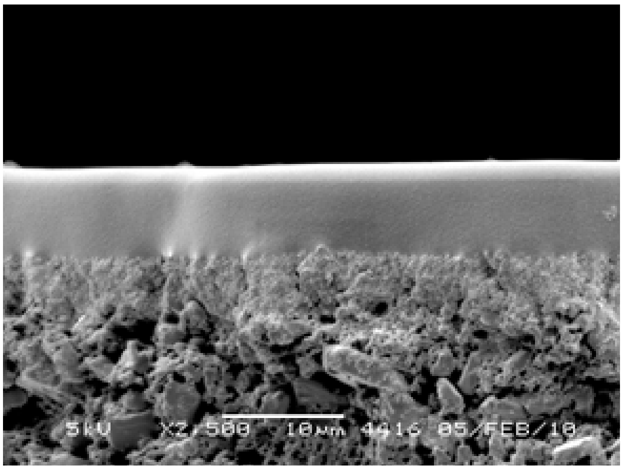

Figure 1 - (a) α-alumina / PDMS (M20/55) composite capillary membranes.

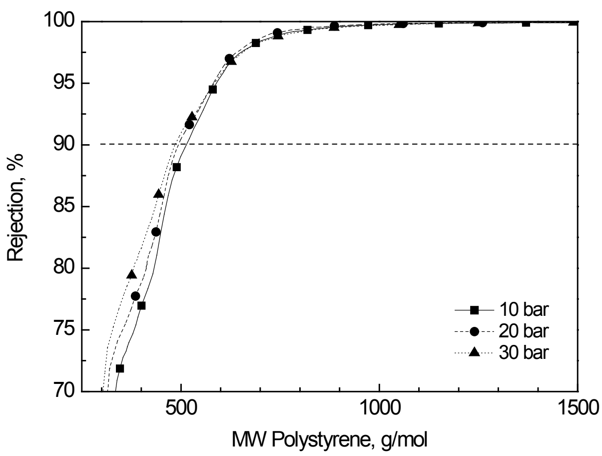

Figure 2 - PS oligomer retention by the M20/55 membrane.

Despite the relatively thick PDMS layer, this membrane has comparable toluene permeances as various laboratory PAN/PDMS membranes with thinner PDMS layers.

Further development of the composite membrane should focus on coating a PDMS layer on tailor made ceramic hollow fibers that would further improve the surface to volume ratio of the resulting module.

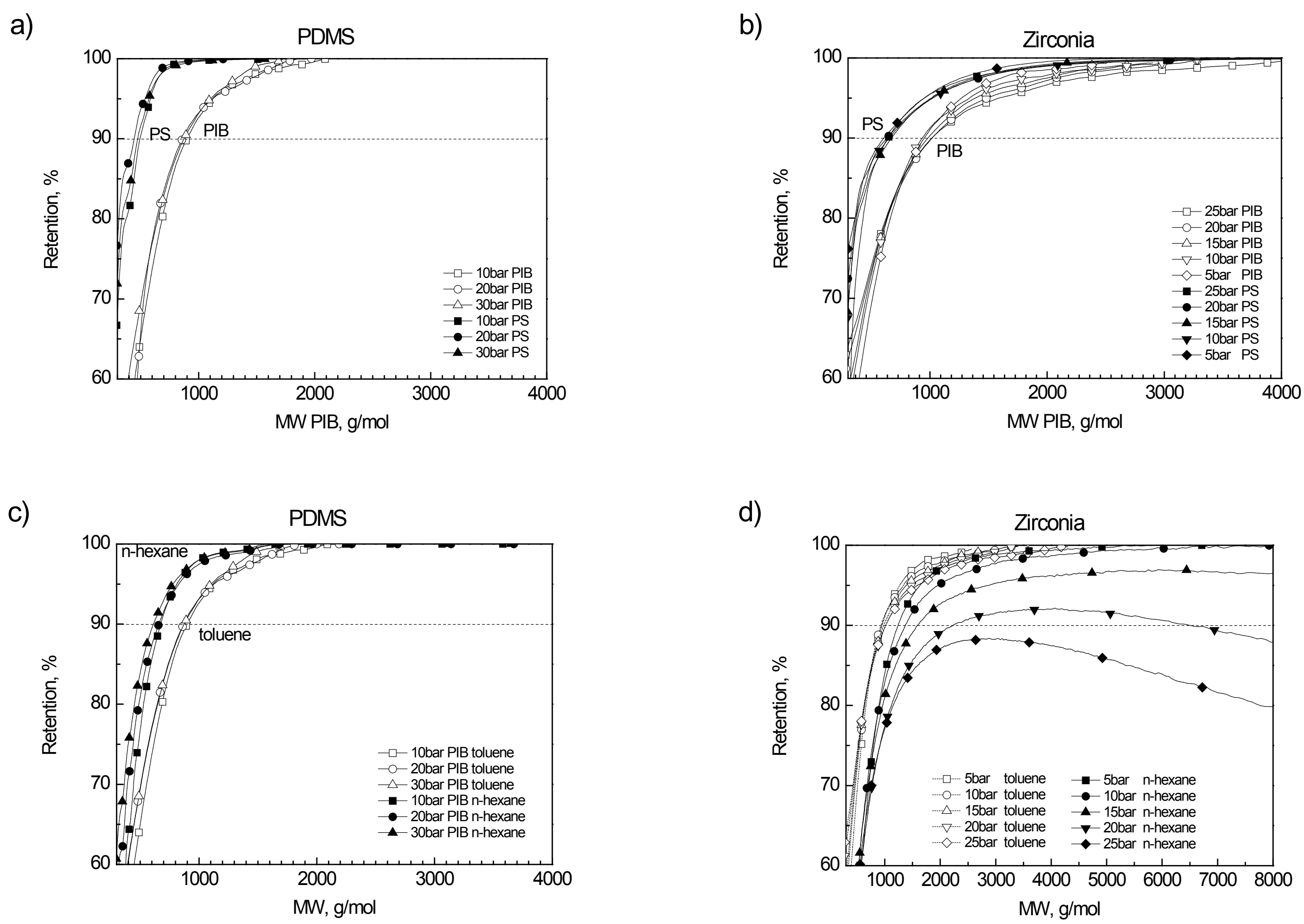

Figure 3 - Transport properties of the zirconia and PDMS membranes: Retention curves of PIB and PS in toluene for: (a) PDMS membrane (b) zirconia membrane. Retention curves of PIB in toluene and n-hexane for: (c) PDMS membrane (d) zirconia membrane. In Figure 3 the depicted symbols present an optical guide to distinguish between the experimental curves derived from the GPC analysis.

II. Important factors influencing molecular weight cut-off determination of membranes in organic solvents

We investigated the effects of solvent, solute, membrane properties, and applied process conditions on molecular weight cut off (MWCO) characterization. For this study a rigid porous membrane (hydrophobized zirconia) and a rubbery dense membrane (α-alumina/PDMS composite) were used. The behaviour of two solutes, a stiff polystyrene and a flexible polyisobutylene was studied in rather low (toluene) and high flux (n-hexane) solvents (Fig. 3).

Our results demonstrate that the α-alumina/PDMS composite membranes show only very little effect of flux on retention and the membrane's MWCO is predominantly affected by solute-membrane and solvent-membrane interactions (Fig. 3a and c). In case of a rigid porous zirconia membrane, a significant effect of applied pressure was observed (Fig. 3b), in particular for the flexible solute polyisobutylene (PIB) (Fig. 3d). The variations in MWCO with pressure indicated combined effects of concentration polarization and shear-induced deformation of the flexible solute. Our results clearly show that the selection of the proper solvent - solute system and the process conditions for the retention measurements as well as the interpretation of MWCO data in organic solvents require great caution.

III. A novel polyamide-imide membrane for applications in harsh polar aprotic solvents

A novel polyamide-imide membrane has been prepared by crosslinking of flat polyamide-imide (Torlon®) based membranes using hexamethylene di-isocyanate (HMDI) (Fig. 4). The crosslinked membrane is stable in N-methyl pyrrolidone, which is a solvent for the non-crosslinked membrane, has an acetone permeance of 1.2 ± 0.1, l $m^{-2}h^{-1}bar^{-1}$ and a MWCO about 300 g mol^{-1}.

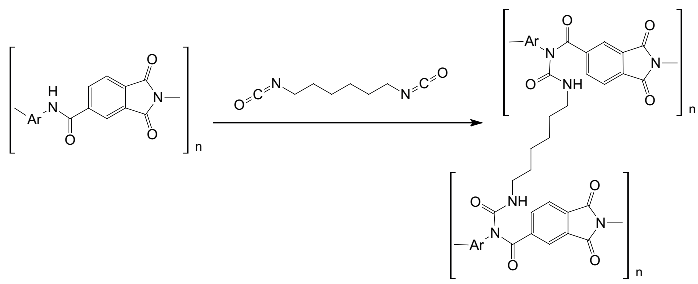

Figure 4 - Crosslinking of polyamide-imide by HMDI.

Due to the creation of amide bonds, the HMDI crosslinked membranes are expected to be thermally stable enabling separation at elevated temperature. Besides, it seems possible to

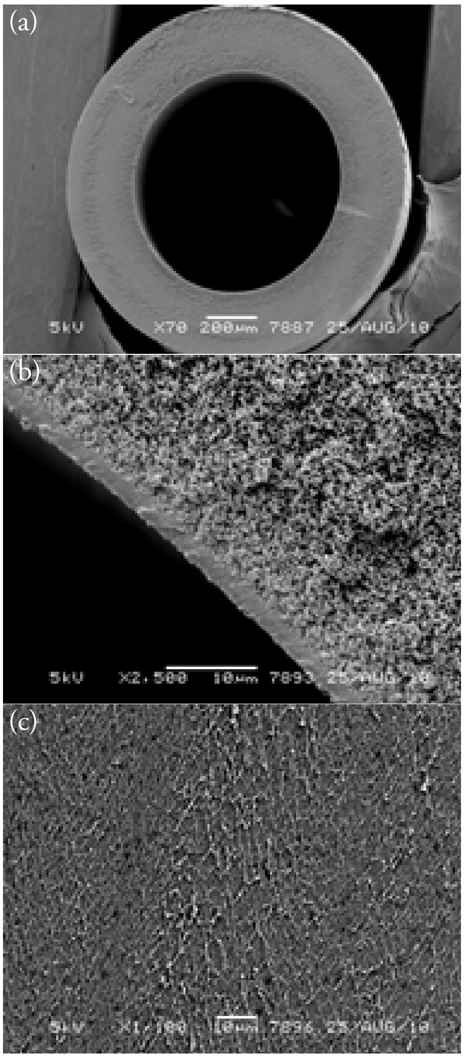

Figure 5 - SEM images of a typical hollow fibre membrane obtained by "chemistry in a spinneret" spinning method: (a) cross section, (b) cross section of the inner selective layer, (c) surface of inner selective layer.

tailor the MWCO of the membrane by varying the crosslinking time: longer crosslinking time corresponds to tighter (lower MWCO) membranes.

IV. Polyimide based hollow fibres prepared by new spinning method "chemistry in a spinneret"

This method integrates the membrane formation and crosslinking reaction into a single step to fabricate a membrane for SRNF (Fig. 5). The membranes studied were based on P84 (Evonik) polyimide as a membrane forming polymer while the crosslinker poly(ethylene imine) (PEI) was dissolved in the bore liquid. The prepared fibres have a MWCO between 2500 – 3500 g mol-1 and toluene permeances in the range of 0.2 - 1.1 $l\ m^{-2}\ h^{-1}\ bar^{-1}$.

Due to crosslinking, the membranes became more hydrophilic resulting in high ethanol/PEG permeances (14.6-17.2 $l\ m^{-2}\ h^{-1}\ bar^{-1}$) and low rejection of PEG (14-23 % of 4000 $g\ mol^{-1}$). The most stable membrane lost 20% of its mass after 11 days after immersion in NMP. Future research in this field should focus on the development and characterization of P84/PEI membranes for polar organic solvents.

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NPS11 Oral presentation award for Olga Kattan

At NPS11 (11th Annual Netherlands Process Technology Symposium), the biggest symposium in The Netherlands for engineers and scientists active in the field of process engineering, held in October 2011, Olga Kattan won the presentation award for her oral presentation. The prize consisted of 2500 Euro to be spent for an international conference. Olga Kattan's presentation was entitled "Isolation of single amino acids for biorefinery applications using electrodialysis". In her talk she emphasized the importance of switching from conventional refinery to biorefinery concepts and introduced the integration of enzymatic conversion and membrane electrodialysis as an interesting route for the production of biobased chemicals. She discussed the importance and challenges of controlling specific process parameters, among which especially pH, for improved process performance. The results show that electrodialysis with commercial ion exchange membranes is a promising technology that leads to pure product streams to be used for the production of biochemicals.

For more information, please contact Dr. Kitty Nijmeijer (d.c.nijmeijer@utwente.nl; phone: +31 53 489 4185).

ICOM 2011 presentation prize Olga Kattan and David Vermaas

At the International Congress on Membranes and Membrane Processes, the world's largest conference on fundamental and applied membrane science, engineering and technology, held in Amsterdam, The Netherlands in July 2011, Olga Kattan and David Vermaas won a student award for their oral presentations. Four of the 6 oral presentation awards were sponsored by the European Membrane Society (EMS) while the other 2 were sponsored by the Australasian Membrane Society (AMS). Besides the compliment of winning an award, the prizes consisted of a certificate and either 500 euro or 500 Australian dollars, respectively.

Olga Kattan's presentation was entitled "Membranes in biorefinery applications". During her talk, she pointed out the need of switching from conventional refinery to biorefinery and discussed the applicability of electrodialysis with commercial ion exchange membranes for the isolation of single amino acids to be used for the production of biochemicals.

David Vermaas presented new developments in the energy generation by Reverse Electrodialysis. He showed in this presentation that the power density obtained from mixing seawater and river water can be significantly improved by using profiled membranes and using small intermembrane distances.

For more information, please contact Dr. Kitty Nijmeijer (d.c.nijmeijer@utwente.nl; phone: +31 53 489 4185).

Ph.D. Vacancy

Within the Membrane Science & Technology group (www.membrane.nl) of the University of Twente and in close collaboration with Wageningen University and Wetsus, we have a vacancy for a Ph.D. position on:

Membrane technology for oil – water separations

Recent developments in oil production technologies emphasize the application of Improved Oil Recovery (IOR) and Enhanced Oil Recovery (EOR) technologies. These processes rely on the injection of aqueous liquids into an oil reservoir. Current practice for offshore applications is that seawater is used as source water. Often, seawater shall be desalinated to meet the injection specifications. As an alternative, produced water can be treated for reinjection. In order to allow the reinjection of produced water, pretreatment and purification of the water is necessary. This project investigates both pre-treatment requirements, membrane fouling and development of new membranes for produced water treatment. The effect of residual oils, production chemicals (scale and corrosion controlling chemicals) and biocides on membrane performance will be evaluated. The purpose of this project is to identify, develop and demonstrate methods to pretreat and purify the produced water for reinjection. Next to artificial feed waters, produced water from oil production locations will be used in laboratory experiments.

The research will be performed at Wetsus, Centre of Excellence for Sustainable Water Technology in Leeuwarden, The Netherlands (www.wetsus.nl). We are looking for highly motivated and enthusiastic researchers with an MSc degree in chemistry, chemical engineering, material science or a related topic, with adequate experimental and theoretical skills. We prefer candidates with a good team spirit, who like to work in an internationally oriented environment. Fluency in English is a requirement. An interview and a scientific presentation will be part of the selection procedure.

Interested candidates can send their motivation letter, CV (including references) and list of courses and grades to Dr. Kitty Nijmeijer (d.c.nijmeijer@utwente.nl; phone: +31 53 489 4185).

Thin film composite nanofiltration membranes for extreme conditions

Typically, nanofiltration (NF) involves the separation of monovalent and divalent salts, or organic solutes with molecular weight in the range of 200 to 1000 g mol-1. Although most commercial NF membranes are suitable to treat aqueous streams at pH levels between 2 and 10, many potential applications in the chemical industry involve much more aggressive conditions. Advances in the development of stable membranes and their performance characterization in these harsh applications will therefore expand the application window of such membranes in commercial processes.

It has been widely reported that the feed pH during NF has a prominent effect on the separation performance. Thus apart from extensive stability tests at relevant pH, a comprehensive evaluation of the membrane performance as a function of pH is essential. Accordingly a new method that allows molecular weight cut off (MWCO) characterization of NF membranes as a function of pH is developed (Figure 1).

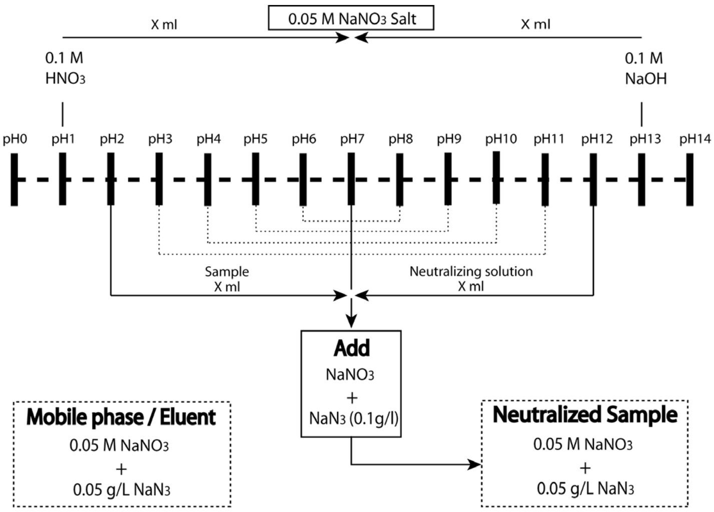

Figure 1 - Protocol for modification of GPC samples obtained at different pH. A sample of X ml with pH = y is neutralized with X ml of liquid with pH = 14 - y. For samples with 1<pH<13 $NaNO_3$ is added to achieve a concentration of 0.05 M.

Such a method is complicated by the variation in the pH of the samples, leading to different ion contents. However by appropriate modification of the ion content of GPC samples obtained at various pH, changes in membrane performance with respect to pH can be monitored quantitatively. Performance evaluation of a well-known commercial NF membrane (NF-270, Dow Filmtec™) via this method reveals reversible changes with respect to pH, signifying the relevance of performance characterization at the relevant conditions.

The above-mentioned method was also used to illustrate the effect of pH on the performance of in-house developed polyamide thin film composite (TFC) NF membranes, fabricated via the interfacial polymerization (IP) route using the experimental set-up shown in Figure 2.

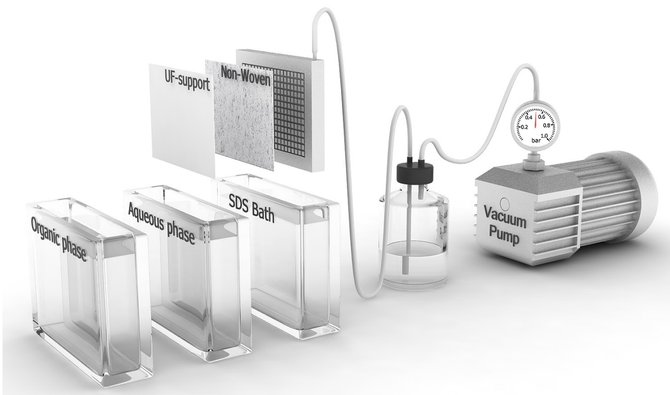

Figure 2 - Schematic of composite membrane fabrication setup.

Using the Donnan steric partitioning pore model (DSPM), the pH induced performance changes have been correlated to morphological changes in the membrane matrix, i.e., changes in the effective average pore size and effective porosity of the membrane (Figure 3).

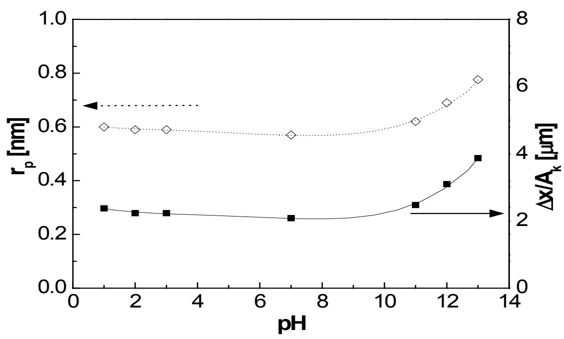

Figure 3 - Effect of pH on the pore size and thickness/porosity ratio rp and Δx/Ak as a function pH of the feed via DSPM.

Of all the membranes investigated in this work, sulfonated poly (ether ether ketone) based TFC's showed the highest pH stability. The developed membranes reveal excellent stability in the entire pH range from 0 to 14, even under prolonged exposure (Figure 4). Similar to commercial and polyamide membranes, performance evaluation at varying pH revealed

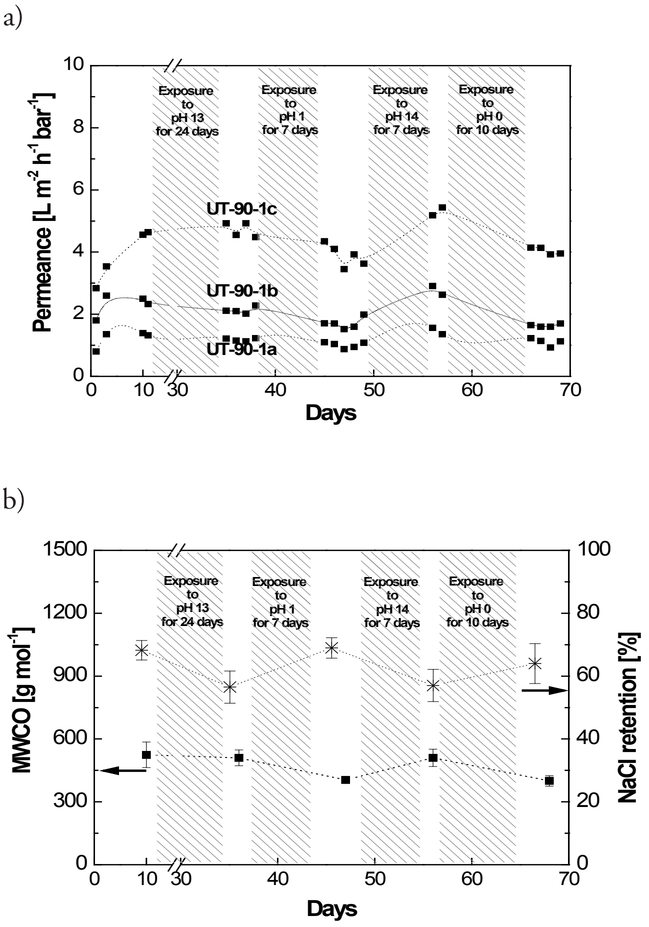

Figure 4 - Long term stability tests on SPEEK membranes. Effect of exposure on a) clean water permeance and b) MWCO and NaCl retention at 10 bar trans-membrane pressure.

reversible changes in the SPEEK membrane properties.

Finally novel hybrid TFC membranes containing polyhedral oligomeric silsesquioxanes (POSS) have been prepared using interfacial polymerization. Both free standing as well as substrate supported films could be obtained. In depth analysis to confirm the chemistry was carried out via Fourier transform infrared spectrometry and X-ray photoelectron spectroscopy. NF permeation experiments were performed on the substrate-supported films and several fabrication parameters were optimized. The presented strategy for film formation can easily be extended to other organic reactants, and to silsesquioxanes decorated with a variety of functional groups. This allows development of a new generation of quasi 2-D hybrid macromolecular networks with amendable chain conformation, and tailored chemical affinity via appropriate choice of the silsesquioxane functional groups.

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New people

Filtration behavior of nanoparticles

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Krzysztof Trzaskus graduated from Chemical Engineering in September 2011 with a binational Polish-German Master degree. During the Master diploma internship at Gambro Dialysatoren GmbH (Hechingen, Germany) he was working on the improvement of antifouling properties of polymeric hollow fiber membranes. The title of his final Master Thesis was “Hydrophilic Modification of Polymeric Membranes”. In October 2011, Krzysztof Trzaskus started his Ph.D. in the group Membrane Science & Technology of the University of Twente. His research project focuses on the filtration behavior of manufactured nanoparticles during filtration and on the analysis of the interactions between these particles, other foulants and the membrane.

Separation of equally charged metal ions from water

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Sinem Tas received her B.Sc. Chemical Engineering degree from Hacettepe University, Ankara, Turkey in 2008. After that, she completed the Material Science and Engineering master program offered at Sabanci University, Istanbul, Turkey. For her Master assignment, she worked on the production of carbon nanotubes and fibers on the transition metal loaded FSM-16 (Folded Sheet Mesoporous Materials) under the supervision of Prof. Dr. Yuda Yurum. During her master studies, she also worked on a project about the synthesis and characterization of novel radiation-grafted membranes for high temperature polymer electrolyte membrane fuel cells under the supervision of Associate Prof. Dr. Selmiye Alkan Gürsel. In August 2011, Sinem started her PhD study in the Membrane Science and Technology Group of the University of Twente. Her project focuses on the separation of equally charged metal ions from water using ion exchange membranes combined with spectroscopic analysis.

Bio-inspired nanoporous layers for molecular separations

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Erik Vriezekolk was born in 1984 in Warnsveld, The Netherlands. He started his study Chemical Engineering at the Saxion Hogeschool in 2003 and graduated in 2007. After graduation he worked for ½ year at the R&D Surface Chemistry department at Akzo Nobel Deventer. In 2008 he started his master study Chemical Engineering at the University of Twente, and obtained his master degree in 2011. The title of his final project was "Mixed matrix membranes for process intensification of HMF removal from aqueous solutions". In October 2011 Erik Vriezekolk started his PhD study in the group Membrane Science and Technology of the University of Twente. The aim of his project is the fabrication of bio-inspired nanoporous layers for performing molecular separations such as desalination and disinfection.

Polymer-Molecular Organic Framework (MOF) membrane architectures for gas separation

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Salman Shahid was born in Pakistan and accomplished in 2007 his B.Sc degree in Chemical Engineering at the University of Engineering and Technology, Lahore – Pakistan. After his B.Sc. degree he continued to work as researcher at the same university at the department of polymer and process engineering and started his Master in Polymer and Process Engineering. He achieved his M.Sc degree in November 2009 with a thesis on "Investigation of the characteristics of thermo-mechanical polystyrene-unsaturated polyester reactive blends". After completing his M.Sc. degree, Salman Shahid got an Erasmus Mundus Master scholarship in engineering rheology in 2009 with a split Master degree from the Université Catholique de Louvain, Belgium (Master in Chemical and Materials Engineering with Professional Focus) and the University of Minho, Portugal. During this master he worked on a project entitled "Rheology as a probe to assess the degradation of EPM rubber". In November 2011, Salman Shahid started his PhD in the Membrane Science and Technology Group of the University of Twente under the Erasmus Mundus Doctorate in Membrane Engineering (EUDIME) Programme. The focus of this research is to develop novel polymer-MOF (metal organic framework) membranes for gas separation. The project will combine the academic expertise of different universities, i.e. the University of Twente (Netherlands), the KU Leuven (Belgium) and the University of Montpellier (France).

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