

# 2010 Membrane News Twente summer



News magazine of the Membrane Technology Group

MNT, p/a University of Twente - TNW/MTO  
postbox 217, NL-7500 AE Enschede, Netherlands  
membrane@utwente.nl - <http://mtg.tnw.utwente.nl>  
Telephone: +31 (0) 53 489 2950 - Fax: +31 (0) 53 489 4611

## NanoMemCourse University of Twente – The place to be

From the 7th of April until the 16th, 2010, the Membrane Technology Group of the University of Twente was the “place to be” for young membrane scientists from all over the world. During that period, the group organized a Marie Curie training course (NanoMemCourse) for young researchers and engineers (mainly PhD students and some Post docs) with background in material science and/or membrane science whose research was closely related to health and sustainable water applications.

The Membrane Technology Group hosted 56 participants mainly working in European countries but also from overseas (from distant places such as United States of America, Australia and Singapore). The lectures were carefully chosen accordingly to the topic and 23 international top experts in the different fields delivered exciting talks about their current research.

The NanoMemCourse program covered basic and advanced courses on nanomaterials and membranes for health and sustainable water applications as well as perspectives in research, industrial and societal issues, complimentary skills etc. Besides following lectures, the participants had the opportunity to exercise their scientific innovativeness and networking skills in several activities proposed by the organizing committee.

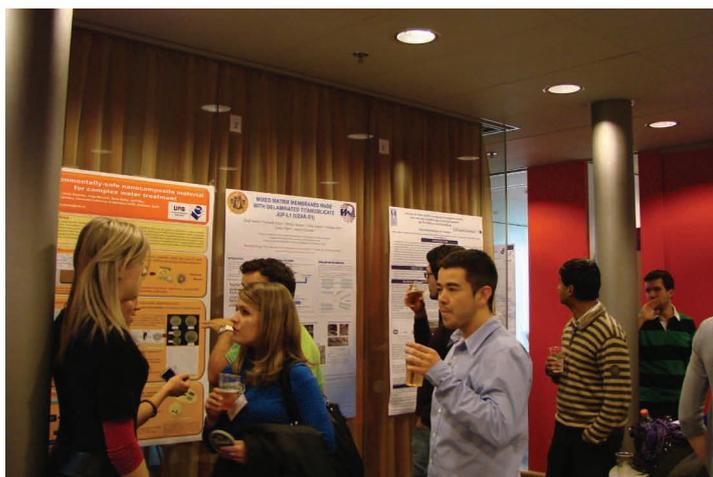
Two poster sessions (Health and Water) were organized and the participants had the chance to show their current research and exchange ideas while enjoying some snacks and drinks. This

event was set on the facilities of the Membrane Technology Group and it allowed all the participants to have a tour and visit to the laboratories of the group. All posters were evaluated by a committee and two winners were announced: Lauren Greenlee



(NIST, USA) with the poster “Development of bimetallic nanoparticles for novel membrane design and water treatment” for the Water poster session; and Zongli Xie (CSIRO Materials Science & Engineering, AUS) with the poster “Sol-gel Derived Pol(vinyl alcohol)/Silica Hybrid Membrane- Synthesis and Characterisation” for the Health poster session.

A research proposal competition was also organized where the participants (working in groups) presented their ideas for



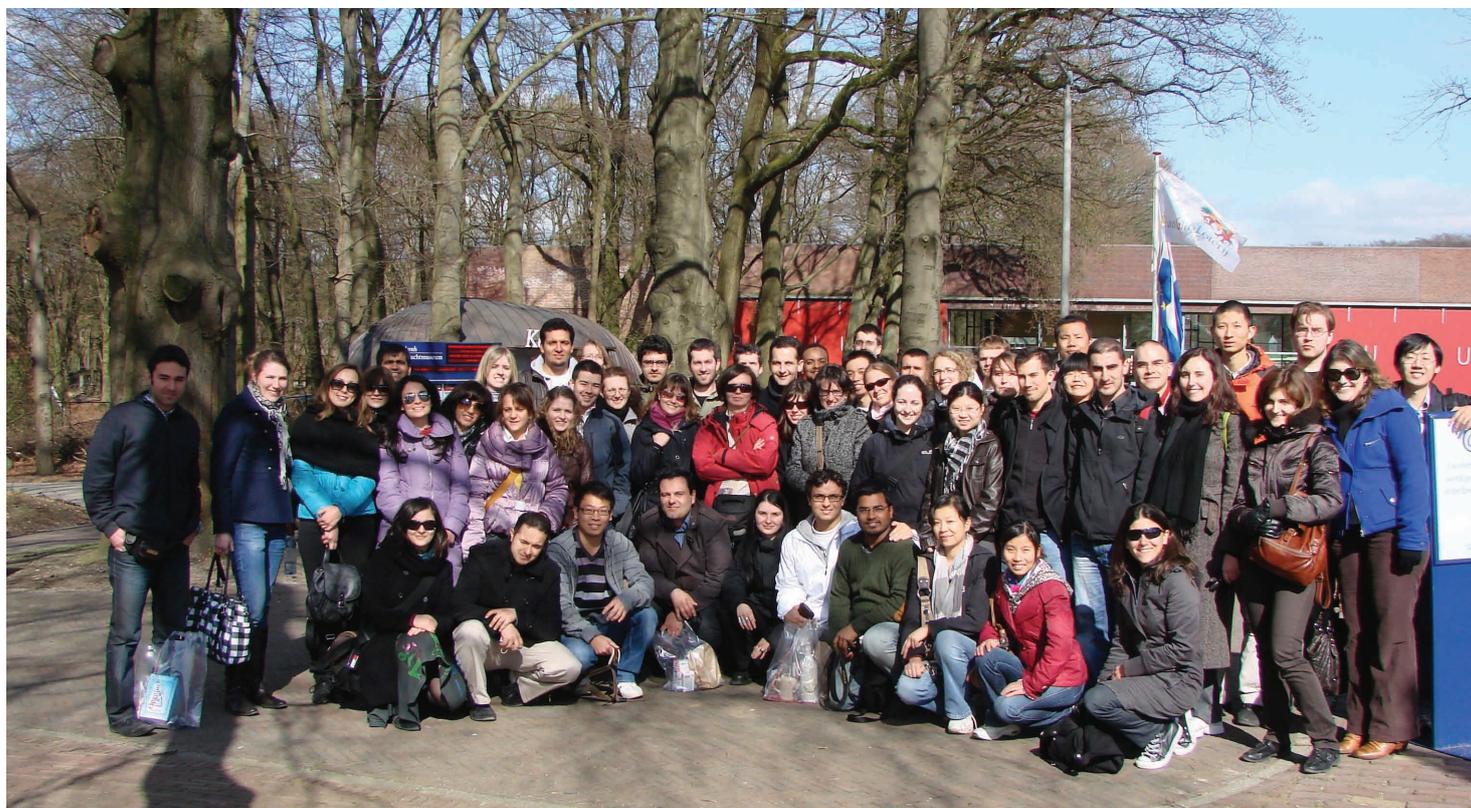
exciting future membrane research. The teams first presented their ideas to other participants and to an evaluation committee and then answered questions. The winning proposal was “Novel approach to real time fouling detection in membranes” proposed by the team: Cesar Rubio (Zaragoza University, Spain), Lauren Greenlee (NIST, USA), Massoud El Jastimi (Oviedo University, Spain), Ewa Stodolak (AGH, Poland), Antonella Piscioneri (ITM/CNR, Italy), Gyorgy Szekely (Hovione FarmaCiencia, Portugal), Majid Warkiani (Nanyang Technological University,

Singapore) and Sina Bonyadi ( Cambridge University, UK). After the competition, all participants were able to join an outdoor BBQ with music where they enjoyed the nice weather and lovely food!

Besides the lectures and poster sessions, the participants had the possibility to learn more about the Dutch culture with a visit the Open Air Museum in Arnhem on a sunny day. To discover the industrial environment in the region, a visit to the local beer factory Grolsch was also organized.



The organizing committee, Dr. Antoine Kemperman, Marlon Tijink, Dr. Dimitris Stamatialis and Dr. Sandra Teixeira did their best to offer the participants a stimulating scientific program combined with a friendly social program to ensure a pleasant atmosphere.



*This NanoMemCourse on Health and Water was the 4th course in a series of 5. NanoMemCourse is a joint initiative of ITM-CNR (Italy, Dr. Lidiatta Giorno), CNRS (Toulouse, Dr. Pierre Aimar), Unizar (Spain, Prof. Miguel Menendez), Forth (Greece, Dr. Vasilis Burganos), Sintef (Norway, Dr. Rune Bredesen), and the University of Twente (The Netherlands, Dr. Dimitris Stamatialis, Dr. Kitty Nijmeijer). NanoMemCourse is funded by the European Union as a Marie Curie conference and training courses action. The topic of the 5th course is ‘Food Processing’ and is organized by ITM-CNR, Italy. More information can be found at: [www.nanomemcourse.eu](http://www.nanomemcourse.eu).*

# Polyether based block copolymer membranes for CO<sub>2</sub> separation



The removal of CO<sub>2</sub> from light gas mixtures such as H<sub>2</sub>, N<sub>2</sub> and CH<sub>4</sub> is an important application in industry, for instance in synthesis gas, flue gas and natural gas processing. In particular the removal of CO<sub>2</sub> from flue gas has attracted a significant amount of attention, as the energy sector is with 50% by far the largest contributor to the worldwide emission of CO<sub>2</sub>. Carbon dioxide capture and subsequent storage (also known as CCS) from these large point sources thus poses a significant potential for the worldwide reduction of CO<sub>2</sub> emissions and the mitigation of climate change.

Several different options exist to separate the CO<sub>2</sub>, which are known as pre-combustion, post-combustion and oxy-combustion. Although the pre-combustion technique has significant potential there is an even more urgent need for post-combustion CO<sub>2</sub> capture systems as a retrofit option for the large fleet of currently existing coal-fired power plants. Post-combustion flue gas streams typically have a temperature around 50-60°C and are generally high in volume due to the 79% nitrogen in air. Furthermore, they have a low pressure (atmospheric) and the concentration of CO<sub>2</sub> is usually low (10-15 vol.%). The large volume, low pressure and very dilute stream makes post-combustion CO<sub>2</sub> capture a huge challenge. As a result of the low driving force a prerequisite for any membrane material to be considered for this separation is the necessity of an extremely high permeability for CO<sub>2</sub>.

Although some commercially available materials exist that have sufficiently high CO<sub>2</sub>/N<sub>2</sub> selectivity (40-50) for this separation, like the commercially available poly(ethylene oxide) (PEO) based block copolymers PEBA<sup>®</sup> and Polyactive<sup>®</sup>, their relative low permeability remains an issue. As a result CO<sub>2</sub> selective membranes with intrinsically higher permeability are required. To achieve this two different strategies have been developed to prepare PEO based block copolymer membranes with five times improved CO<sub>2</sub> permeability characteristics.



Figure 1 - Schematic representation of a block copolymer with rigid hard segments (red) and flexible soft segments (black).

## Strategy 1

In general, block copolymers contain a phase separated morphology in which the hard segments (usually polyamides,

polyurethanes or polyimides) provide mechanical stability and only the soft segments are able to allow the permeation of gases and thus control the gas transport properties (Figure 1).

The first strategy focused on the improvement (simplification) of the microdomain morphology of block copolymer systems by tailoring its specific design. This is essential as the microdomain morphology of commercially available block copolymer systems (such as the PEBA<sup>®</sup> family) is very complex (Figure 2a). The phase separation between the hard and soft segments is poor

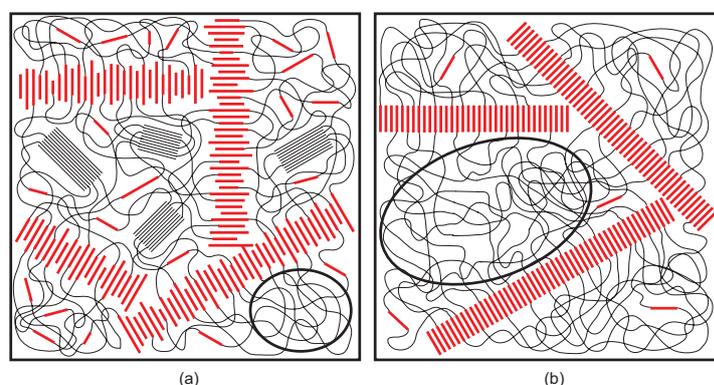


Figure 2 - Schematic representation of the morphology of (a) commercially available block copolymers (e.g. the PEBA<sup>®</sup> family) and (b) block copolymers as developed by strategy 1.

and the resulting complexity negatively influences the mass transport properties, as mass transport only takes place through the amorphous soft domains of the block copolymer (indicated by circled area). The microdomain morphology can to a very large extent be simplified by smart design of the block copolymer system by tailoring the molecular design of the hard and soft segments used. This simplified morphology resulted in a very pure amorphous soft domain available for gas transport (Figure 2b, circled area) and as a consequence significantly enhanced overall mass transport characteristics (Figure 4).

## Strategy 2

The second strategy that proved to be successful as a way to improve the CO<sub>2</sub> permeability of commercially available block copolymers, in particular the block copolymer PEBA<sup>®</sup>1657, is the addition of smart polymeric additives to this matrix polymer and obtain so-called 'blend membranes'. In our case, we used an additive based on poly(dimethyl siloxane) (for high permeability) and poly(ethylene oxide) (for high selectivity

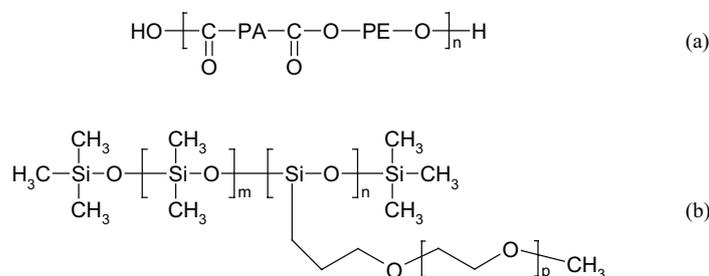


Figure 3 - Chemical structure of (a) the PEBA<sup>x</sup>1657 block copolymer and (b) the smart additive containing 80 wt.% PEO and 20 wt.% PDMS.

and blending compatibility) (Figure 3). The CO<sub>2</sub> permeability could be increased up to 5 times its original value, while CO<sub>2</sub>/N<sub>2</sub> selectivity only slightly decreased (Figure 4). Although only one specific additive has been tested, the range of potentially interesting polymers and additives is much larger. Furthermore, these blend membranes show great promise due to their easy preparation technique.

The successful development of these highly permeable and selective membranes makes a membrane based process a viable alternative for carbon dioxide capture and storage from flue gas.

## Literature

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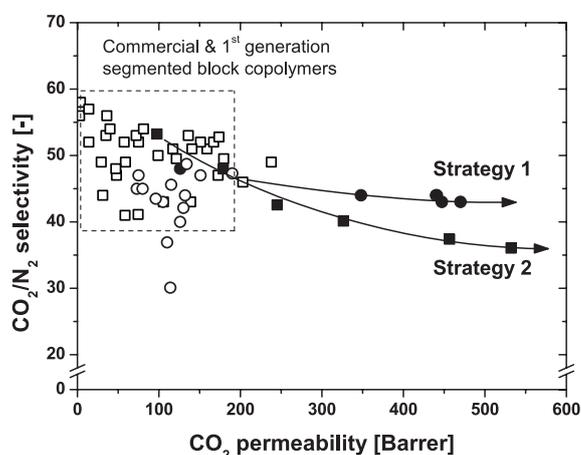


Figure 4 - Robeson CO<sub>2</sub>/N<sub>2</sub> upper bound relationship at 35°C. The data obtained according to strategy 1 (●) and strategy 2 (■) are compared to the data available on commercial & 1st generation segmented block copolymers and show the success of both strategies.

For more information please contact Dr. Ir. Kitty Nijmeijer ([d.c.nijmeijer@utwente.nl](mailto:d.c.nijmeijer@utwente.nl); phone: + 31 (0)53 489 4185).

## University of Twente hosts ICOM 2011

The organizing committee of ICOM 2011 has the pleasure to invite you for ICOM 2011. ICOM, International Congress on Membranes and Membrane Processes, is the world's largest conference on fundamental and applied membrane science, engineering and technology. It offers a platform for extensive exchange of ideas, thoughts and discussions on membranes and membrane processes.

ICOM 2011 will be hosted by the Membrane Technology Group of the University of Twente, The Netherlands. It will be organized in Amsterdam, The Netherlands from July 23 till July

29, 2011. More information on important deadlines, abstract submission, preliminary program, registration etc. will be posted on this website ([www.icom2011.org](http://www.icom2011.org)) very soon.

We are looking forward to an inspiring ICOM 2011.

Kindest regards,

Kitty Nijmeijer  
Antoine Kemperman  
Matthias Wessling



## Membrane runners participate in Batavierenrace

The Membrane Technology Group participated again in the Batavierenrace, which was organized on April 23 and 24, 2010. The Batavierenrace is a relay race of more than 175 kilometers, starting at the University Sports Centre in Nijmegen at midnight, going via Germany, the 'Achterhoek' and the 'Oude Markt' in Enschede to the campus of the University of Twente. The total distance is divided into 25 stages with 17 men's distances and 8 women's stages ranging from 3.3 to 11.2 kilometers. More than 300 teams participated and this made the organization of the Batavierenrace to be mentioned in the Guinness Book of Records.

The first Batavierenrace was organized in 1972 by a group of students from the University of Nijmegen. The name of the race refers to the route the Batavians took in 50 B.C. They sailed down the river Rhine from Nijmegen to Rotterdam, and the first race followed this original route. Later, due to infrastructural



problems the route was redirected to finish at the campus of the University of Twente.



The Membrane Technology Group has a long history in participating in this event, which is a mixture of a competitive as well as a social event for the group. The Membrane runners were ranked at the 289<sup>th</sup> place. Considering that not all distances could be occupied and some runners even ran two distances, this result is very good.



Hopefully the Membrane Runners will keep running in coming years and continue to show their competences in a field outside their daily research.



As a social group event and of course to refresh and reward the runners, a BBQ was organized after the race at Rob and Cindy Lammertink's place in Hengelo.



*This event would not have been possible without the financial support of our sponsors. The Membrane Technology Group would like to express their gratitude to: Norit X-flow BV, Wetsus, the European Membrane Institute Twente (EMI Twente) and Gambro Dialysatoren GmbH.*

# Real time visual characterization of membrane fouling and cleaning



Membrane fouling is simply described as the deposition of unwanted matter on the membrane surface during the course of operation, resulting in reduction in filtration efficiency. Fouling studies typically involve the use of invasive or non-visual techniques. This research is aimed at visually characterizing membrane fouling as well as fouling removal.

In a bid to better characterize membrane fouling, a novel technique was developed whereby fouling can be observed visually and in real time. The key to successful observation of the membrane is the fabrication of embedded channel membranes using phase separation micromolding (PS $\mu$ M) (Figure 1). These

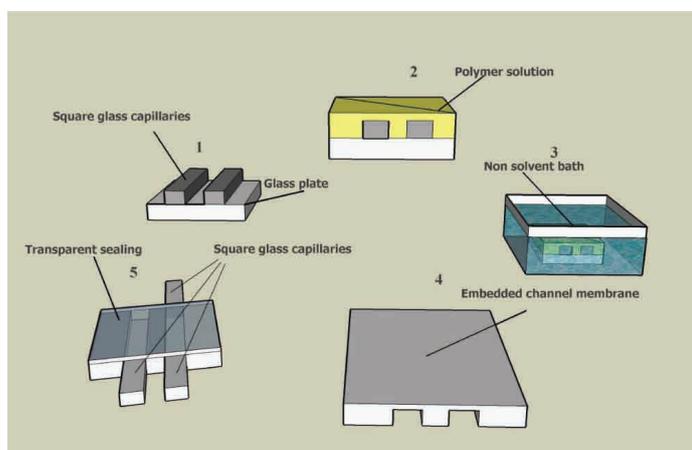


Figure 1 - Illustration of templating method of preparing embedded channeled membranes.

membranes are sealed and placed in a setup with a camera to view the membrane surface during operation. Using a model feed solution containing 6  $\mu$ m polystyrene particles, deposition on the membrane surface was studied. It was observed that initially, there is the buildup of cake towards the channel exit and with increasing local resistance, there is a change in hydrodynamics leading to buildup closer to the channel entrance.

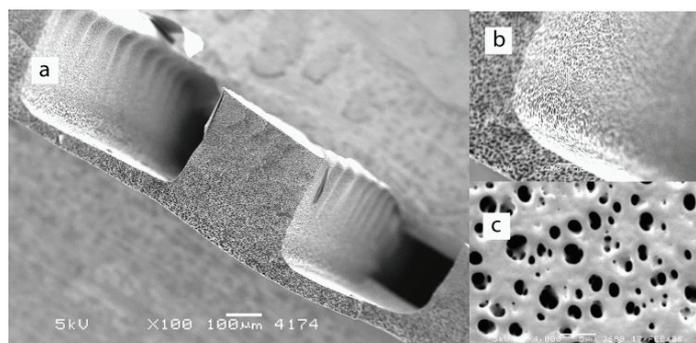


Figure 2 - SEM images of channeled membranes showing pore symmetry.

To better describe “normal” feed solutions, we studied membrane fouling using a feed solution containing bidisperse suspension (3.3 and 5.7  $\mu$ m) of polystyrene particles. Increasing the fraction of larger particles in the suspension resulted in an initial reduction in cake porosity leading to a minimum at number fraction of 0.5 (Figure 3). On the other hand, the specific cake resistance

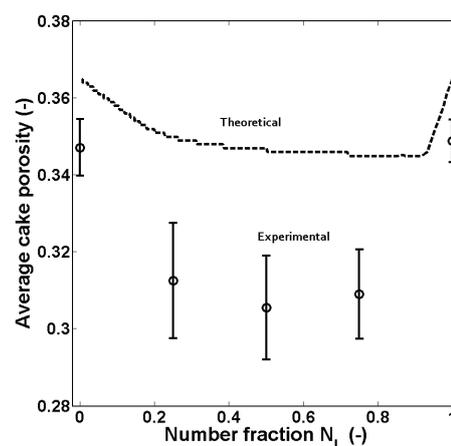


Figure 3 - Average cake porosity as function of number fraction (o - experimental data and dashed line is theoretical data from Tokumitsu method).

showed a continuing decline with addition of larger particles. These results compared favorably with theoretical calculations of cake porosity (using Tokumitsu’s method) and specific cake resistance (using Kozeny Carman relation). However, the experimental results gave lower absolute values which could be due to better ordering of the cake closer to the membrane (Figure 4).

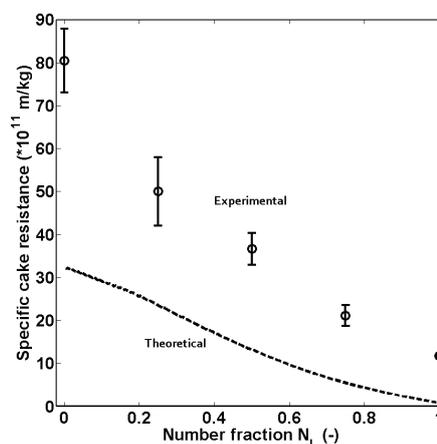


Figure 4 - Specific cake resistance as function of number fraction. (o - experimental data and dashed line is theoretical).

There have been reports of feed spacers contributing to (bio) fouling. This is interesting considering that spacers are used in spiral wound modules as “surface shear generators”. We studied the flow and (bio)fouling around micro structured membranes designed to mimic spacer nodes (Figure 5 and 6).

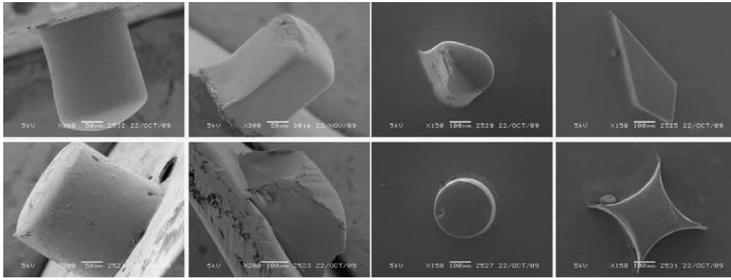


Figure 5 - SEM images of micro-structured membranes showing different geometries.

It was observed that the biofilm formation and growth occurred upstream of these structures contrary to reports in literature. Particulate fouling of the structures suggested that biofilm initiation is similar to particulate deposition whereby biomass on contacting the structure, adheres and grows leading

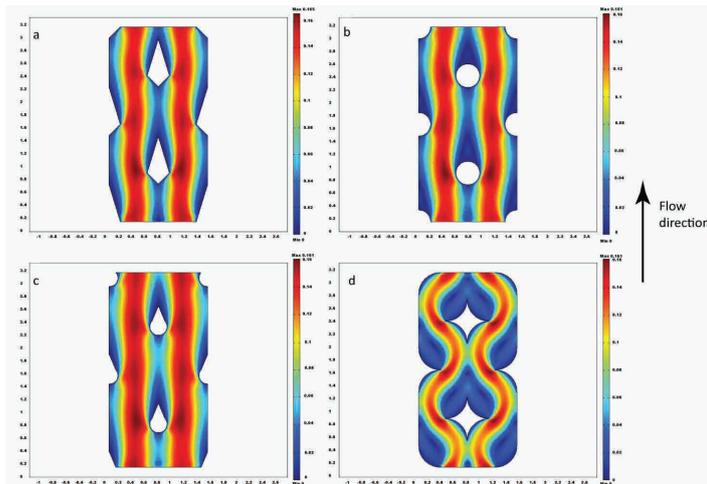


Figure 6 - CFD simulations of flow around micro-structured membranes.

to a biofilm. Deposition on these structures were compared to (bio)fouling of commercial net shaped spacers (Figure 7).

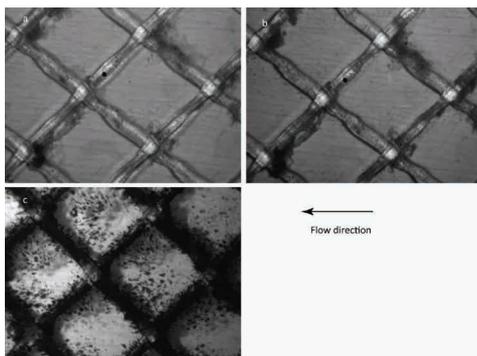


Figure 7 - Biofouling of net shaped spacers showing biofilm formation initiation and growth in time.

Air sparging is a technique which has been applied in industries for the removal of (bio)fouling from the spiral wound module. We described a novel sparging technique which was compared to typical sparging and forward flush in terms of removal of biofouling from membrane/spacer channels. Dissolved  $\text{CO}_2$  was applied as a cleaning agent with nucleation of the gas within the channels due to pressure drop as well as imperfections on the spacer surface acting as nucleation sites. It was observed that forward flush resulted in 40 % restoration of channel resistance

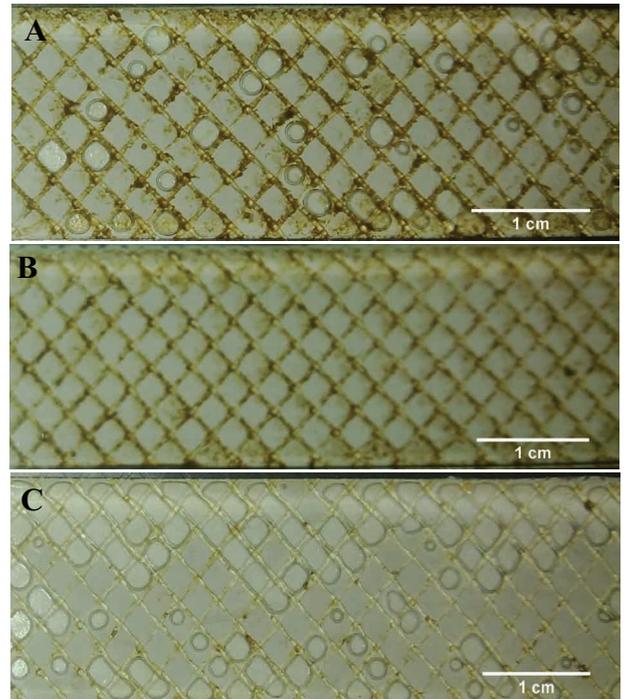


Figure 8 - Images of flow cells after cleaning protocol - (A) water rinsing, (B) water/ $\text{N}_2$  sparging and (C) water/ $\text{CO}_2$  nucleation.

while the water/ $\text{N}_2$  sparging resulted in 80 % restoration. Water/ $\text{CO}_2$  nucleation resulted in 100 % restoration of the channel resistance due to better distribution of the bubbles within the channel (Figure 8).

For more information please contact Prof. Dr. Ir. Rob Lammertink (r.g.h.lammertink@utwente.nl; phone: + 31 (0)53 489 2063).

### MNT- Information

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

#### Editors

Matthias Wessling  
Kitty Nijmeijer  
Enver Güler  
Zeynep Çulfaz

membrane@utwente.nl

www.membrane.nl

# Membrane technologies for CO<sub>2</sub> capture

## Introduction

This thesis investigates the potential of membrane technology for the effective removal of CO<sub>2</sub> from CH<sub>4</sub>. The work focuses on two distinctively different membrane processes to accomplish the separation, i.e. 1) the use of a gas-liquid membrane contactor for the selective absorption of CO<sub>2</sub> from CH<sub>4</sub> and 2) the use of thin, dense gas separation membranes to establish the separation.

## Gas-liquid membrane contactors for CO<sub>2</sub> separation

A membrane contactor (Figure 1) combines the advantages of membrane technology with those of an absorption liquid.

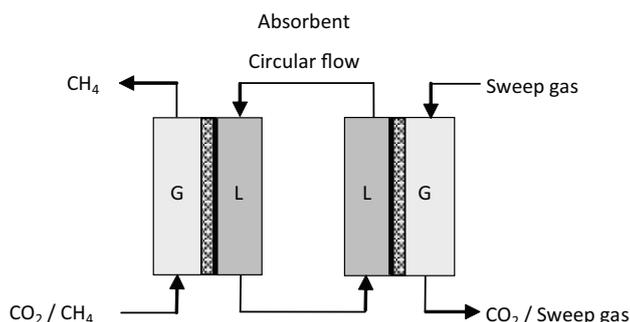


Figure 1 - Schematic representation of a membrane contactor for the separation of CO<sub>2</sub> and CH<sub>4</sub>.

In a gas-liquid membrane contactor the membrane acts as an interface between the feed gas and the absorption liquid. Gas-liquid membrane contactors offer a unique way to perform gas-liquid absorption processes in a controlled fashion while they have a high operational flexibility. The two major parts in a gas-liquid membrane contactor that determine the separation are the membrane and the absorption liquid.

Next to porous (PP) hollow fiber membranes, asymmetric

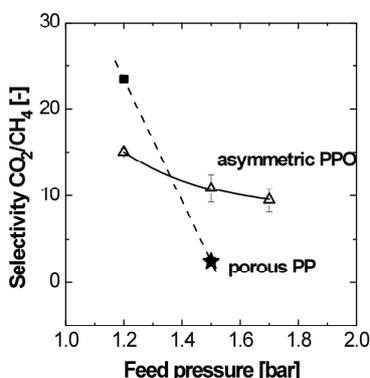


Figure 2 - CO<sub>2</sub>/CH<sub>4</sub> selectivity as a function of the feed pressure for both the porous PP membranes and the asymmetric PPO membranes ( $T_{\text{abs}} = T_{\text{des}} = 29^\circ\text{C}$ ; liquid flow rate = 160 ml/min). The star for the PP fibers at a feed pressure of 1.5 bar represents the occurrence of visible gas bubble formation in the absorption liquid.

(PPO) hollow fiber membranes with a dense, ultrathin skin at the outside of the membrane have been used in a membrane contactor and the influence of different process parameters on productivity and selectivity has been evaluated. The



PP membranes outperform the PPO membranes in terms of productivity and selectivity, but the PPO membranes are much less sensitive towards variations in feed pressure, which increases the operating window and potential of the membrane contactor process (Figure 2).

Alkanol amines (e.g. MEA) are traditionally used as absorption liquid in CO<sub>2</sub> absorption processes, but have certain disadvantages (corrosiveness, energy consumption, oxidative stability). Amino acid salt solutions (Figure 3) are a competitive alternative for such alkanol amines (e.g. MEA).

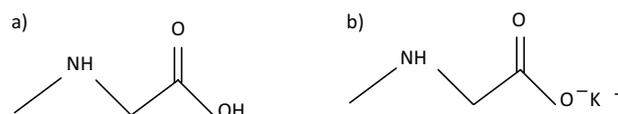


Figure 3 - Chemical structure of a) sarcosine and b) the potassium salt of sarcosine.

Amino acid salt solutions have similar reactivity and CO<sub>2</sub> absorption capacity as alkanol amines, but in addition they can be made non-volatile by adding a salt functionality, which significantly reduces the liquid losses. The kinetics of the absorption CO<sub>2</sub> in an amino acid salt solution (sarcosine) have

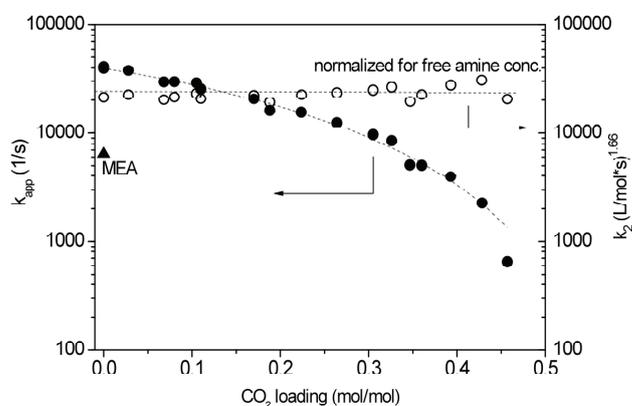


Figure 4 - Apparent reaction rate constant for sarcosine  $k_{\text{app}}$  (full circle) and the amine concentration normalized reaction rate constant  $k_2$  (empty circle) as a function of the degree of CO<sub>2</sub> loading of a 1.45M aqueous sarcosine salt solution at 298 K (MEA value is given as a reference).

been studied. Reaction rate constants significantly higher than those found for MEA were obtained. A strong decrease in the apparent reaction rate constant (full circle) was observed with increasing CO<sub>2</sub> loading of the absorption liquid (Figure 4). Consequently, higher partial loadings result in lower absorption fluxes, which will lead to larger process equipment and increased costs in real applications. When normalized for the free amine concentration available for the reaction with CO<sub>2</sub>, the value of the reaction rate constant  $k_2$  (o) is independent of the CO<sub>2</sub> loading of the absorption liquid and directly related to the free amine concentration available for the reaction.

The performance of this amino acid salt solution in a membrane contactor was evaluated. Very high CO<sub>2</sub>/CH<sub>4</sub> selectivities already at low temperature differences between absorber and desorber were obtained due to very low CH<sub>4</sub> permeances (Figure 5).

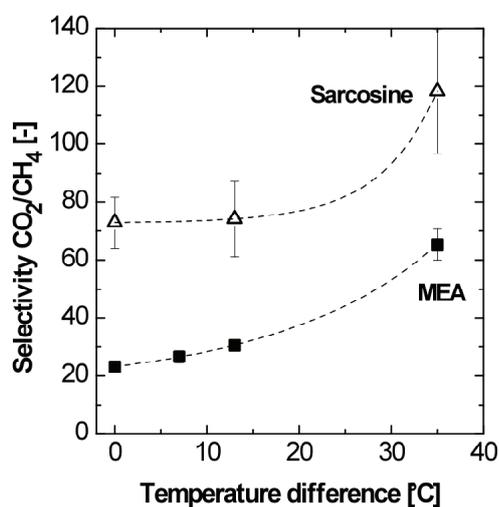


Figure 5 - CO<sub>2</sub>/CH<sub>4</sub> selectivity as a function of temperature difference between absorption and desorption (T<sub>abs</sub> = 29°C; Liquid flow rate = 160 ml/min; Feed gas mixture: CO<sub>2</sub>/CH<sub>4</sub> (20/80 vol.%); Feed pressure = 1.2 10<sup>5</sup> Pa).

## Gas separation membranes

The first type of polymer membrane investigated for gas separation is based on room temperature ionic liquids. Room-temperature ionic liquids (RTILs) are a class of organic solvents that have been explored as novel media for CO<sub>2</sub> separations. Polymerized RTILs (poly(RTILs)) can be synthesized from RTIL monomers to form dense, solid gas selective membranes. An imidazolium-based poly(RTIL) (Figure 6) is used as base material and the length of the alkyl chain serves as a tool to strengthen or weaken the ionic interactions within the poly(RTIL) and consequently the plasticization resistance of the membranes.

High pressure gas permeation experiments were performed. As opposed to regular glassy polymers, poly(RTIL)s do not

show a minimum in permeation rates for CO<sub>2</sub>; the permeability increases continuously with increasing feed pressure. In gas mixtures, CO<sub>2</sub> accelerates the transport rate of CH<sub>4</sub>. The poly(RTIL) membranes with the short, methyl substituted side chain and stronger ionic interactions showed an increased selectivity and resistance towards plasticization at the expense of the permeability compared to the polymers with the longer side chains.

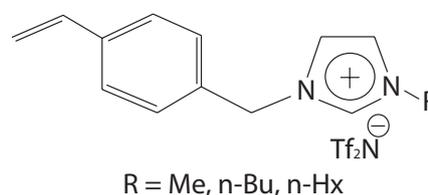


Figure 6 - Structure of the RTIL monomers.

Next to these poly(RTIL) membranes, we have investigated the plasticization behavior of thin and thick polymer films of ODPa-based polyetherimides for CO<sub>2</sub> separation. ODPa-based PEI polymers with 1, 2 or 3 para-arylene rings were investigated under conditions where commercial membranes suffer from plasticization. The glassy polymers display increasing CO<sub>2</sub> sorption with increasing T<sub>g</sub>. The larger extent of sorption results from a larger non-equilibrium excess free volume. Swelling of the polymers is induced by sorption of CO<sub>2</sub> molecules in the non-equilibrium free volume as well as from molecules dissolved in the matrix. The partial molar volume of CO<sub>2</sub> is similar for molecules present in both regions. Sorption of CO<sub>2</sub> in thin films only differs from that in bulk materials regarding the Langmuir capacity (C<sub>H</sub>), while the values obtained for the Henry's law constant (k<sub>D</sub>) and the Langmuir affinity parameter b coincide (Figure 7). This indicates that the overall affinity of CO<sub>2</sub> for the

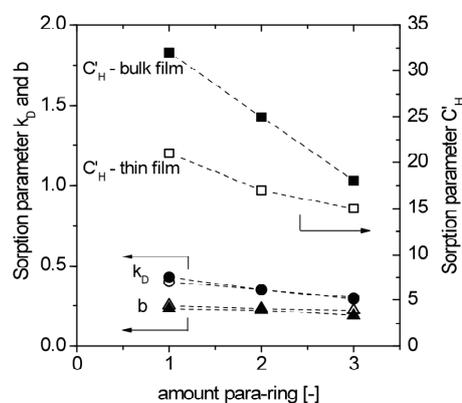


Figure 7 - Gas sorption parameters in thin (open symbols) and thick, bulk (closed symbols) ODPa PEI (-P1, -P2 and -P3) films at 35 °C. k<sub>D</sub> (full circle and empty circle) expressed in (cm<sup>3</sup> (STP)/ (cm<sup>3</sup> polymer•bar)), C<sub>H</sub> (full square and empty square) expressed in (cm<sup>3</sup> (STP)/cm<sup>3</sup> polymer) and b (full triangle and empty triangle) expressed in (1/bar).

polymer materials is determined predominantly by the inherent chemical characteristics of the material.

The extent of swelling of the ODPA PEI films is very low, which is beneficial for CO<sub>2</sub> removal at elevated pressures. Mixed gas separation experiments showed high CO<sub>2</sub>/CH<sub>4</sub> selectivities for the ODPA PEI films at elevated pressure, confirming the potential of this material for the effective separation of CO<sub>2</sub> from CH<sub>4</sub>.

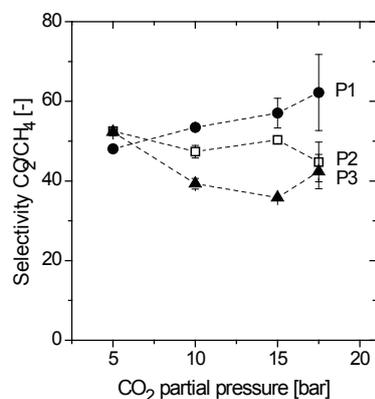


Figure 8 - CO<sub>2</sub>/CH<sub>4</sub> selectivity as a function of CO<sub>2</sub> partial pressure for thick ODPA P1, P2 and P3 films at 35°C. Feed gas mixture: CO<sub>2</sub>/CH<sub>4</sub> (50/50 vol.%).

The mixed gas permeation experiments (Figure 8) show high CO<sub>2</sub>/CH<sub>4</sub> selectivities for the ODPA PEI films even at elevated pressure and consequently have potential as membrane material

for effective CO<sub>2</sub> removal.

For more information please contact Dr. Ir. Kitty Nijmeijer (d.c.nijmeijer@utwente.nl; phone: + 31 (0)53 489 4185).

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## Introducing...

### Treatment of nanofiltration concentrates

Name

Christina Kappel

Origin

Germany

Contact

<http://mtg.tnw.utwente.nl>

0031 (0) 58 284 30 175

[christina.kappel@wetsus.nl](mailto:christina.kappel@wetsus.nl)



Christina Kappel

finished her bachelor degree in Water Science at the University Duisburg-Essen (Germany) with a research thesis on “Effects of media filling ratio and

dissolved oxygen concentration on IFAS media nitrification” in 2008. After that she studied Water and Wastewater Technology at Cranfield University (UK) and finished her master degree working on „Simplified Evaluation of the Operational Oxygen Transfer Efficiency of a novel dispersed integrated fixed film activated sludge process” in 2009. Since November 2009 she is a member of the Membrane Technology Group, working at Wetsus in Leeuwarden. She is working on her PhD topic “Treatment of nanofiltration concentrates, produced during polishing of municipal wastewater treatment plant effluent”.

### High temperature creep behavior of mixed-conducting perovskite membranes

Name

Bo Wang

Origin

China

Contact

<http://mtg.tnw.utwente.nl>

0031 (0) 53 489 2998

[b.wang@tnw.utwente.nl](mailto:b.wang@tnw.utwente.nl)



Bo Wang received

his PhD degree from the University of Science and Technology of China, Hefei, China, where he focused his research on dual-phase dense oxygen permeable

membranes and application of this type of membranes in catalytic membrane reactors. For one year, he worked as a postdoc in Imperial College, London, where he worked on ceramic hollow fibre membranes. Bo Wang joined the Inorganic Membranes group in April 2010 as a postdoc. He will study the high temperature creep behavior of mixed-conducting perovskite membranes materials in a project funded by the Dutch Ministry of Economic Affairs via the EOS-LT program.

## Introducing...

### Ultra thin film geometries of membranes

Name  
Wojciech Ogieglo

Origin  
Poland

Contact  
<http://mtg.tnw.utwente.nl>  
0031 (0) 489 2962  
[w.ogieglo@utwente.nl](mailto:w.ogieglo@utwente.nl)



Wojciech Ogieglo obtained the BSc. degree in Polymer Technology at Cracow University of Technology. In 2008, he took part in an internship at the Technological Department of Synthos S.A., polish chemical company, main Central European supplier of butadiene rubbers and polystyrenes. During the Master course he joined a binational Polish-German student exchange program which resulted in MSc. degree in Applied Chemistry obtained at Münster University of Applied Sciences. The topics of his Master Thesis included matrix effects investigations in the analysis of heavy metal content in plastics with a use of Laser Ablation coupled with Inductively Coupled Plasma technique. In 2010, he started his PhD in the Membrane Technology Group where he investigates the influences of nanoconfinement on various properties of polymer films. The research is focused, in particular, on membrane related implications of ultra thin film geometries.

### Air/water cleaning in spiral wound RO modules

Name  
Yusuf Wibisono

Origin  
Indonesia

Contact  
<http://mtg.tnw.utwente.nl>  
0031 (0) 53 489 3674  
[y.wibisono@utwente.nl](mailto:y.wibisono@utwente.nl)



Yusuf Wibisono was born in Indonesia. He obtained his B.Eng. degree in Agricultural and Biosystems Engineering from IPB – Indonesia, where he worked on the design process of cooling tower for agricultural product storage at the Center for Research on Engineering Application in Tropical Agriculture. He pursued his MSc in Chemical Engineering and Materials at Chung Yuan University – Taiwan, where he worked on computational fluid dynamics (CFD) modeling of vegetable oil degumming for biodiesel manufacturing by ultrafiltration with ceramic membranes. He joined the Membrane Technology Group as a PhD student in April 2010. His PhD research project is entitled “The Use of Two Phase Flow in Controlling Fouling in Spiral Wound Membranes”, a joint project between Membrane Process Technology - University of Twente, KWR - Watercycle Research Institute and Wetsus - Centre of Excellence for Sustainable Water Technology.

### Carbon molecular sieve (CMS) membranes for olefin/paraffin separation

Name  
Irdham  
Kusumawardhana

Origin  
Indonesia

Contact  
<http://mtg.tnw.utwente.nl>  
0031 (0) 53 489 2962  
[i.kusumawardhana@utwente.nl](mailto:i.kusumawardhana@utwente.nl)



Irdham graduated from the Department of Chemical Engineering, of the Chung Yuan University-Taiwan in 2008. In his master, he studied nanofiltration performances of poly(tetrafluoroethylene) thin film composite membranes from interfacial polymerization. After he obtained his master degree, in October 2008, he worked as researcher at the department of membranes and sustainable energy, GKSS Germany on the development of carbon nanotube membranes for gas separation and desalination. On March 1, 2010 Irdham joined the Membrane Technology Group. His PhD research focuses on the design and development of carbon molecular sieve (CMS) membranes for olefin/paraffin separation in order to identify structure-properties relationships.

## Ph.D. Defenses

December 3, 2010

Zeynep Culfaz

Microstructured Hollow Fibers  
and Microsieves: Fabrication,  
Characterization and Filtration  
Applications

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