2009 winter

Membrane News Twente

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

A NEW TURN

Matthias Wessling

After 10 years of heading the chair of Membrane Science and Technology, a new development will bring change into the operations of the Membrane Technology Group in Twente. The RWTH Aachen University proposed me as a candidate for a Alexander von Humboldt (AvH) Professor. To position this into the academic landscape, I would like to cite the website of the AvH Society: *The most valuable international award for research in Germany is being granted by the Alexander von Humboldt Foundation to globally leading academics of all disciplines working abroad.* It has been an honor to be proposed by RWTH Aachen University, and much more so to being nominated and awarded this position.

The award goes along with the fact that I need to relocate the majority of my research activities to the hosting university. RWTH Aachen University has proposed and organized that I will succeed Prof. Dr.-Ing. Thomas Melin on the Chair of Chemical Engineering within the Faculty of Mechanical Engineering from 2011 on. From the 1st of January 2010, I will start in Aachen to build up new research lines with the help of the AvH grant.

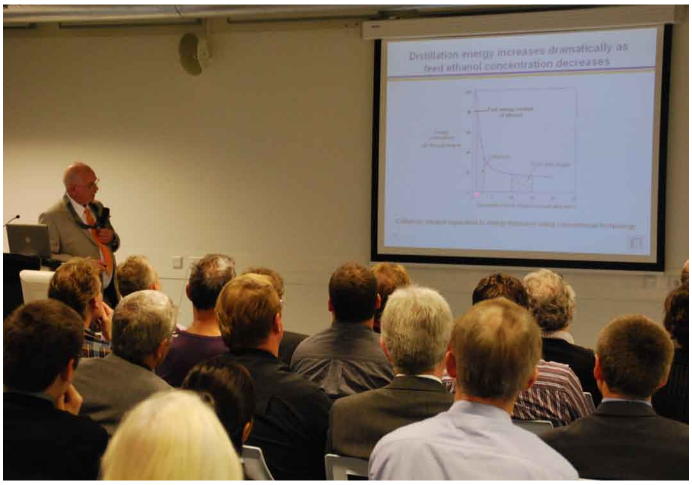
Today, the Membrane Technology Group in Twente has a significant momentum. Over the past ten years, I had the pleasure to materialize today's structure and research program together with many of the Ph.D. students and permanent staff. The satisfaction and pleasure we had over the years has resulted recently in an excellent evaluation within a national evaluation of Chemical Engineering research. I am proud of this success that is based on a tremendous effort of the team of membrane lovers. This effort, the commitment of each of the group members, and the passion for Research & Development in the field of Membrane Science and Technology has also been an important pre-requisite for me winning the AvH-Professorship. Hence the award also recognizes all collaborators that I have had the pleasure to work with. I am grateful for the fruitful collaborations and their endeavors.

From 1st of January 2010 on, I will work at the RWTH Aachen University. None of my current Ph.D. students will join, as this would be impossible contractwise. Fortunately, I will be able to continue the supervision of the PhD students currently working in the group in Twente. I foresee many complementarities in the research program of both universities, with the strong emphasis on membrane preparation and materials processing in Twente and the module and process engineering expertise in Aachen. The future will hopefully show a strong alliance between the two labs.

The AvH Award is meant as a lighthouse, signaling the various stakeholders in academia and industry where to sail with respect to future science and technology. I am happy to join RWTH Aachen since it (1) is a Technical University with Excellence Status, fully dedicated to engineering and technology, (2) has a clear ambition to bridge the gap between science and engineering which I find to be increasingly difficult with the pressure on publishing in high impact journals and (3) has a strong chemical engineering program focusing of sustainable processes, in particular on tailor-made fuels from bio feed stock and (4) has a strong activity in nanostructured interactive materials. I will continue my journey between the worlds of nanomaterials and chemical process engineering. I wish that my personal enthusiasm will also stimulate future collaborators in Aachen, today's colleagues and group members in Twente, as well industrial and governmental project partners.

Symposium to the honor of Prof. Heiner Strathmann

In August 2009 Prof. Heiner Strathmann, former head of the Membrane Technology Group, celebrated his 75th birthday. To honor Heiner, a symposium was organized by his former Group on Friday November 20th 2009 at the University of Twente.



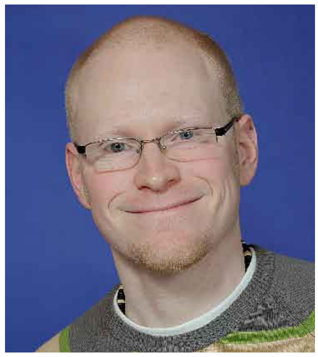
In total 88 participants attended this meeting. Part of the guests arrived already on Thursday evening and enjoyed a splendid dinner at bistro de Broeierd. During the dinner Heiner's predecessor, Prof. Kees Smolders, and Heiner's successor, Prof. Matthias Wessling, shared some of their memories on Heiner.

On Friday morning the lecture room was completely filled with family, friends and former colleagues of Heiner. This reunion took place in a stimulating atmosphere, with lots of laughter and with reviving good old remembrances. The official part of the symposium consisted of 9 presentations on membrane and non-membrane related subjects, interrupted by the lunch organized in an olive tree filled atrium.

Some of Heiner's former colleagues who could not attend the symposium sent presentations with their memories. Heiner himself closed the meeting by showing some nice drawings reminding us of his period in Twente and his lost tennis and ping -pong matches.

The symposium was concluded by a buffet at the campus restaurant. All participants made clear they very much appreciated the symposium and the contacts with their former colleagues.

Phase Separation MicroFabrication Fundamental Aspects



Phase Separation MicroFabrication (PSμF) is a new technique that is used for patterning the surface of polymeric films. This process relies on the phase separation of polymer solutions while they are in contact with microstructured molds (Figure 1). Virtually all polymers for which a solvent (and a nonsolvent) can be found, can be processed with PSμF. The molds in use are normally silicon wafers with a pattern of microstructures created through etching.

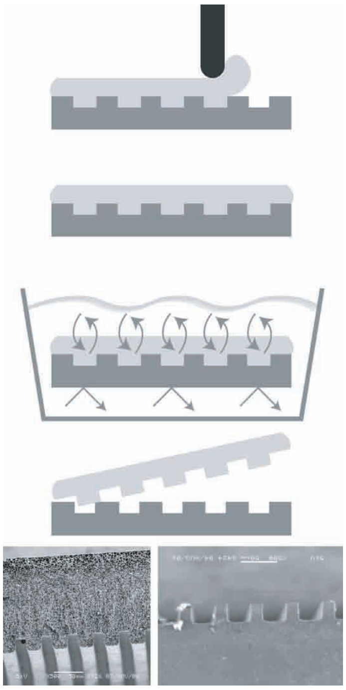


Figure 1. Scheme of the PSμF process. As an example, the cross section of a membrane with a line pattern is shown next to the mold used for making it.

Phase separation processes take place through the creation of two phases from an originally homogeneous system. Normally, a polymer-rich phase is created, which solidifies and constitutes the matrix of the membrane. Next to it, a polymer-lean phase appears. This phase is dispersed in the polymer-rich phase but cannot solidify. This creates the pores inside the membrane. In liquid-induced (LIPS), a nonsolvent is added to the polymer solution. The nonsolvent is miscible with the solvent of the polymer solution, but does not dissolve the polymer. The way in which the nonsolvent is added, along with the composition of the polymer solution, determines the morphology of the membrane (Figure 2).

Shrinkage is known to accompany the phase separation of polymer solutions. Since it can affect the performance in replication of the features in the mold, its study is key to a successful implementation of PSμF. To study this phenomenon, membranes from several solutions with different concentrations of polyethersulfone in NMP have been prepared using three molds. The concentration of polymer strongly affected the porosity of the final product. With increasing PES concentrations, higher compaction levels were observed, indicated by denser and thinner membranes, for constant casting thicknesses. This variation occurred within two limits given mainly by the minimum and maximum achievable pore

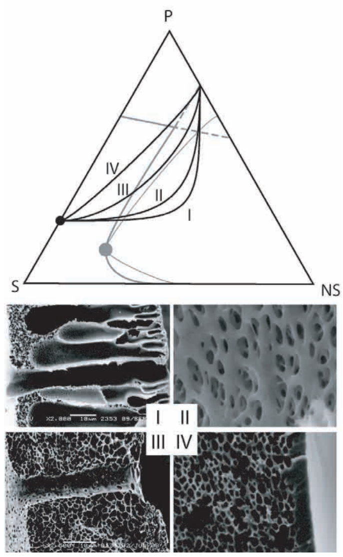


Figure 2. Different membranes obtained from PES solutions with different additives. The composition paths show that the coagulation was milder from I to IV.

sizes. Outside of this range the final thickness varied in the same direction as the polymer concentration, for a constant casting thickness. The porosity of the membranes was found to follow the same trend but not to a similar extent in all cases (Figure 3).

In the case of thickness shrinkage, each layer contributes to the total shrinkage. However, in lateral shrinkage each layer shrinks over the remaining polymer solution and in contact with the solidified layers on top. Hence, lateral shrinkage is somewhat hindered by the layers that already solidified, causing tensions on these layers. As a result, lateral shrinkage was as small as one-sixth of the normal one. When the area occupied by the features (directly related to the amount of features, their size, and type) is low, there is little force pulling the film on top during coagulation. As a result, the non-solvent in the coagulation bath can easily access the space between the film and the mold. This is also the case when the formed membrane is skinless and extremely porous. In either case, the access of non-solvent between membrane and mold allowed for polymer retraction from the mold and inaccurate replication.

Upon casting a polymer solution on a wafer with wells, the wells do not get filled directly. This is caused by entrapped air bubbles that interfere with the filling of the

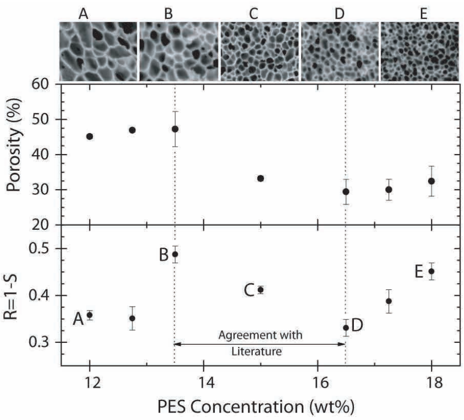


Figure 3. Dependence of porosity and shrinkage (S) as a function of polymer concentration in the casting solution.

wells in the mold. The variation of the volume of the bubble in time showed that the dissolution of the entrapped gas in the solution is of high importance for the filling process. This has been verified through measurements in a custom made chip (Figure 4). It has been seen that carbon dioxide dissolves quite quickly. Almost 800 times faster than oxygen, which in turn dissolves roughly twice as fast as nitrogen. Based on this, it can be concluded that the casting atmosphere is an important parameter in the production of polymeric structures via phase separation microfabrication. Particularly, if the mold has wells.

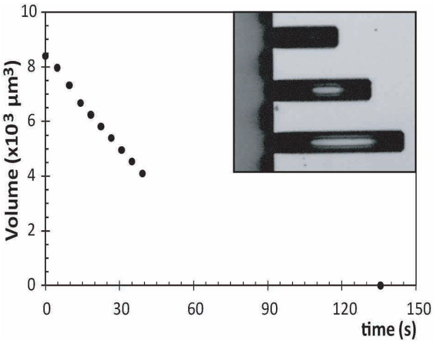


Figure 4. Picture of bubbles in the channels of our chip. The shape of the bubbles has been modeled with the help of Surface Evolver. In this way, the volume of the bubbles could be followed in time.

PSµF has been shown to be a simple, cost effective way to make polymeric microsieves. Pillar density has been presented as one of the main parameters affecting the quality and feasibility of release from the mold. This was measured through the use of a peeling machine equipped with a force transducer for measuring the force required for peeling (Figure 5). It has been proven that square arrays of pillars with fewer pillars per row or higher distance between rows contribute to lower peeling forces. Furthermore, the effect of the distance between rows is more important than that of the distance between pillars in a single row. The increased distance between pillars caused a decrease in peeling force varying between 10 and 30% for different pillar densities.

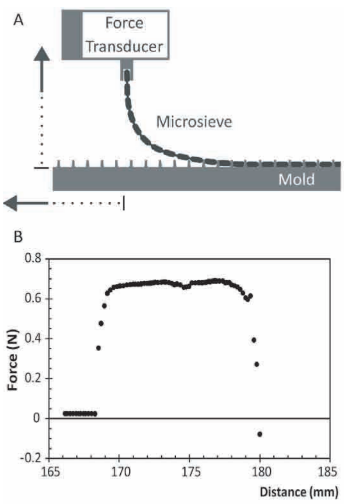


Figure 5. A. Representation of the peeling device. B Typical result obtained when peeling a microsieve from the mold.

Different pillar dispositions showed the importance of breaking shrinkage lines along the fields. In fields with repeating units, the lines between these units are occupied entirely by polymer solution, without features. As a result, the shrinkage of these lines pulls the polymer against the pillars, increasing friction, with the highest possible force for this system. Therefore, it is required to cut these lines shorter. In case of the square disposition, these lines are abundant, so almost any change is an improvement.

Permeable PDMS molds have been developed as part of this research. This is of high importance for a better replication of the structures. The opportunity to start the solidification process from the structured size guarantees that the features are extremely well replicated. Their structure is fixed and the layers of solution underneath them will not have an effect on them. This is not the case for PSµF on silicon wafers, as these are impermeable.

The use of permeable molds on both sides of the polymer solution allows for membranes with structures on both sides. Furthermore, pressing these molds against one another creates mold contact regions where no polymer solution is present. Upon solidification, these voids become pores through the film. Using this phenomenon and two molds containing line patterns, microfences were made (Figure 6). These fences present an arrange of square pores produced without need for perforation by the mold, simplifying the process of micromesh making.

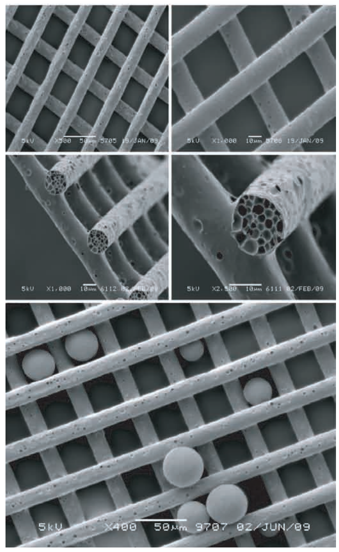


Figure 6. Microfence and an example of its use to filter polymeric beads.

For more information please contact Dr.ir. Rob Lammertink (r.g.h.lammertink@utwente.nl; phone: + 31 (0)53 4892063).

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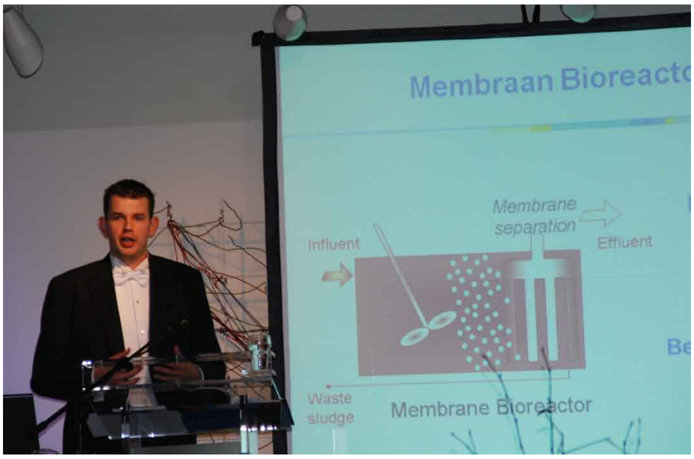
Two Ph.D. defenses on the same day

On the 18th of November, two Ph.D. candidates of the Membrane Technology Group at Wetsus defended their Ph.D. work in the Fries Museum in Leeuwarden (located in the Northern part of The Netherlands).



Perry van de Marel, supervised by Dr. Antoine Kemperman and Prof. Walter van der Meer, developed a measuring method to reduce fouling in membrane bioreactors (MBRs). More information on the work of Perry can be found on pages 4 and 5 of this news letter.

Piotr Długołęcki, supervised by Dr. Kitty Nijmeijer and Prof. Matthias Wessling, investigated the potential of Reverse

Electrodialysis, the generation of power from the mixing of fresh and salt water. The work investigates the design, optimization and practical potential of reverse electrodialysis, with a special focus on membrane and spacer properties, mass transfer of the ionic species and concentration polarization phenomena. The results of this work show the high potential of RED as renewable energy source and it also brings RED closer to practical implementation.

Both Ph.D. students performed their work at Wetsus in Leeuwarden. Wetsus is a centre of excellence for sustainable water technology and offers a platform for collaboration between universities and industries. It combines scientific excellence with practical applications and commercial relevance. More information can be found at www.wetsus.nl.

For more information on the work on Reverse Electrodialysis, please contact Dr. Kitty Nijmeijer (d.c.nijmeijer@utwente.nl; phone: + 31 (0)53 489 4185). For more information on the work on membrane bioreactors, please contact Dr. Antoine Kemperman (a.j.b.kemperman@utwente.nl; phone: + 31 (0)53 489 2956).

Influence of membrane properties on fouling in MBRs

Nearly all wastewater treatment plants (WWTPs) apply an activated sludge process. In conventional activated sludge (CAS) systems the activated sludge is separated from the purified water before discharge using large clarifiers. A membrane bioreactor (MBR) applies membranes for separating sludge and water, resulting in a smaller foot print and better effluent quality compared to CAS systems. Operational costs of MBRs, however, are higher due to measures necessary to reduce membrane fouling (Figure 1). The subject of this Ph.D. project was to elucidate optimal membrane properties in order to reduce membrane fouling.

Figure 1 - Membrane configuration used during the studies showing a fouled membrane.

A new experimental flux-step protocol was developed. This improved flux-step method (IFSM, figure 2) [1] was applied to determine optimal membrane properties out of a series of flat-sheet membranes. In the IFSM a relaxation step is applied, enabling determination of both the critical flux and the critical flux for irreversibility. Membranes with optimal properties will show a high critical flux (low fouling) and a much higher critical flux for irreversibility. This makes application of supra-critical fluxes in long-term MBR operation possible, where normally sub-critical filtration is applied. A higher flux considerably lowers the membrane surface area required, thereby reducing energy consumption for coarse bubble aeration.

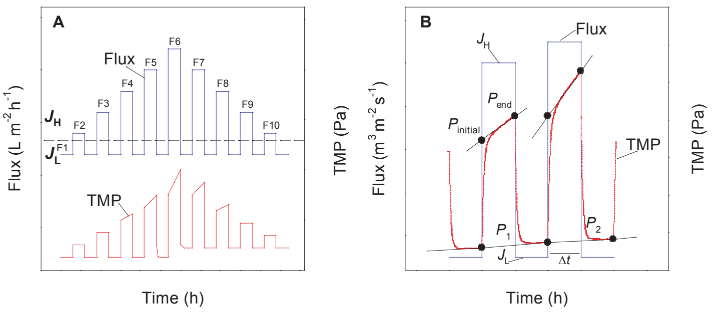


Figure 2 - Example of a flux–TMP profile of the improved flux-step method (A) and a schematic representation of the improved flux-step protocol (B).

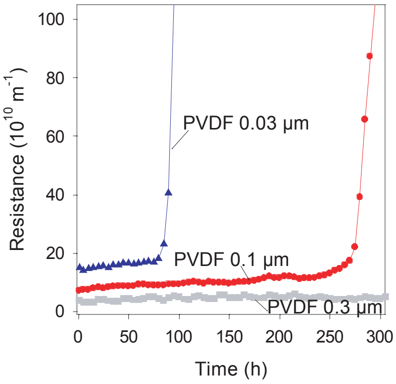


Figure 3 - Critical flux (J_{Cri}) and critical flux for irreversibility ($J_{cri'}$) for different membranes in activated sludge of a pilot-scale MBR.

The IFSM was applied in three different activated sludge mixtures and for five different polymeric membrane materials with varying membrane properties. The optimal membrane that suffered the least from membrane fouling was elucidated, as well as the influence of sludge type on the optimal membrane properties. The fouling behaviour was different for the five membrane materials, and was not the same for membranes made of the same polymeric material but differing in pore size, surface porosity, and hydrophobicity (Figure 3). The results also showed the same trend towards membrane fouling was observed for the membranes in all three activated sludge mixtures. Tests with 15 different membranes showed that a hydrophilic asymmetric PVDF membrane with an interconnected pore structure, a nominal pore size of 0.3 µm, and a large surface porosity of 27% gave the best membrane performance. Long-term filtration experiments revealed gel formation as the dominant fouling mechanism. Gel layer formation was the least severe for the optimal membrane, indicated by the longest stable filtration resistance period (Figure 4).

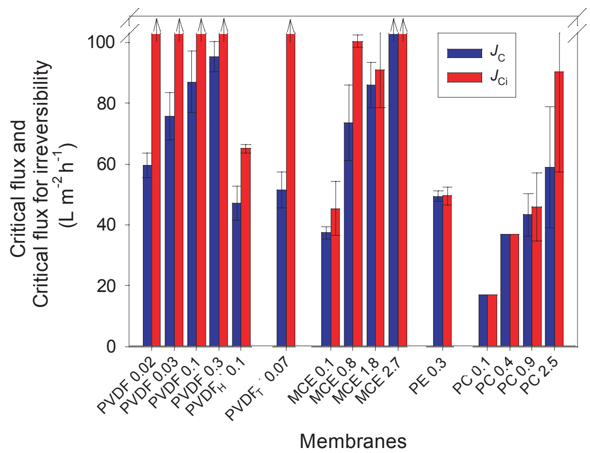


Figure 4 - Filtration resistance of three PVDF membranes (pore size indicated) during subcritical filtration at a flux of 50 L m^{-2} h^{-1}.

The gel layer formation process seemed inevitable since also during sub-critical filtration sooner or later an exponential increase in resistance occurred. A pioneering study with corrugated membranes was performed to extend the period until gel layer formation happened and to extend the sustainable filtration time in this way. The corrugated membranes were placed either with a vertical orientation (C2) or with a horizontal orientation (C3) of the corrugates (Figure 5). The results showed that the corrugations increased the critical flux and the sustainable filtration time compared to a smooth flat-sheet membrane configuration (C1). On the long term, however, the horizontally oriented corrugates were less favourable, since sludge particles deposited in the valleys between the corrugates. The use of vertically positioned corrugated membranes may extend the sustainable filtration time in an MBR without increasing its operational costs.

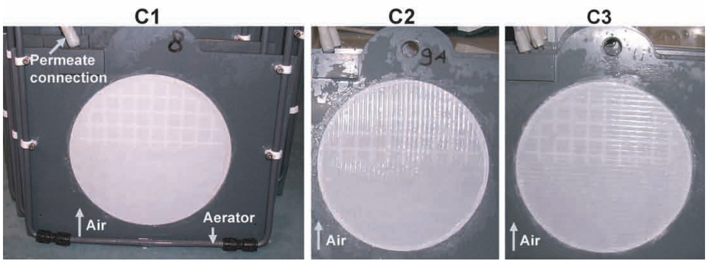


Figure 5 - Membrane plates with smooth flat-sheet membrane (C1), and corrugated flat-sheet membranes with vertical (C2) and horizontal orientation of the corrugates (C3).

In MBRs with flat-sheet membranes usually relaxation is applied as cleaning method to remove the reversible membrane fouling deposited on the membrane surface. Backwashing however, may offer the advantage over relaxation that also internal fouling is (partly) removed. The results described in this thesis showed that cleaning by backwashing and relaxation were equally efficient in removing membrane fouling, but both did not remove irreversible fouling caused by internal fouling and gel layer formation. From the three membrane materials investigated a PVDF membrane fouled the least, which could be attributed to its asymmetric pore morphology. The ceramic membrane and a PE membrane tested both had a symmetric pore morphology in the top layer. Particles became trapped in this top layer and were not removed by neither relaxation nor backwashing. A chemically enhanced backwash (CEB) with sodium hypochlorite was able to remove the internal fouling partially.

This work showed that by a smart choice of the membrane applied in an MBR the operational costs can be lower since fouling is reduced and higher fluxes can be applied.

For more information please contact Dr.ir. Antoine Kemperman (a.j.b.kemperman@utwente.nl; phone: + 31 (0)53 489 2956).

Presentation awards at Euromembrane 2009

During Euromembrane 2009, Montpellier, two Ph.D. students of the Membrane Technology Group, Karina Kopec and Jigar Jani, received a presentation award for their oral presentation. The 5 presentation awards that were provided by the European Membrane Society and the organizing committee of Euromembrane 2009 consist of a certificate and 500 euro.

Karina Kopec was awarded for her presentation entitled "Capillary electrochromatography and membrane technology: Merging the advantages". She presented the application of charged polymeric small-bore fibers, made by immersion precipitation spinning, as stationary phase for capillary electrochromatography columns. Karina emphasized the novelty of her project and discussed the advantages of membrane technology in the field of microcolumn separations.

The second winner, Jigar Jani, received the recognition for his presentation "Gas-liquid contacting in ceramic membrane microreactors: Numerical and experimental study". Jigar discussed various microreactor designs and focused in his presentation on the numerical simulation of the fluid dynamics in the hollow fiber microreator. He showed the numerical prediction of the enhancement obtainable in mixing and mass transfer using hollow fiber geometry.

Marcel Mulder Award for Piotr Długołęcki

Piotr Długołecki, Ph.D. Student of the Membrane Technology Group of the University of Twente and Wetsus, Centre of Excellence for Sustainable Water Technology, is the fourth winner of the Marcel Mulder Award. The award of 5000 euro is a remembrance to Prof. Marcel Mulder, who was, next to professor in Membrane Process Technology at the University of Twente, the originator of Wetsus. The prize is awarded annually to a Wetsus researcher that combines scientific excellence with technological a and practical relevance.

In his Ph.D. project, Piotr Długołecki worked on Reverse Electrodialysis (RED), also named Blue Energy. Blue Energy is a process to generate sustainable energy from the mixing of sea and river water. It can be applied everywhere where river water flows into the sea. It is a sustainable, environmentally friendly, renewable source of energy.

Piotr's work was dedicated to the fundamental understanding of mass transport phenomena in the Blue Energy process. The result of his work directly led to a significant increase in the energy output obtainable from Blue Energy. The work combines strong practical relevance with excellent scientific achievements.

The work resulted in 4 publications: 3 in the Journal of Membrane Science and one in Environmental Science & Technology. One other publication is recently submitted. In addition, Piotr is inventor of 2 patents: one on the cleaning of membranes using an electrical field and another one on the use of ion conductive spacers.

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

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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) very soon.

We are looking forward to a fruitful and inspiring ICOM 2011.

Kitty Nijmeijer
Antoine Kemperman
Matthias Wessling

ICOM 2011

Meander Reactor

Name
Elif Karatay

Origin
Turkey

Contact
http://mtg.tnw.utwente.nl
0031 (0) 53489 3674
e.karatay@utwente.nl

Elif Karatay is a new Ph.D. in the Membrane Technology Group. She graduated in 2007 with a Bachelor of Science degree in Chemical Engineering at Middle East Technical University (METU) Ankara – Turkey. Following the graduation, she started her M.Sc. studies in the same university. Her research in Turkey focused on the development of mixed matrix membranes for gas separation. She received her M.Sc. degree in August 2009. Her Ph.D. research project is a collaboration between the membrane technology group and the group of catalytic processes and materials of the University of Twente and aims to perform chemical conversions that are not reasonable without the use of multifunctional micro-reactors. She will focus on gas-liquid contacting membranes and the selective removal of desired products.

Improving hydrodynamics in Reverse Electrodialysis

Name
David Vermaas

Origin
The Netherlands

Contact
http://mtg.tnw.utwente.nl
0031 (0) 58 2843 196
David.Vermaas@Wetsus.nl

David is a new PhD student on Reverse Electrodialysis (RED), also called Blue Energy, where the free energy of mixing fresh and river water is used to generate electrical power. David obtained a MSc degree in Hydrology at Wageningen University. At the Fluid Mechanics laboratory of the Delft University of Technology he studied mixing layers above smooth and rough channel beds. In his Ph.D. project he will focus also on the hydrodynamics, although on a much smaller scale, in Blue Energy. The challenge is to improve the mixing rate and lower the hydraulic resistance and biofouling sensitivity in RED. Davids project is a collaboration between Wetsus (Leeuwarden, NL), centre of excellence for sustainable water technology and the Membrane Technology Group.

Membranes with improved plasticization resistance for olefin-paraffin separation

Name
Jeroen Ploegmakers

Origin
The Netherlands

Contact
http://mtg.tnw.utwente.nl
0031 (0) 53 489 3474
j.ploegmakers@utwente.nl

Jeroen Ploegmakers was born in 1984. In 2008, he received his M.Sc in Analytical Chemistry at the Vrije Universiteit after developing a Comprehensive Two-Dimensional Liquid Chromatography method for the separation of ethylene oxides and propylene oxides at Shell Global Solution in Amsterdam. He recently started as a Ph.D. student in the Membrane Technology Group. His research focuses on the development of new polymer membranes with improved plasticization resistance for gas separation of olefins and paraffins.

Microreactor chips with integrated work-up functionality

Name
Sreenath Kariveti

Origin
India

Contact
http://mtg.tnw.utwente.nl
0031 (0) 53 489 2631
s.kariveti@tnw.utwente.nl

Sreenath Kariveti is a new Ph.D student working in the Membrane Technology Group as well as in the group of Mesoscale Chemical Systems. He obtained his bachelor degree in chemical engineering from JNTU, Hyderabad, India. In January 2007, he started his Masters at I.I.T., Madras, India. His research focused on theoretical and experimental applications of micro-fluidics for different reactions. In September 2009, he started working on his Ph.D project i.e. Microreactor chips with integrated work-up functionality. The major focus of this project is on liquid-liquid extraction. The key challenge is to quantitatively separate emulsions after transfer of the desired component has been completed or after equilibrium between the phases has been established. This will be achieved either by integration of a selective membrane of a nano or microstructured surface "coalescer", which will separate the liquid phases based on their difference in surface tension i.e. based on the wetting properties of the two fluids.

Introducing...

Ion exchange membranes for blue energy

Name
Enver Güler

Origin
Turkey

Contact
http://mtg.tnw.utwente.nl
+31 (0) 53 489 2962
e.guler@utwente.nl

Enver Güler was born in Bulgaria. In 1989 he immigrated with his family to Turkey, where he studied Chemical Engineering at the Ege University in Izmir. During his MSc. he studied the process of reverse osmosis and the removal of boron from reverse osmosis permeate. In 2008, he worked in the Wolfson Department of Chemical Engineering at the Technion – the Israelian Institute of Technology and investigated the effects of halogen based disinfectants on polyamide reverse osmosis membranes. After he obtained his MSc. degree in September 2009, he joined the Membrane Technology Group as a PhD student in October 2009. His project is a collaborative project between the University of Twente and Wetsus, Centre of excellence for sustainable water technology located in Leeuwarden (The Netherlands). His research project is entitled 'ion exchange membrane development for blue energy' and focuses on preparation and development of ion exchange membranes for reverse electrodialysis – a nonpolluting sustainable technology for the production of electrical energy.

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Matias Bikel
December 18, 2009
Phase Separation μ-Molding:
Fundamental Aspects

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Ph.D. Vacancy

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Contact person: Nieck Benes (n.benes@utwente.nl)

More information: http://www.wetsus.nl

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Editors

Matthias Wessling

Kitty Nijmeijer

Zeynep Çulfaz

membrane@utwente.nl

www.membrane.nl

Picture showing membrane with star-shaped structures, by Ikenna Ngene.