(M)Ethanol separation from organic process streams in the chemical industry

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H.M. van Veen *

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H.M. van Veen 7/8/06	P.P.A.C. Pex 7-8-06	ECN Energy Efficiency in Industry
Checked by:	Issued by:	Separation Technology
Y.C. van Delft 14/8/06	P.T. Alderliesten	

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Abstract
The aim of the project was to develop and make available robust and cost effective inorganic membrane technology for large-scale separation of methanol and/or ethanol from other organics in the (petro) chemical industry. This has been done by membrane development and testing, development of membrane transport models, process specific testing and process development, design of modules and test installations and pilot scale testing. Finally a technical and economic evaluation was made.

A model that describes the transport through the microporous membranes was made. Methanol selective pervaporation membranes with high flux and good selectivity can be made based upon modified silica sol-gel chemistry. Recipes for making the modified microporous silica membranes have been transferred to a commercial scale and it was shown that the pervaporation membranes can be made using a robotised technique. Pilot plant testing using a feed mixture taken directly from an industrial process has shown that promising process improvements are possible. For methanol separation from MTBE the payback time, however, is still too long. For the separation of methanol from toluene a payback time of less than 1 year is possible. The membranes can be used up to at least 140^{o}C, though a (reversible) flux decline is observed. After 150 days of continuous use at 125^{o}C in MeOH-MTBE separation the membranes start to show a loss of selectivity. As the stability of the membrane is lower than expected and the membrane producer has changed their strategy towards new technologies with a high added value the commercial production and marketing was stopped. Further improvements towards improving the membrane stability have started.

Keywords
English
Pervaporation, inorganic membrane, membrane development, silica membrane, methanol separation, membrane transport studies, energy saving, process design, scale-up.

Dutch
Pervaporatie, anorganisch membraan, membraan ontwikkeling, silica membraan, methanol scheiding, membraan transport studie, energie besparing, proces ontwerp, opschaling.

Contents

Summary

The aim of the project was to develop and make available robust and cost effective inorganic membrane technology for large-scale separation of methanol and/or ethanol from other organics in the (petro) chemical industry. This has been done by membrane development and testing, development of membrane transport models, process specific testing and process development, design of modules and test installations and pilot scale testing. Finally a technical and economic evaluation was made.

The pervaporation transport through the membranes can be described by a combination of transport through micropores and through a small amount of defects in the membrane. It was shown that methanol selective pervaporation membranes can be made based upon modified silica sol-gel chemistry. These membranes show a high flux and good selectivity in the separation of methanol from organics. Pilot plant testing using a test installation with 1 m^2 of membrane area and with a feed mixture taken directly from an industrial process has shown that promising process improvements are possible. For methanol separation from MTBE the payback time, however, is still too long. For the separation of methanol from toluene a payback time of less than 1 year is possible. Process testing has shown that the membranes can be used up to at least 140^{o}C, though a (reversible) flux decline is observed. After 150 days of continuous use at 125^{o}C in MeOH-MTBE separation the membranes start to show a loss of selectivity.

The modified microporous silica membranes have been scaled up by using commercially available ceramic support tubes with a length of 1 meter. Recipes for making these membranes have been transferred to this scale and it was shown that the pervaporation membranes can be made using a robotised technique. As the stability of the membrane is lower than expected and the membrane producer has changed their strategy towards new technologies with a high added value the commercial production and marketing was stopped. Further improvements towards improving the membrane stability have started.

1. Introduction

An increasing number of industries recognize the possible use of advanced separation techniques to operate plants in an energy efficient and ecological way. It appears that membrane pervaporation will have a very high potential in doing so. Pervaporation is a combination of the words permeation and evaporation and can be considered as the selective evaporation over a membrane. This new separation technique is an alternative for distillation. In distillation, separation takes place by reboiling and condensing the liquid mixture in multiple stages. The desired component(s) can be obtained at a high concentration as top product or bottom product because of the difference in volatility. A large drawback of distillation is the large energy requirement, which is the consequence of evaporating the total liquid mixture at the bottom of a distillation column. In contrast, in pervaporation separation takes place by selective evaporation of only one of the components directly from the liquid feed. Therefore, applying pervaporation instead of distillation could potentially save a significant amount of energy. In some cases separation by distillation is even extremely energy consuming. This occurs when the boiling points of the components are close to each other. In other cases, the liquid and vapour phase have the same composition (azeotrope) and an entrainer (distillation modifying agent) or a multiple column distillation is needed. In these cases a potential energy saving of up to 90% can be reached with pervaporation.

Pervaporation has been developed with polymeric membranes, which have been tested and commercialised in several applications, both for the separation of water from organics and for the separation of methanol from higher organics. Furthermore polymeric membranes can separate (low concentrations of) organics from water streams or recover organics from vapour streams. The limited stability of the polymeric membrane materials under process conditions is the main reason why pervaporation has not been able to replace distillation on a large and process integrated scale and the progress of this technology is slow. Especially handling mixtures with aprotic solvents or high boiling components is difficult with polymeric membranes. These problems could be overcome with inorganic membranes, which are mechanically robust, resistant to aggressive organic solvents and can withstand higher temperatures.

Since a couple of years it is clear that the stability of pure silica membranes for dewatering of organics by pervaporation is limited, as a flux decline in time was observed for both water and the organic component in the feed mixture, even below 100°C. Incorporating methyl groups into the silica structure (MeSi or methylated silica membranes) can almost stop this flux decline. Recent tests have shown that these membranes can now be used in dewatering by pervaporation. However another problem was encountered as the membrane looses its selectivity within a few weeks at 115°C and even within days at 165°C. At the moment the MeSi membranes can therefore be used only up to 95°C in dehydration applications, which is much lower than the originally foreseen temperature of about 250°C. The reason for this seems to be a hydrothermal attack of the membrane layer.

These methylated membranes have shown to be able to separate methanol from higher organics on lab scale. As these process mixtures hardly contain any water the stability problems as seen for the separation of water are not expected. By modifying the membranes it is expected that also ethanol could be separated from organics. Lab scale measurements have shown that these membranes combine high fluxes with good selectivities for the separation of methanol.

2. Project Goals

2.1 English

The aim of the project was to develop and make available robust and cost effective inorganic membrane technology for large-scale separation of methanol and/or ethanol from other organics in the (petro) chemical industry. This has been done by membrane development and testing, development of membrane transport models, process specific testing and process development, design of modules and test installations and pilot scale testing. Finally a technical and economic evaluation was made for three selected processes.

2.2 Dutch

Het doel van het project was het ontwikkelen en beschikbaar maken van robuuste en qua kostprijs aantrekkelijke anorganische membraantechnologie voor de scheiding van methanol en/of ethanol uit organische processtromen in de procesindustrie. Dit is gedaan door uitvoeren van membraan ontwikkeling en testen, maken van membraan transportmodellen, proces specifiek testen en procesontwikkeling, ontwerp van modules en testinstallaties en het op pilot schaal uitvoeren van testen. Als bewijs van een succesvol R&D traject is een technische en economische evaluatie van de membranen in een drietal geselecteerde processen uitgevoerd.

3. Approach and partners

3.1 English

For reaching the goals of the project different tasks and activities have been identified and planned. The main tasks in the project are:

Task 1 Process design
- 1.1 Basic research (F): transport model development
- 1.2 Applied research (I): process overview, development and evaluation

Task 2 Membrane/module development
- 2.1 Basic research (F): membrane development, modification, standard PV testing + preparation for pilot test installation
- 2.2 Basic research (F): module modelling, calculations and development
- 2.3 Fabrication technology (I): membrane development and scale up
- 2.4 Fabrication technology (I): module engineering, design and building

Task 3 Separation tests
- 3.1 Build test installations (F)
- 3.2 Lab scale testing (F): process testing, long-term tests, hybrid PV/distillation installation and testing

Task 4 Implementation/technology transfer

Task 5 Co-ordination

Remark: "F" means fundamental research, "I" means industrial research.

The partners involved, their main expertise and roles in the project are given in the next table.

Partner	Expertise/role
Energy research Centre of the Netherlands, ECN	• research institute • membrane and membrane module R&D • membrane and process testing • project management/coordination
Delft Technical University	• university • process design • membrane transport modelling • testing
Akzo Nobel Chemicals BV	• end-user • membrane process testing • process development
Uniqema BV	• end-user • process development
Quest International Netherlands BV	• end-user • membrane process testing • process development
Cytec Manufacturing BV	• end-user • process development
Lyondell Chemical NL	• end-user • membrane process testing • process development
Purac BV	• end-user • process development

Sulzer Chemtech Netherlands BV	• membrane producer and system builder • scale up of membranes and modules • membrane testing

3.2 Dutch

Om de doelen van het project te behalen zijn er een aantal taken en activiteiten vastgesteld. De belangrijkste taken zijn:

Taak 1 Proces ontwerp
- 1.1 Basis onderzoek (F): transport model ontwikkeling
- 1.2 Toegepast onderzoek (I): proces overzicht, - ontwikkeling en evaluatie

Taak 2 Membraan/module ontwikkeling
- 2.1 Basis onderzoek (F): membraan ontwikkeling, modificatie, standaard PV testen + bereiding voor skid
- 2.2 Basis research (F): module modellering, berekeningen en ontwikkeling
- 2.3 Fabricatie technologie (I): membraan ontwikkeling en opschaling
- 2.4 Fabricatie technologie (I): module engineering, ontwerp en bouwen

Taak 3 Scheidingstesten
- 3.1 Bouwen van test installatie (F)
- 3.2 Lab schaal testen (F): proces testen, lange duur testen, pilot plant schaal testen

Taak 4 Implementatie/technologie overdracht

Taak 5 Coördinatie

Opmerking: "F" betekent fundamenteel onderzoek, "I" betekent industrieel onderzoek

De partners, hun belangrijkste expertise en rol in het project worden in de volgende tabel geven.

Partner	Expertise/rol
Energieonderzoek Centrum Nederland, ECN	• onderzoeksinstituut • membraan and membraan module R&D • membraan en proces testen • project management/coördinatie
Technische Universiteit Delft	• universiteit • proces ontwerp • membraan transport modellering • testen
Akzo Nobel Chemicals BV	• eindgebruiker • membraan proces testen • proces ontwerp
Uniqema BV	• eindgebruiker • proces ontwerp
Quest International Netherlands BV	• eindgebruiker • membraan proces testen • proces ontwerp
Cytec Manufacturing BV	• eindgebruiker • proces ontwerp
Lyondell Chemical NL	• eindgebruiker • membraan proces testen • proces ontwerp
Purac BV	• eindgebruiker • proces ontwerp
Sulzer Chemtech Nederland BV	• membraan producent and systeem bouwer • opschalen van membranen en modules • membraan testen

4. Results and perspective

4.1 Results and project goals reached

Recipes for inorganic methanol selective membranes have been developed based upon methylated silica materials and sol-gel technology. In pervaporation these membranes combine good selectivities with very high fluxes for the separation of methanol from organics like MTBE and toluene but also for water separation from organics. The lab scale recipes have been translated from research size substrate tubes into commercially available substrates (tubes with a length of 1 meter) and it has been shown that these membranes can be scaled up to commercial scale using a robotised technique. Different modules up to a commercial sized isothermal 5 m^2 membrane area module have been made and tested. This is worldwide the most advanced inorganic membrane in combination with membrane modules for pervaporation.

Ethanol selective membranes are available on lab scale but the selectivity is still to be improved

Transport studies and transport model development have shown that the membrane performance can be described by a combination of transport through the micropores of the membrane and through a small amount of defects in the membrane layer. The models can be used to predict the behaviour for the separation of methanol from other organics but there is still some lack in understanding details of the transport phenomena.

In pervaporation the methanol flux and selectivity showed a rather large variation when a substrate tube available on lab scale was used. By upscaling and changing to a commercial substrate tube this variation was strongly reduced. These variations are not important anymore for commercial use, as an average flux will appear when the membranes are used in large modules. Furthermore the ‘robotised’ production will probably rule out these variations even further. The reproducibility of the selectivity of the membranes is still to be improved further. How the membranes are stored is crucial for maintaining the selectivities.

Process testing has shown that the membranes can be used up to at least 140^{o}C for several days in the separation of methanol from MTBE without any problem. During continuous testing at constant temperature in MeOH-MTBE an important methanol flux decline of 50-80% occurs while the selectivity is almost constant. This flux decline occurs during the first few days and after this period the flux is more or less stable. This decline is reversible as the flux can be restored by a temperature increase. Testing continuously for 175 days at 125^{o}C has shown that the selectivity of the membrane is constant up to about 125 days. After this period a gradual selectivity decrease of the membrane is observed and after about 150 days of testing the selectivity is too low. At lower temperatures the membranes can be used without any problem.

Process testing using a pilot plant test installation with 1 m^2 of membrane area has been performed for the separation of about 900 litre of a methanol-MTBE mixture. This testing was performed at the location of an end-user (Lyondell) using a feed liquid directly from the MTBE production plant. Very good selectivities and fluxes have been obtained on this scale and the separation goals (reducing the methanol content in the feed from about 24 wt.% to less than 1 wt.%) have been reached. By this testing it has been proven that the membranes can be made on a pilot scale with the same quality as on lab scale. For the separation of methanol from MTBE a cost reduction in the MTBE production process of 100-200 k€/year is possible because of energy savings as compared with the existing distillation process. However, the payback time is in the order of 10 years, which is too long for the end-user.

The membranes have been used to evaluate the replacement of an existing methanol-toluene separation process based upon extraction and distillation. In the separation of methanol from toluene investment costs for the membrane-based process are about 30% higher than for the extraction-distillation based process. The variable operating costs are 60% lower for the membrane process, mainly due to the much lower energy use of pervaporation. This leads to a payback time of less than 1 year, which is very good. The energy use is about 3 GJ/ton methanol produced for the pervaporation process compared with 11 GJ/ton for the extraction-distillation process.

For other applications like high temperature trans-esterifications and mild separations in the food and flavour industry the membrane quality has to be improved with respect to selectivity. For the moment the quality of the tested membranes was not good enough to perform detailed process calculations. For optimisation of the production of lactic acid ethanol selective membranes will have to be used. When a membrane can be made with an ethanol flux of 1 kg/m^2h a payback time of less than 1 year is possible. High temperature trans-esterifications at e.g. 200-250°C are not possible with the existing membranes as the membrane stability is not high enough.

In the project manufacturing procedures for membrane layers were transferred from lab-made support tubes to commercial (cost-effective and already large scale available) support tubes. Obviously it was not trivial and easy to obtain thus membrane tubes ready for production with similar performance as the membrane that was totally lab-made. Final successful transfer was a significant step forward for the membrane producer. The lower quality membranes that were available in the meantime were experienced as a step backwards by the end users. This should be kept in mind in possible future choices or needed changes. There is a general feeling that the membranes do have potential. From an R&D point and membrane production technology transfer the project was very successful, though more R&D focussed towards stability and reproducibility is needed together with more fundamental insight. The end-users in this project have obtained a better view of the possibilities of the membranes and would like to be involved as a user group rather than partners in further developments, as they have the feeling that they have participated too early with maybe too high expectations in this project.

4.2 Commercial perspective

As planned in other projects/co-operations between Sulzer and ECN, the first focus was on silica membranes for dewatering applications. Main strategy behind the silica membrane development by Sulzer was an expansion of the market. The membranes were expected to be used at higher temperatures than the existing polymeric membranes that can only be used up to 120°C maximum. The main application of these membranes was high temperature separation of water from organics and to separate water from organic media that are too aggressive or too difficult for existing polymeric membranes. Following that strategy, the methylated silica (MeSi) membrane production for methanol separation would be the next step in the marketing sequence. As the methylated silica membrane is also effective in separating water from organics it has been decided only to produce the MeSi membrane. During the project the methylated silica membrane was scaled up to a commercial production size. Obviously it was not trivial and easy to obtain membrane tubes ready for production with similar performance as the membrane that was totally lab-made. Final successful transfer was a significant step forward for the membrane producer.

For dewatering applications the stability of the membrane was, however, lower than expected and this could not be solved within the foreseen time period of getting a reliable marketable product. Furthermore Sulzer Chemtech as membrane producer has decided to change their strategy from new technologies with high added value (like the membranes developed in this project) to well known techniques that are proven and already accepted by the market.

Therefore commercial production and marketing of the inorganic pervaporation membranes was stopped. It has, however, been shown that these membranes can be produced on large scale with rather good pervaporation characteristics in the production plant of Sulzer. Pilot scale testing has shown good results. This is word-wide the most advanced inorganic membrane for molecular separations in combination with the developed isothermal membrane module for pervaporation.

The membranes are to be used in the separation of methanol or ethanol from higher organics. The end-users Akzo Nobel Chemicals BV, Uniqema BV, Quest International Nederland BV, Cytec Manufacturing BV, Lyondell Chemical NL, and Purac BV all have different processes towards which the membrane and membrane processes have been developed. These processes need stable and reliable membranes and membrane performances. By lab scale testing and process calculations it was shown that several of the process demands could be met and a feasible process can be obtained. However, the performance of the membranes is still fluctuating between different batches of membranes.

4.3 Overview of all project reports open to the public (chronological order)

[1] Pex, P.P.A.C. (2002): *Ontwateren van organische processtromen d.m.v. pervaporatie*. Presentation at the symposium '11 jaar Membraangekte' organised by the Nederland's Membraan Genootschap, 11 June 2002, Ede.

[2] Pex, P.P.A.C. (2002): *Dewatering of organics by pervaporation with silica membranes*. Contribution to the Summerschool 'Membrane product & Membrane process development', organised by the European Membrane Society, 1-6 September 2002, Enschede.

[3] Veen, H.M. van and P.P.A.C. Pex (2003): *Recente ontwikkelingen op het gebied van pervaporatie met anorganische membranen*. Presentation for NL-GUTS at Lyondell in the Botlek, 16 January 2003.

[4] Veen, H.M. van, Y.C. van Delft, W. Bakker, C. Chau, S. Sommer and P.P.A.C. Pex (2002): *Energy savings by pervaporation with silica membranes*. Poster presented at the 7th International Conference on Inorganic Membranes (ICIM7), the 5th International Conference on Catalysis in Membrane Reactors (ICCMR5), both in Dalian, China, 23-28 June, 2002 and the International Conference on Membranes (ICOM), Toulouse, France 8-12 July 2002.

[5] Bruijn, F.T. de, L. Sun, Ž. Olujić, P.J. Jansens and F. Kapteijn (2003): *Support layer effect on the flux through ceramic pervaporation membranes*. Preprints of the ECCE-4 conference, Granada 2003.

[6] Bruijn, F.T. de, F.W. Oskamp, L. Sun, Ž. Olujić, P.J. Jansens, F. Kapteijn: *Support layer resistance of a composite silica membrane for separation of methanol-MTBE mixtures*. Preprints 9. Aachen Membrane Colloquium, Aachen 2003.

[7] Bruijn, F.T. de, L. Sun, Ž. Olujić, P.J. Jansens, F. Kapteijn (2003): *Influence of the support layer on the flux limitation in pervaporation.* Journal of Membrane Science 223 (2003) 141-156.

[8] Bruijn, F.T. de, Ž. Olujić, F. Kapteijn and P.J. Jansens (2004): *Mass-transport in methylated silica membranes for the separation of organic mixtures*, oral presentation, proceedings 8th Int.Conf. on Inorganic membranes, p. 234-237, Cincinnati, July 18-22, 2004, Ed. F.T. Akin and Y.S. Lin.

[9] Veen, H.M. van, Y.C. van Delft, B. Bongers, C.W.R. Engelen and P.P.A.C. Pex (2004): *Methanol separation from organics by pervaporation with modified silica*

membranes. Keynote lecture, proceedings 8th Int.Conf. on Inorganic membranes, p. 266-270, Cincinnati, July 18-22, 2004, Ed. F.T. Akin and Y.S. Lin. Also submitted and accepted as poster presentation at the Euromembrane conference, 28 September-1 October 2004, Hamburg.

Fleur Oskamp of the TU Delft has obtained the Unilever Research price for her MSc-graduation work on methanol-MTBE separation with silica membranes.

5. Outcome indicators

The project strongly contributes to the EET programme goals. The output indicators that have been formulated in the project plan will be discussed below per item.

5.1 Economy

- The Dutch industry is recognised to be among the ones playing a leading role worldwide. In this project new unit-operations have been developed that are expected to play a significant role in both the process industry (Akzo Nobel, Uniqema, Quest, Cytec, Lyondell and Purac) and the supplier industry (Sulzer Chemtech). Also the membrane producing industry and suppliers of raw materials will benefit from this. All can then take a leading role in the field of process intensification, process optimisation, new process equipment, and membrane production.
- The pervaporation/vapour permeation technology based upon inorganic membranes is a very new technology in a rather conservative market. Even though, first commercial results are to be expected within 7 years from the start of the project. The market will only accept this new technology when it is proven to be robust, reliable and cheap. The involvement of knowledge providers, suppliers and end-users has guaranteed that all available results and technology has been transferred.
- For the silica membrane production for dewatering by pervaporation a first plant of Sulzer was started mid 2003. The methylated silica membranes for organic-organic separation would be the natural follow-up of this. The commercial production of the membranes that was initiated during the project was, however, stopped. Future new commercial activities will be in membrane production, building new process equipment, and by implementing the membranes making new, cheaper and better products.
- When 1/5 of the investments in distillation in the Netherlands will be replaced by membranes the turnover will be larger than 100 million Euro/year. World-wide this will be a multi billion Euro business.
- The expected market share in the year 2010 in the Netherlands is expected to be about 3.4 million Euro. At the top of the market (about 2015-2020) the market share will be about 40 million Euro. The spin-off market in e.g. nanofiltration could be as large as the pervaporation market.
- Several processes and also the development of new products (e.g. esters) are now on the edge of economic feasibility. Because of the increased efficiency of the separation processes, especially in combination with chemical reactions, these processes or products will become profitable.
- The investment costs for a pervaporation installation strongly depend upon the size of the installation/ application. In general payback times from 1-4 years are obtained.
- In the food industry new and/or higher quality products will be possible because of the new technology of mild isolation and concentration. Membranes, however, have to be further improved for reaching this goal.
- As a spin-off, the membranes that have been developed in the project can be used in nanofiltration applications. This will have a big impact to the (petro) chemical industry as well.
- During the project the amount of MeOH separation applications has not grown significantly. There is a strong interest in water separation as well.

5.2 Ecology

- Compared to thermal separation processes nowadays used, like conventional distillation, is it expected that the pervaporation technology would at least save 50% energy on unit operation level. The energy saving potential in the Netherlands is about 2 PJ/year in NL and 60 PJ/year worldwide for methanol separation. When water separation from organics is included these numbers are 7 and 240 PJ/year respectively.
- The use of the membranes in nanofiltration applications could lead to savings as large as those for pervaporation. The membranes can also be used in gas separation and especially hydrogen separation when there is no need for very high purity hydrogen or in a combination with further purification techniques can be made.
- Several distillation processes use either extra chemicals or more than one column to separate azeotropes. Membrane pervaporation is not hindered by azeotropes and compared to azeotropic distillation more than 70% reduction in energy use is possible. Additionally the toxic chemicals that are used in azeotropic distillation are not needed anymore.
- By combining the pervaporation technology with chemical reactions (process intensification), the processes will be optimised leading to higher conversions of the feedstock and higher selectivities towards the product. This means, products formed against lower costs and with less by-products. The effectiveness of the separation will increase leading to smaller installations. Because of this the investment and maintenance costs and emissions of hydrocarbon and/or toxic components will be reduced.
- Because of the separation possibilities the solvents methanol and ethanol can be recycled and reused leading to i) less waste streams (burn, dispose, or treat), ii) less production of these chemicals, and iii) less transport needed.

5.3 Technology

- It is expected that the first commercial implementation of the inorganic membranes for (M)ethanol separation will be before 2010. This means that the membranes are being produced and membrane modules and installations are available and running in process streams in the process industry.
- A main technical risk could be the stability of the membranes in aggressive, especially strong acidic and alkaline, conditions. First development and implementation should therefore focus towards applications were polymeric membranes cannot be used because of their temperature or low organic solvent resistance, but that are still relatively mild with regard to pH and temperature.
- A non-technical risk is laying in the fact that Sulzer Chemtech GmbH stopped commercialisation of ceramic pervaporation membranes. This throws back the possibilities for implementing the technology. New partners should be found for commercialisation.
- Distillation has proven to be reliable and robust for more than 50 years and has a broader applicability than membranes, though membranes offer a potential for process improvement, better economics and energy savings.