Methanol Separation from Organics by Pervaporation with Modified Silica Membranes

H.M. van Veen, Y.C. van Delft and P.P.A.C. Pex

Energy research Centre of the Netherlands ECN,

Unit Energy Efficiency in Industry
P.O. Box 1, 1755 ZG Petten, the Netherlands

Background

- Microporous silica membranes can be used for water separation from organics by pervaporation.
- By modification with methyl groups these silica membranes can be made selective towards methanol as well.
- Large energy savings expected compared to distillation.
- Testing and process evaluation of MeOH separation from toluene.

Objective

Improve energy efficiency in industry by replacing/combining distillation processes with inorganic pervaporation membranes: what is the potential of methylated silica (MeSi) membranes in MeOH separation?

Membranes

- Alumina porous substrate: ID=10 mm, OD=14 mm, L=1 meter
- Apply MeSi layer by sol-gel processing
- Pore size active layer: 0.3-0.5 nm

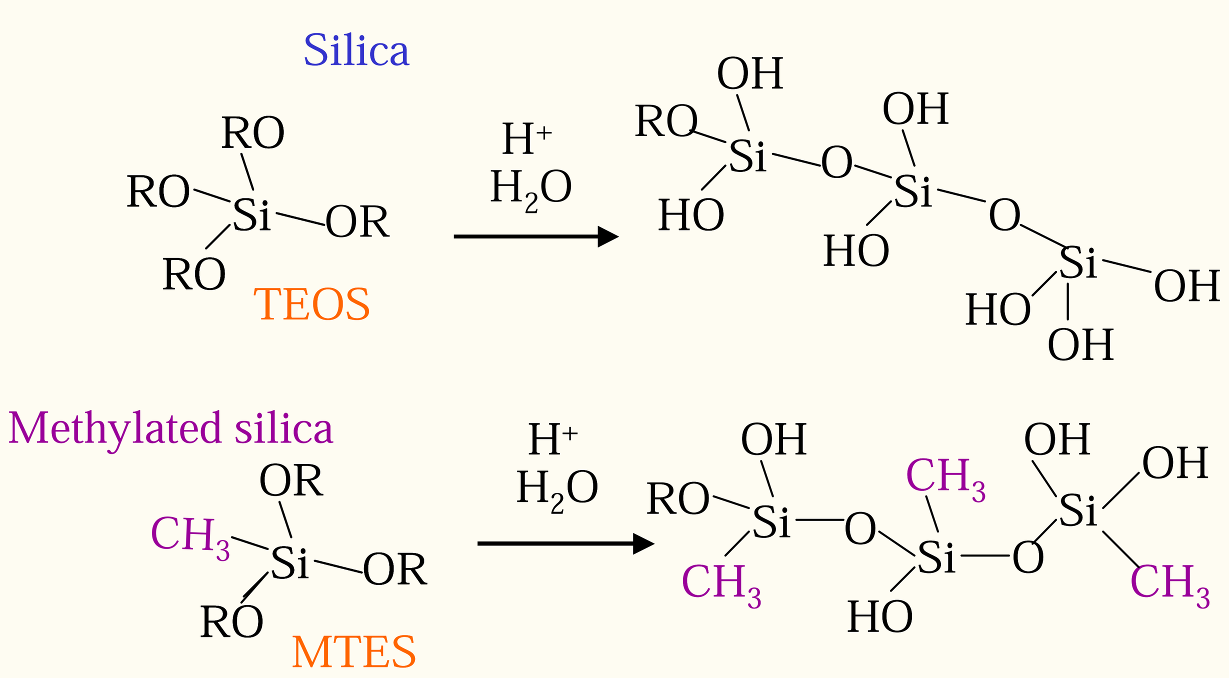

Figure 1: Silica and MeSi sol structure

Lab scale pervaporation test results

Mixture	MeOH feed conc. (wt.%)	Total flux (g/m^2h)	Permeate conc. MeOH (wt.%)
MeOH pure	100	1620	100
MeOH - EtOH	5	270	47
MeOH - 1-propanol	5	130	90
MeOH - n-butanol	5	150	92
MeOH - MTBE	5	370	90
MeOH - toluene	5	1230	97

Table 1: Lab scale testing MeSi membrane: area appr. 50 cm², T=45°C and 10 mbar permeate pressure

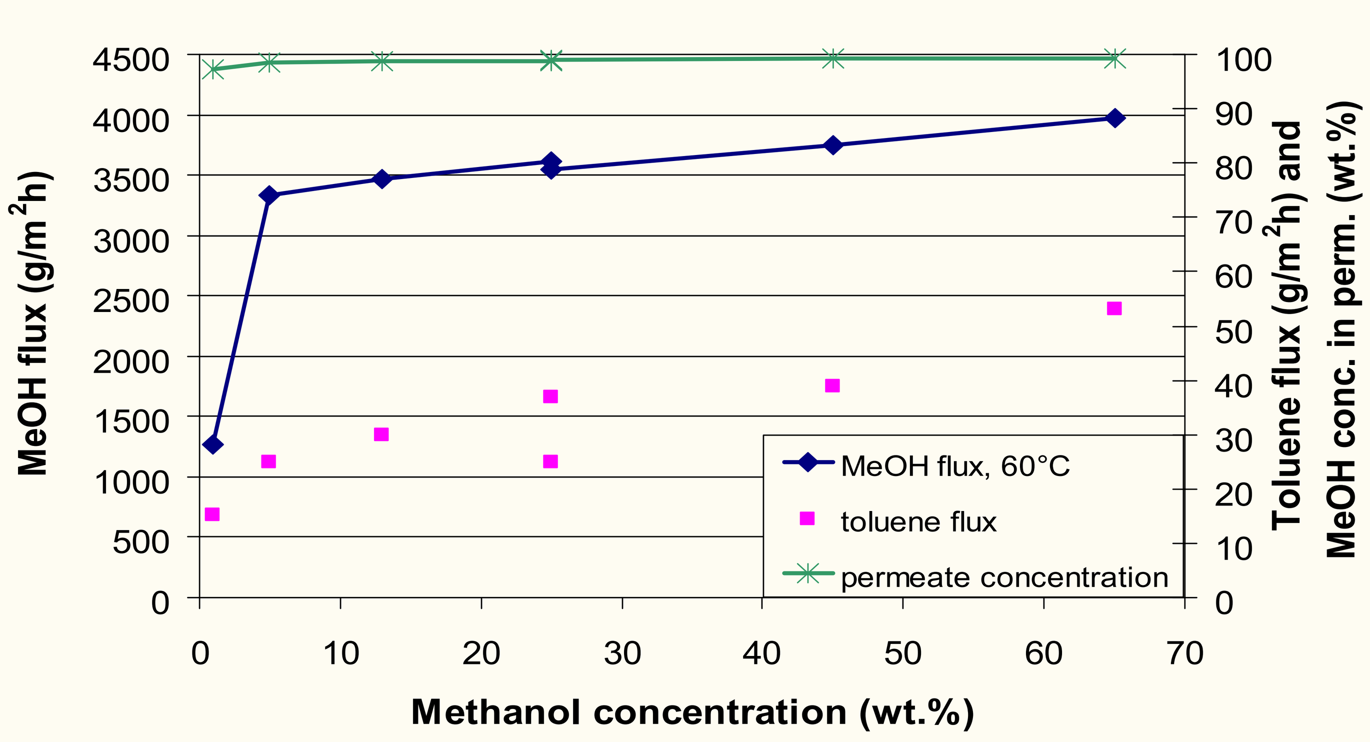

Figure 2: Influence of feed concentration on flux and permeate purity for MeOH-toluene, T=60°C, permeate pressure=10 mbar

For MeOH-toluene, the permeate contains > 97 wt.% MeOH even at 1% MeOH in the feed. When the MeOH flux is plotted vs. the (real) driving force, a straight line through the origin is obtained.

Techno-economic evaluation of MeOH-toluene separation

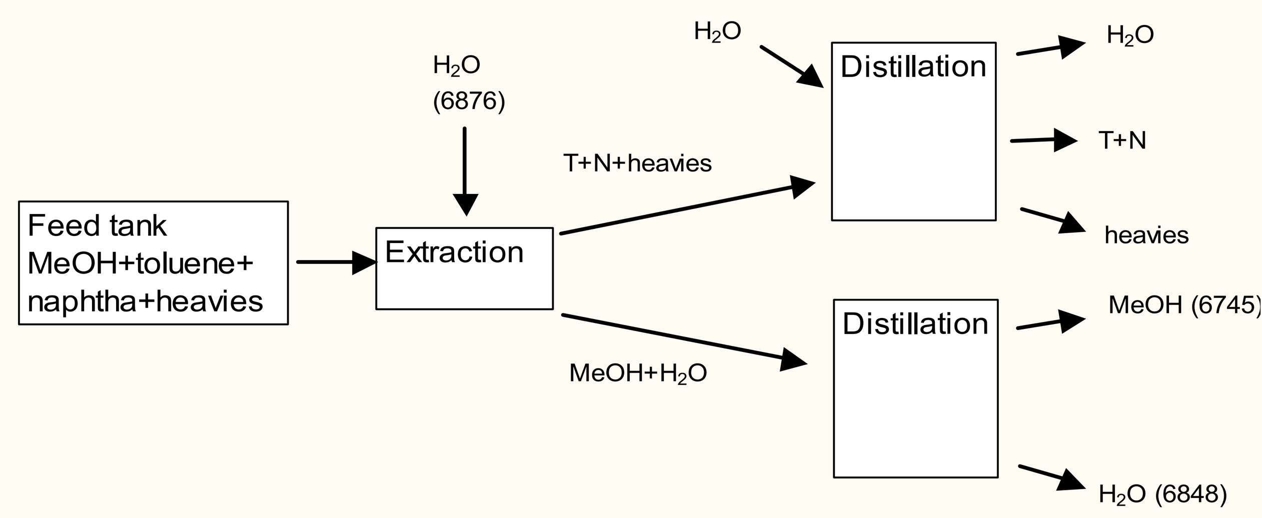

Figure 3: Existing MeOH-toluene process (flow numbers are in kg/h)

Feed:	MeOH 50-80%, toluene 10-40%, aliphatic solvents <10%, salts, organic amines, high boilers, water
Demands:	Methanol phase: water and toluene < 0.5% Toluene phase: MeOH < 0.5%
Aim:	Reduce costs, load to distillation column and energy consumption by replacing water extraction by pervaporation

Process calculations using test data, ASPEN+ flowsheeting and costing:

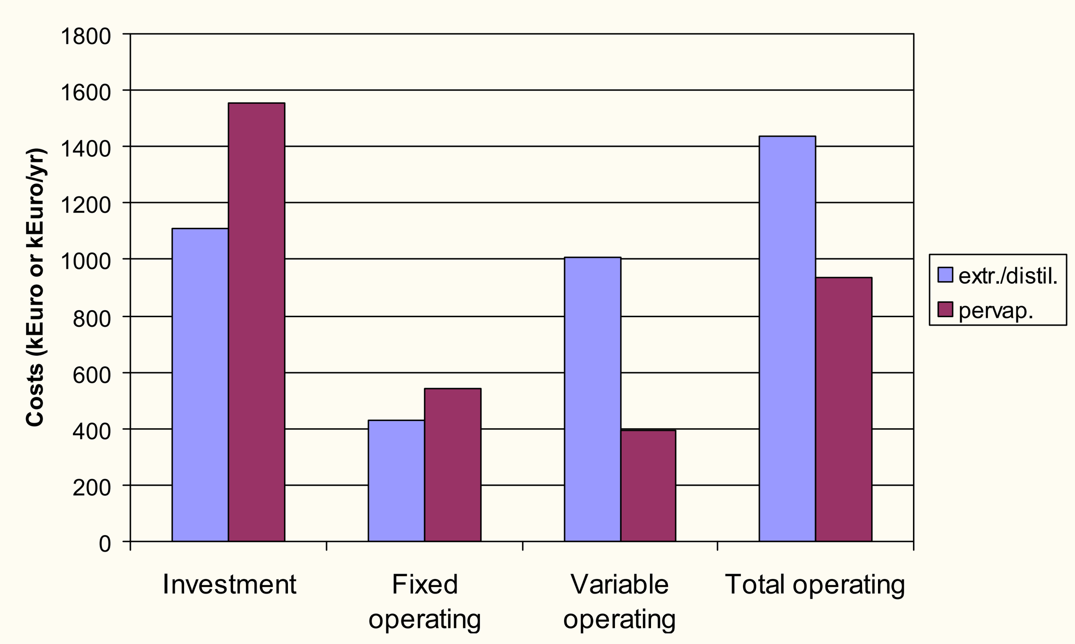

Figure 4: Cost comparison of conventional extraction/distillation vs. pervaporation

Pervaporation leads to 80% less energy use: return on additional investment less than 1 year. Pilot scale testing will be started:

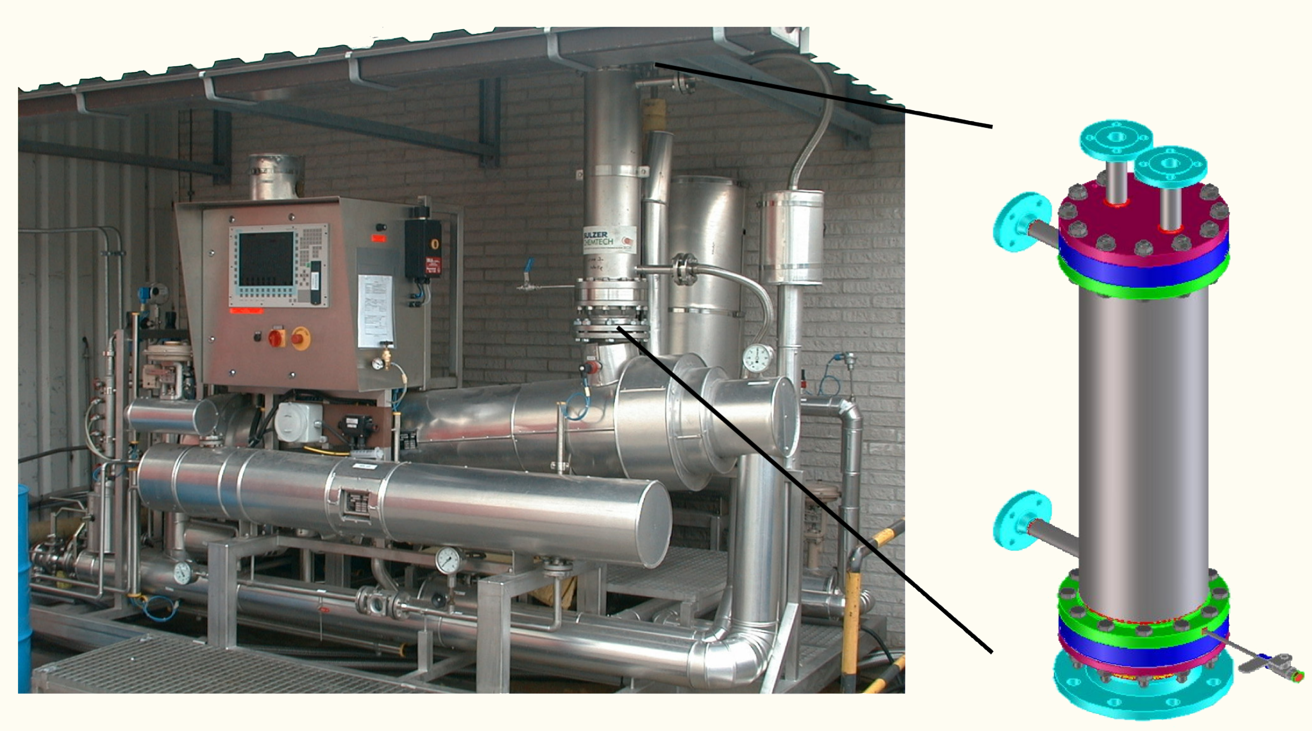

Figure 5: Pilot plant PV/VP installation for on-site testing (A_{mem}= 1 m² (24 tubes of 1 meter length), T_{max}=150°C, P_{max}=10 bar, batch $feed_{max}$=1000 l or continuous processing)

Conclusion

Methylated silica membranes can be used for methanol separation from organics. Pervaporation processes based upon these membranes can very well compete with distillation-based processes or at least significantly debottleneck these processes.

More information

Please contact: Henk van Veen
Tel. +31 (0) 224 564606
Fax. +31 (0) 224 568615
E-mail: vanveen@ecn.nl

ECN
energy innovation

We gratefully acknowledge the financial support by the Dutch programme bureau EET, Energy, Ecology and Technology.
Frans van Schoote of Cytec is gratefully acknowledged for his input in the process evaluation.

PS-P-04-050