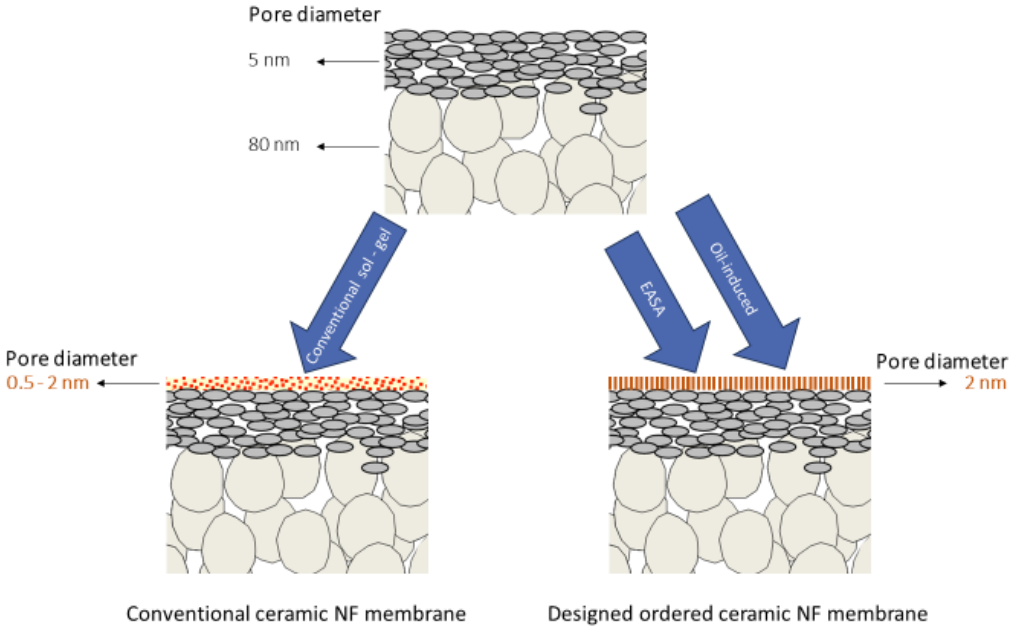
17th International Conference on Inorganic Membranes
New Frontiers for Inorganic Membranes
July 1 - 5, 2024
Oceania Convention Center - Florianópolis - Brasil

Synthesis techniques for ordered mesoporous organosilica deposited on porous substrates

A.D. Borger[a,*], A. Nijmeijer[a], J.E. ten Elshof[b] and M.A. Pizzoccaro-Zilamy[a,c,**]
[a] Inorganic Membranes group, University of Twente, Enschede, The Netherlands
[b] Inorganic Material Science group, University of Twente, Enschede, The Netherlands
[c] Gas Separation Membrane division, Materials Synthesis and Processing (IEK-1) Forschungszentrum Jülich, Germany

*a.d.borger@utwente.nl,**m.d.pizzoccaro@utwente.nl

The stability and chemical resilience of nanofiltration (NF) ceramic membranes are well-known. These qualities make ceramic membranes ideal for separations under challenging industrial conditions in both aqueous and non-aqueous streams [1]. Typically ceramic NF membranes present a high tortuosity as the mean path of the solvents traveling through the membrane is longer than the thickness of the membrane. To reduce this path length, pores ordered and oriented perpendicularly to the membrane surface are desired to increase the solvent transport rate through the membrane as compared to conventional membranes with a similar thickness and pore size [2].
In this presentation, two synthesis approaches to prepare ordered mesoporous silica and organosilica thin films with oriented pores will be compared in terms of reproducibility and resulting pore diameter. The ordered pores were fabricated on porous substrates of either silica or hybrid silica as the membrane material and cetyltrimethylammonium bromide (CTAB) as the structure directing agent [3].
First, the oil-induced co-assembly method using dodecane as the co-structuring agent was explored. This method resulted in a thin layer (< 50 nm) with a measured pore diameter similar to the sizes reported in [4]. However, the synthesis method is prone to defects. The influence of various processing parameters on the ordering of the pores will be discussed using a set of porous analysis techniques and a new formation mechanism different from the one reported in [5] is proposed.
Second, preliminary results obtained via the electrochemically assisted self-assembly method will be presented. This method uses potential-controlled surface aggregation of the structure directing agent [6]. Combined with the film growth originating from the electrode surface, this method is expected to lower the rate of defect formation of the ordered mesoporous layer [6].
In conclusion, ordered mesoporous silica thin films were deposited on porous supports. Both synthesis methods explored resulted in thin films with pores in the nanofiltration-low ultrafiltration range. Either of the methods showed requirements in the supports for the successful deposition of these thin films.



[1] R. Merlet et al., “Comparing the Performance of Organic Solvent Nanofiltration Membranes in Non-Polar Solvents,” Chem Ing Tech, vol. 93, no. 9, pp. 1389–1395, Sep. 2021, doi: 10.1002/cite.202100032.

[2] J. Yang, G. S. Lin, C. Y. Mou, and K. L. Tung, “Mesoporous Silica Thin Membrane with Tunable Pore Size for Ultrahigh Permeation and Precise Molecular Separation,” ACS Appl Mater Interfaces, vol. 12, no. 6, pp. 7459–7465, Feb. 2020, doi: 10.1021/acsami.9b21042.

[3] X. Ma et al., “Uniform and ordered periodic mesoporous organosilica thin films by a facile solution growth approach,” Journal of Porous Materials, vol. 25, no. 2, pp. 489–494, Apr. 2018, doi: 10.1007/s10934-017-0460-8.

[4] K. C. Kao and C. Y. Mou, “Pore-expanded mesoporous silica nanoparticles with alkanes/ethanol as pore expanding agent,” Microporous and Mesoporous Materials, vol. 169, pp. 7–15, Mar. 2013, doi: 10.1016/j.micromeso.2012.09.030.

[5] K. C. Kao, C. H. Lin, T. Y. Chen, Y. H. Liu, and C. Y. Mou, “A General Method for Growing Large Area Mesoporous Silica Thin Films on Flat Substrates with Perpendicular Nanochannels,” J Am Chem Soc, vol. 137, no. 11, pp. 3779–3782, Mar. 2015, doi: 10.1021/jacs.5b01180.

[6] A. Walcarius, “Electroinduced Surfactant Self-Assembly Driven to Vertical Growth of Oriented Mesoporous Films,” Acc Chem Res, vol. 54, no. 18, pp. 3563–3575, 2021, doi: 10.1021/acs.accounts.1c00233.