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Title:

Determining wetted membrane microstructures using ESEM and DIC microscopy and their implications to membrane performance

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Abstract: (Your abstract must use **Normal style** and must fit in this box. The abstract should be written in English and should be around 1000 words long, i.e., 2 pages excluding title, authors & affiliations, keywords and references. Authors are encouraged to show important figures/tables etc in the abstract so as to make a full presentation.)

The fabrication of most polymeric nanofiltration membranes is currently more of an 'art' than a 'science', as there is no established relationship linking the fabrication variables, material characteristics when in use (or even wetted) with the performance (separation) characteristics. Consequently, the overall objective of this project is to determine the relationship between nanofiltration membrane fabrication variables, the resulting membrane microstructure when wetted and the characteristics of the separation (flux and rejection) that result from that microstructure.

Polyimide nanofiltration membranes intended to be used for organic solvent nanofiltration are the main focus of the current work. Such membranes have been found to be effective for applications such as: the separation of solvents from lube oil and separation of aromatics, recycling homogeneous catalysts and bases, separating chiral diastereomers and green organic synthesis. The membranes in this study were cast by phase inversion from P84 co-polyimide (HP polymer GmbH) at 250 μm thickness on a non-woven backing layer, varying formation parameters (dope solvent, solvent evaporation time, polymer concentration and post formation heat treatment temperature). The innovation in this work is in using wetted microstructural techniques to determine the macroscopic and microstructural changes of the membranes both from different casting conditions, wetted in different solvents before and after use, primarily using an environmental scanning electron microscope (ESEM; FEI Quanta 200F) and differential interference contrast (DIC) microscopy (Nikon Eclipse 80i, Nikon Instruments Inc., NY), with sectioning for the latter using a sledge microtome (Leica Microsystems Model SM2000R, Germany). This is compared to typical 'dry' membrane analysis by Field Emission Scanning Electron Microscopy (SEM; Philips XL30S Field Emission Gun).

Both ESEM and DIC imaging showed clear differences between dry and wetted microstructures that correlate to membrane formation parameters. When saturated in different organic solvents, these membranes exhibit considerable structural swelling that significantly changes the microstructure (Figures 1 and 2) compared to the dry membrane microstructures (e.g. Figure 1a and 3).

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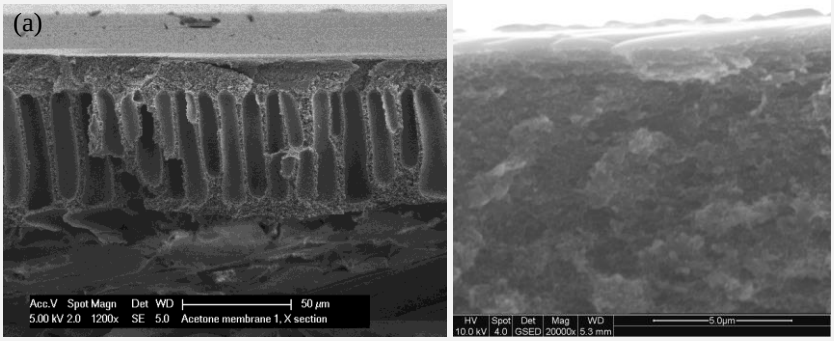

Figure 1: Membrane (25wt% P84 + 65wt% NMP+ 10wt% acetone, 60s evaporation time) imaged (a) dry by SEM, and (b) wetted after soaking in ethanol in the ESEM, where a more gelatinous microstructure is revealed.

Figure 2: Imaging by DIC microscopy in acetone, showing the difference between evaporation times in the microstructure. Both membranes are from 25 wt% P84, 75wt% NMP, with evaporations: (a) 0 sec, (b) 60 sec.

DIC microscopy revealed structural details not readily apparent in typical dry SEM and transmission electron microscopy membrane images. In particular, it allows boundaries between microstructurally different regions to be more readily determined. Importantly, the technique can be used with different solvents at normal pressure (water and acetone were tested) allowing for the first time a vivid visualisation of the differences in microstructure these solvents create.

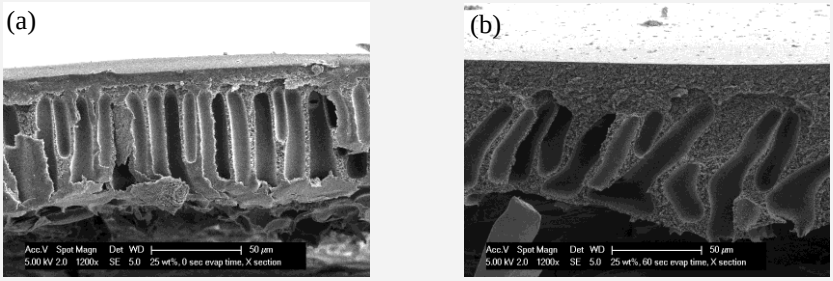

Figure 3: Traditional dry SEM images showing the effect of evaporation time on the microstructure, Both membranes are from 25 wt% P84, 75wt% NMP, with evaporations: (a) 0 sec, (b) 60 sec.

Separation (flux and rejection) was assessed using dead-end filtration (Sterlitech HP4750 stirred cell) using Rose Bengal in ethanol at 30 bar nitrogen pressure. Results correspond to the differences in these microstructural regions, with larger top and

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transition layers in particular corresponding to lower fluxes. Rejection could not be correlated with microstructural regions nor the formation parameters studied – further work is therefore required utilising molecular weight cut-off curves.

Overall these results indicate that since wetted membrane microstructures are significantly different to the dry microstructures (that are also typically used in membrane characterisation), techniques such as ESEM and DIC microscopy should be more widely used to guide bespoke membrane design and provide further insight into the structural factors defining membrane performance, in particular when trying to reconcile performance variations in different solvents.