



Influence of different solvent and time of pre-treatment on commercial polymeric ultrafiltration membranes applied to non-aqueous solvent permeation

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ABSTRACT

Several pre-treatments and solvents were tested through permeation in commercial polymeric membranes (UH004, UP005, UP010 and UH050 – Microdyn-Nadir) with different molar mass cut-offs (4 kDa, 5 kDa, 10 kDa and 50 kDa, respectively), in order to evaluate their efficiency and stability through non-aqueous solvent permeation. After pre-treatments and permeations, membranes were characterised with contact angle, Fourier Transform Infrared Spectroscopy (FTIR), and Field Emission Scanning Electronic Microscopy (FESEM) to evaluate structural integrity. The results of solvent permeation suggest that selectivity of the polymeric matrix depends not only on pore size, but also, in great extent, on the interaction between solvent and polymer. A strong relation can be noted between the conditioning length and permeability for ethanol pre-treatments. Permeability to *n*-hexane increased from 4 to 18 times after pre-treatment, depending on the time of exposure to ethanol and *n*-hexane. Characterisation analyses show no significant changes on the membranes surface. In some cases, discrepancies observed among permeate fluxes and contact angles might be an indicative of the occurrence of swelling and plasticisation. However, results suggest the feasibility in the use of these membranes for the recovery of solvents in the oil industry, if suitable process parameters are chosen.

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1. Introduction

The use of polymeric membranes is already a feasible technology for water and aqueous solutions treatment. Although there are many studies on the preparation of solvent resistant membranes, only few are commercially available as Borsig, PolyAn, Duramem[®], Puramem[®] and SolSep[®] membranes. Most of the commercial available polymeric membranes are produced for aqueous systems

and they present some drawbacks to industrial implementation in non-aqueous solvent-based processes. The problems are generally related to membrane lack of stability, due to the changes caused by the solvents in its structure, and low permeate fluxes [1–3]. Therefore, studies in this field could benefit industry segments from edible oil processing to pharmaceuticals and petrochemical for its reduced energy consumption and process simplification.

Many authors suggest that the pre-treatment of polymeric membranes by immersion in organic solvents can prevent the pore collapse during permeation and enable the solvent entrance into the pores. This enhances the

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permeate flux and promotes better stability to non-polar solvents. Besides, the conditioning step can also change the surface polarity by grouping hydrophilic and hydrophobic sites, improving the permeability to organic solvents [4–7]. In addition, according to Coutinho et al. [8] and Yang et al. [9] studies, the efficiency of membranes in organic solvent permeation is not only function of pore size (size exclusion), but also function of chemical interactions between the solute and/or solvent and membrane active layer.

Several pre-treatment solvents and pre-treatment times have been proposed for different polymeric membranes. Firman et al. [10] flooded the membranes with pure solvents of decreasing polarities (ethanol, *iso*-propanol, *n*-hexane) for 24 h. Araki et al. [7] used conditioning times of 30 min in *n*-hexane before its permeation in polymeric ultrafiltration (UF) membranes (PVDF – polyvinylidene fluoride, PES – polyethersulfone, PC – polycarbonate, CME – mixed cellulose esters). Darvishmanesh et al. [11] used immersion treatments in various organic solvents for one week before permeation in nanofiltration (NF) membranes produced with polyamide (PA). Van der Bruggen et al. [5] also reported treatments of 24 h by immersion in ethanol and *n*-hexane in their study with NF membranes, and conclude that the permeate fluxes can be influenced by the pre-treatment solvent.

However, to the best of our knowledge, membrane pre-treatments were evaluated concerning solely solvent immersion, without mentioning the influence of the binomial solvent and exposure time. Therefore, the present work aims to evaluate the permeability of different solvents (ethanol, *iso*-propanol and *n*-hexane) and the influence of these solvents and time of exposure in the permeation of *n*-hexane through four commercial polymeric membranes with different molar mass cut-offs (MMCO). Permeate fluxes of the pure solvents were also investigated as well as surface hydrophilicity and structural changes.

2. Materials and methods

Permeation experiments were carried out in a bench scale dead-end stainless steel filtration unit. The flat sheet membranes tested were from Microdyn-Nadir, and are listed in Table 1. The effective surface area was 9.08 cm² and the working volume of the cell was 250 cm³.

Membranes were submitted to different pre-treatments. The experimental procedure for all the membranes initiated with immersion in ethanol for 15 min to remove preservatives and fill the pores with the solvent. Then, the conditioning started with immersion in the solvent (deionised water, ethanol, *iso*-propanol or *n*-hexane) at different exposure times. Each treatment was performed with a new membrane. Permeate fluxes are an average of five measurements with the same membrane sheet at each pressure. All the experiments were carried out at least in duplicates. All solvents used in conditioning were analytical grade.

All the assays started with membrane compaction at 8 bar for 45 min, after which, permeation was carried out

Table 1

Manufacturer's data for the membranes tested.

Membrane	UH004	UP005	UP010	UH050
Material	PESH	PES	PES	PESH
MMCO (kDa)	4	5	10	50
T max (°C)	95	95	95	95
pH range	0–14	0–14	0–14	0–14
Retention (%)	92–99 ^a	91–98 ^a	63–85 ^b	75–88 ^c

PESH – Hydrophilic polyethersulfone.

PES – Polyethersulfone.

^a Tested solute – Dextran 10.

^b Tested solute – PVP K17.

^c Tested solute – PVP K30.

with pressure variation from 1 to 6 bar, in intervals of 0.5 bar. The temperature was maintained at 25 °C.

For permeation with the treatment solvent (Fig. 1), membranes were pre-treated with the respective solvents for 2 h. Then, permeation assays were performed by the filtration of the solvent used in the conditioning. For *n*-hexane permeation (Fig. 2), the conditioning consisted in sequential immersion of each membrane in solvents whereas the first is either ethanol or *iso*-propanol and the second *n*-hexane, for 2 or 12 h. Figs. 1 and 2 summarise the experimental strategy used in the study.

Both new and used membranes were characterised to check for the structure's integrity and changes in hydrophilicity by contact angle measurements (sessile drop method, Ramé-Hart Instrument Co., USA), Fourier Transform Infrared Spectroscopy with Attenuated Total Reflection (FTIR-ATR, Frontier, Perkin Elmer, USA) and Field Emission Scanning Electronic Microscopy (FESEM, JEOL JSM-6701F, Japan). All the characterisation analyses were performed with membranes dried at room temperature for at least 72 h.

3. Results and discussion

3.1. Solvent permeability evaluation

The measurement of pure solvent (water, ethanol, *iso*-propanol and *n*-hexane) permeate fluxes through pre-treated membranes allowed the calculation of permeability (Table 3), as well as the evaluation of membrane performance in contact with the solvents. Since both pressure and solvent can influence the permeate flux, the values for these parameters can be chosen to increase the efficiency of the filtration process [6]. Permeabilities were normalised by the viscosity of each solvent for better assessment of other factors that may have influenced membranes performance with the different solvents. Solvent viscosities used are showed in Table 2.

Most of the studies concerning membranes in non-aqueous systems point out the influence of viscosity on the mass transport through the active layer and in membrane permeability and selectivity. According to Sarmiento et al. [12], a fluid can be considered as a resistance to mass transport that promotes load losses between the interfaces of the separation process. For flows in which Reynolds number (Re) is very low, the flow rate is directly

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