



Investigation of UF membranes fouling and potentials as pre-treatment step in desalination and surface water applications

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ABSTRACT

The surface fouling of UF membranes used upstream as pre-treatment stage is critical for the long-term stability of the subsequent treatment stage (NF/RO membranes). In this paper, an attempt was made to probe and compare the potential of versatile UF membranes structures in terms of flux decline and selectivity, for more convenient pretreatment membranes selection. The role of polyethersulfone (PES) host polymer concentration, on the morphology and surface characteristics of asymmetric flat sheet ultrafiltration (UF) membranes, has been comprehensively investigated. Distinctly, as the casting solution viscosity decrease, a higher pore size, pore size distribution and pure water flux was observed along with lower mechanical properties and wider cross-section morphologies. However, this impact was trivial on water contact angle, surface roughness parameters and charge negativity of the membrane. To further assess the potential performance of the hand-made fabricated membranes, they were systematically evaluated against three organic model foulants with dissimilar origins; humic acid (HA) – as natural organic matters (NOM), sodium alginate (NaAlg) – as polysaccharide, and bovine serum albumin (BSA) – as protein, under different initial feed concentration and pH chemistry. A disparate fouling behavior was observed depending on the membrane characteristics and the organic model foulant used. Depending on the UF membrane cut-off used, lower MWCO membranes, PES22 (6 kDa) and PES20 (10 kDa) exhibited a negligible relative flux decline while extremely low relative flux patterns were observed in the filtration with the 100 kDa membrane (PES16), as a result of one or more pore blocking mechanisms observed.

1. Introduction

UF membranes have been given a special consideration as pre-treatment membranes for desalination plants via providing high quality feed water to ensure stable and reliable operation of the desalination units. In addition to remarkable potentials against much more difficult liquid environments such as industrial and municipal wastewaters. Regardless of raw water turbidity, UF membranes provide an absolute barrier to pathogens and particulates, and act as a safe guard for the RO membranes by physical separation of solids, unlike conventional pre-treatment methods [1]. Thus, UF can bestow significant opportunities for the future prospects of water treatment, taking into account its cost and energy efficiency when compared with conventional separation techniques. In addition, introducing the technology enables many industries to become eco-friendlier by facilitating the recycling of waste materials and resources recovery [2].

Substantial effort has been devoted in the last few decades to fabricate synthetic polymeric membranes with desired selectivity, permeability, structure and physiochemical properties. Numerous synthetic membranes structures have been produced via a variety of techniques and synthetic materials. One of the most notable techniques is the phase inversion (PI) or phase separation (PS) technique, induced by immersion precipitation, and most of the flat sheet-polymeric membranes are made using this technique [3]. Compared to conventional techniques, PI is extremely versatile and allows high flexibility in membrane material selection, and a wide range of pore sizes (1–10,000 nm) can be obtained, as long as the system (polymer-solvent) miscibility gap is over a defined temperature and concentration range [4]. However, during the fabrication of asymmetric polymeric membranes by PI, many major and secondary variables can also be adjusted to control the overall membrane properties. The fabricated membrane morphology, mechanical strength, permeability, and selectivity are influenced by the

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interplay of all synthesis parameters. One of the key factors affecting membrane characteristics and performance is the host polymer concentration used to prepare the membranes.

It is well established in the literature that membrane's structure, obtained at the end of the PI process, relies on the physio-chemical characteristics of dope casting solution. As the concentration of a dope solution changes, surface characteristics of that synthesized polymeric membrane will vary with the formation of a range of diverse complex structures, depending on the process conditions and solution composition. Indeed, this would reflect on the permeation characteristics, selectivity and antifouling behavior of the membrane. Particularly, a higher polymer concentration will induce the formation of a higher viscosity dope solution at the bimodal-phase separation point and form a denser structure.

Currently, membrane process sustainability is still doubted in some industrial applications and fouling of membranes represents an inherent and significant issue due to its complexity and variety [5]. According to Hilal et al., membrane fouling phenomena can take place at different locations in membranes, including at both selective layer and pore walls. Internal fouling refers to the deposition and adsorption of small particles or molecules onto the pore entrances or the inside of a pore of the membrane whereas external fouling refers to the accumulation of rejected molecules upon the membrane surface [6]. These fouling mechanisms could occur at different degrees depending on the characteristics of the membrane and targeted organic molecules. For instance, the fouling behavior of porous microfiltration (MF) and ultrafiltration (UF) membranes are significantly different to those observed for seawater nanofiltration (NF) and reverse osmosis (RO) membranes. Pore blocking is the common fouling mechanism in the MF and UF membranes while it is not considered for the NF and RO membranes. Moreover, pore blocking is suggested to be a larger contributor than cake formation in UF membrane fouling and is influenced by the surface density of membrane pores and the pore size distribution [7, 8]. Thus, understanding the contributing fouling process mechanisms is a fundamental approach to provide better means for minimizing membrane fouling and its sub-consequences, enabling the adoption of an adequate membrane separation process and operating conditions for targeted industrial applications.

Organic compounds such as humic substances, proteins, and polysaccharides in water are the most abundant foulants in UF of many industrial operations. These foulants are inherently severe for pressure-driven membranes due to their strong irreversible adsorption on the surface of the membrane, causing a dramatic decline in membrane permeation, and raise many issues regarding product quality and process cost [9]. The degree of selectivity and fouling does not only depend on membrane properties but also on the complexity of the foulants' composition, their molecular weight and the charge of organic compounds, alongside the hydrodynamic conditions and solution chemistry [10]. Solution chemistry is critical for controlling the charge and configuration of organic foulants that will influence the intramolecular and molecules-membrane interactions, and hence membrane performance [11]. More precisely, performance may differ for different solute types (charged or neutral) and shapes (globular, linear or branched solute), that have similar molecular weight [12]. This may be the case even under the same operating conditions.

In the present research, an investigation was made into the influence of PES concentration on UF membrane morphology and surface characteristics. PES/UF membranes have been synthesized with a wide range of structures that correspond to typical commercially available UF membranes. Fouling behavior and retention efficiency tests were conducted to give a more precise and comprehensive assessment of UF membrane fouling. Three different organic model foulants (HA, BSA, and NaAlg) were applied for this comprehensive fouling assessment under a broad range of feed concentration and solution pH.

Table 1
Composition of PES membranes.

Membrane ID	PES wt%	NMP wt%	PVP K30 wt%
PES22	22	76	2
PES20	20	78	2
PES18	18	80	2
PES16	16	82	2

2. Experimental

2.1. Materials and reagents

Polyethersulfone (PES) flakes (M.wt 75,000) were kindly donated by BASF Co. Ltd. (Germany). Polyvinylpyrrolidone (PVP 40 k), *N*-methyl-2-pyrrolidone (NMP) with $\geq 99\%$ purity, polyethylene glycol PEG (600 Da–35 kDa), polyethylene oxide PEO (100 kDa), humic acid (HA, ~ 2 –500 kDa) as natural organic matter (NOM), Sodium Alginate (NaAlg, ~ 12 –80 kDa) from Brown algae as polysaccharide, and bovine serum albumin (BSA, ~ 66 kDa) as protein were all purchased from Sigma Aldrich, UK. While 200 nm carboxylated polystyrene latex as tracer particles were supplied by Polysciences Inc., PA, USA.

2.2. Membranes fabrication

Since a very low and high casting solution viscosity results in a brittle and tight membrane structure, respectively. Four asymmetric PES flat sheet membranes, with a different PES polymer weight percent (wt%), were fabricated. Each membrane was denoted according to the membrane composition, as tabulated in Table 1. The compositions used here correspond to the range of PES concentration used in literature for producing loose to tight UF membranes.

All membranes were fabricated via the classical non-induced phase separation (NIPS) technique as described elsewhere [13]. In brief, for membranes fabrication, a fixed amount (2 wt%) of PVP K30 was dissolved in NMP using a double neck-round bottom flask. The PES flakes were then gradually added to the solution and mechanically stirred overnight at 50 °C until a homogenous, yellowish and clear solution was achieved. The casting solution was then degassed under vacuum for an hour to get rid of air bubbles. For solution casting, about 10 ml of casting solution was poured onto a glass substrate and cast at a regular shear rate (225 s^{-1}) via an automated casting knife (RK film applicator). The resultant thin film was directly placed in a DI water bath at 20 °C for precipitation. Within less than 2 min, the membrane was detached from the glass substrate indicating that the phase inversion was completed. The membranes were then repeatedly rinsed with DI water to remove residual solvent and stored wet at 4 °C until used. All membranes were cast with 200 μm clearance gap at ambient temperature and relative humidity (RH% 45 ± 5). Prior to testing, the membranes were inspected under light to make sure there was no pinholes, wrinkles or any defect that could produce a performance discrepancy.

2.3. Characterization

Pure water flux (PWF) and organic filtration experiments were conducted under a crossflow condition with an active membrane area of 12.6 cm^2 and controlled temperature of 20 ± 0.5 °C. After 30 min compaction time at 0.5 MPa, the pressure was reduced to 0.4 MPa and the DI water permeate flux recorded automatically every 1 min, using data collection software interfaced with an electronic balance. The automated software converted the permeate weight data received from the balance into a flux and recorded the values on an excel spreadsheet for previously set membrane area and time intervals. For the evaluation of UF membrane performance, the pH and concentration of the organic molecule in feed solutions were varied. After the compaction with DI

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