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High performance flat sheet forward osmosis membrane with an NF-like selective layer on a woven fabric embedded substrate

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ABSTRACT

High performance flat sheet forward osmosis (FO) membranes have been successfully fabricated using Torlon® polyamide-imide (PAI) materials via phase inversion followed by polyelectrolyte polyethyleneimine (PEI) post-treatment to form a nanofiltration (NF)-like rejection layer with positive charges. The structure and charge property of the FO membranes were carefully tailored by optimizing casting conditions and selecting different immersing methods for the post-treatment. Especially, when the PAI micro-porous substrate was embedded with a woven fabric, the enhanced mechanical strength of the membrane made it possible to reduce the thickness of the substrate to 55 µm, which is in favor of high water flux. The resultant membranes can reach a water flux of 29.65 L m⁻² h⁻¹ with a ratio of salt flux to water flux (J_s/J_v) of <0.8 g L⁻¹ in the active layer facing draw solution (AL-facing-DS) configuration, or the water flux of 19.2 L m⁻² h⁻¹ and the J_s/J_v of <0.5 g L⁻¹ in the active layer facing feed water (AL-facing-FW) configuration using 0.5 M MgCl₂ as the draw solution and de-ionized water as the feed water at room temperature.

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1. Introduction

There has been an enormous increase in demand for water and energy worldwide. As a potential alternative solution for seawater desalination, wastewater treatment and power generation, etc., osmotic membrane processes such as forward osmosis (FO) and pressure retarded osmosis (PRO) have received considerable attention in recent years [1,2]. Stimulated by the increased interests in FO/PRO processes, novel semi-permeable membranes, a core element in FO/PRO system, are experiencing rapid development [3–12].

Conventional reverse osmosis (RO) or nanofiltration (NF) membranes are semi-permeable membranes. In principle, they can be used for FO process. However, the water flux of RO/NF membrane was extremely low when used in the FO process due to severe internal concentration polarization (ICP) occurred inside the thick support substrate of these membranes [6,13]. The unique feature of highperformance FO membrane lies in that its substrate should be very thin and/or highly porous, which is able to minimize ICP and thus maintain a high osmotic pressure difference across the membrane rejection layer to achieve high water flux [3,7].

The first commercial FO membranes were developed by Hydration Technology Inc. (HTI) [14]. These membranes, made from cellulose triacetate (CTA) embedded with a very thin polyester mesh instead of thick nonwoven fabric support in the traditional membrane, resulted in better FO performance than typical RO/NF membranes. This work provides inspiration for later studies. Nevertheless, their water permeability and salt rejection are not sufficiently high. In addition, they can only be used in a narrow pH range [15]. Extensive efforts have been made to develop high performance FO membranes with high water flux and low salt passage, and considerable achievements have been attained. For example, Wang et al. [3,4] have fabricated high performance thin film composite (TFC) FO hollow fibers with a RO-like selective layer on the inner surface of highly porous polyethersulfone hollow fiber substrates. Similarly, Yip et al. [7] reported a high-performance flat sheet thin film composite FO membrane supported with a thin polyester nonwoven fabric.

In parallel to the advancement of FO membrane with a RO-like selective layer, FO membranes with a NF-like skin layer are also under development, as this type of FO membranes can be used for the feed containing multivalent ions or large bio-molecules using divalent ions such as MgCl₂ as the draw solutes. Wang et al. [11,12] reported the FO application of polybenzimidazole (PBI) NF hollow fiber membranes. Su et al. [9] fabricated cellulose acetate NF hollow fiber membrane and applied it to FO process. The former membranes showed a water flux of 4 and 6 L m⁻² h⁻¹ for the configurations of active layer facing feed water (AL-facing-FW) and active layer facing draw solution (ALfacing-DS), respectively, while the latter had a water flux in the range of 2.7–7.3 L m⁻² h⁻¹ using 0.5–2.0 M MgCl₂ as the draw solutions. The water fluxes of these membranes were considered to be low, as both were made using high-concentration dope solutions via phase inversion method. Recently, Setiawan et al. [5] developed a new type of NF-like FO hollow fiber membranes by utilizing asymmetric

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microporous hollow fibers made of Torlon® polyamide-imide (PAI) material as the porous substrate followed by a polyelectrolyte (polyethyleneimine (PEI)) post-treatment. The water flux of this FO membrane can reach 9.74 L m⁻² h⁻¹ when using 0.5 M MgCl₂ as the draw solution and DI water as the feed water. This approach opens a new channel for making high performance FO membrane with a NF-like selective layer.

The objective of this study is to demonstrate the feasibility of fabricating a positively charged NF-like FO flat sheet membrane by using PAI asymmetric micro-porous flat sheet membranes as the porous substrate without/with a thin (50 μ m) polyester woven fabric support, followed by the PEI post-treatment. The performances of final resultant membranes were tested in a FO setup to explore their potential for FO applications.

2. Experimental

2.1. Materials and chemicals

Torlon® 4000T-MV (copolymer of amide and imide) (PAI), supplied by Solvay Advanced Polymers (Alpharetta GA), was used to make micro-porous flat sheet substrates. A commercial polyethylene terephthalate (PET, grade 165–31) woven fabric with a thickness of 50 µm and a porosity of 23% was purchased from Anping Shengyuan (Hebei, China). N-Methyl-2-pyrrolidone (NMP, >99.5%, CAS#872-50-4, Merck Chemicals, Singapore) and lithium chloride (LiCl, anhydrous, CAS#7447-41-8, MP Biomed) were used as solvent and pore former, respectively. Polyethyleneimine (PEI, molecular weight (M_w) ~800, Sigma Aldrich) ethylenediamine end-capped (Sigma Aldrich) was used to perform chemical post-treatment of the flat sheet substrate. For filtration experiments, magnesium chloride (MgCl₂, hexahydrate) was purchased from Merck. All the reagents were used as received.

2.2. Fabrication of microporous PAI flat sheet substrates and post-treatments

Torlon® 4000T-MV was dried in a vacuum oven at 50 °C for at least 12 h to remove moisture prior to the preparation of dope solutions. Pre-weighed amounts of the polymers (PAI, 14 wt.%, PEG 600, 10 wt.%) was then dissolved into NMP in a jacket flask equipped

with a mechanical stirrer at 60 °C for 3 days. Once the polymer was dissolved completely, a desired amount of LiCl (2 wt.%) was added into the flask followed by mixing, filtering, and degassing prior to film casting.

PAI flat sheet substrates, designated as P1# and P2#, were made based on the same polymer material but different casting conditions. For the P1#, the PAI solution was directly poured onto the surface of a glass plate, then a cast knife (Elcometer (Asia) Pte. Ltd) at a gate height of 175 µm was used to spread the solution onto the glass plate. For the P2#, the PAI solution was spread onto the surface of a glass plate, using a cast knife at a gate height of 60 µm, then a tailored PET woven fabric was embedded into the PAI solution from the top. The whole composite was immediately immersed into tap water at room temperature to initiate phase separation. The resultant membranes were kept in flowing tap water, rinsed overnight and then stored in deionized (DI) water for further polyelectrolyte post-treatment or characterization. Chemical post-treatment was conducted by immersing the PAI membrane substrate completely into a 1.6 L of PEI aqueous solution at 70 °C. The reaction time was fixed at 120 min, and the PEI concentration used was 1.0 wt.% as described elsewhere [12]. The resultant PEI modified PAI membranes were designed as PAI 1#, PAI 2#, respectively. Another PAI membrane sample, which was prepared by only immersing the top surface of the P2# substrate to the PEI aqueous solution, was designed as PAI 2#-S.

2.3. Characterization of PAI flat sheet membranes

Membrane cross-sections and surface morphologies were observed by Zeiss EVO 50 SEM. All samples were coated with a thin layer of gold using EMITECH SC7620 sputter coater. A Perkin Elmer Spectrum 2000 ATR-FTIR spectroscope was used to confirm the chemical changes on the membrane surface before and after crosslinking reaction. The details procedure was described elsewhere [12]. Pure water permeability (PWP) and salt rejection experiments were conducted in a bench scale cross-flow RO filtration unit. Compaction was carried out at 1 bar for 3 h prior to the measurement. The salt rejection test was carried out using a 500 ppm MgCl₂ salt solution based on conductivity measurements (Mettler Toledo) of permeate and feed water.



Fig. 1. (a) microscopic image of blank PET woven fabric, and SEM images of top surfaces of (b) PAI membrane without woven fabric (P1#) and (c) PAI membrane embedded with woven fabric (P2#); SEM images of the cross-section of the membranes: (d) without woven fabric (P1#), (e) with woven fabric (P2#), and (f) a magnified view of the dense, sponge-like morphology close to the top surface of P2#.

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