



Novel design of hydrophobic/hydrophilic interpenetrating network composite nanofibers for the support layer of forward osmosis membrane

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HIGHLIGHTS

- A novel high flux FO membrane was designed and fabricated.
- The HH-IPN-CNF used for FO membrane support layer was produced by electrospinning.
- The HH-IPN-CNF structure effectively reduced the ICP.
- The water-transferring function of the HH-IPN-CNF structure is critical for flux improvement.
- The highest water flux of 47.2 LMH is among the available published literatures so far.

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ABSTRACT

Forward osmosis (FO) is an emerging technology, however the internal concentration polarization (ICP) problem needs to be resolved in order to improve the water flux. Hydrophobic/hydrophilic interpenetrating network composite nanofibers (HH-IPN-CNF) was used for FO membrane support layer, which was successfully designed and fabricated by electrostatic spinning technology. Hydrophobic polymer is polyethylene terephthalate (PET) and hydrophilic polymer is polyvinyl alcohol (PVA).

The FO membrane flux has a significant increase due to the HH-IPN-CNF support layer. The membrane flux improved with the increase of PVA nanofiber content in the HH-IPN-CNF support layer. When the ratio of PET/PVA composite nanofibers was 1/4, the FO membrane exhibited the highest water flux (47.2 LMH) and low salt leakage (9.5 gMH). In the test, deionized (DI) water was used as feed solution (FS), and 0.5 M NaCl solution was used as draw solution (DS) in the mode of active layer facing DS. The improvement of FO flux was attributed to the increase of wetting performance of support layer and the water-transferring function. The existence of the HH-IPN-CNF structure formed between PET and PVA nanofibers leads to the reduction of the ICP. The FO membranes with HH-IPN-CNF support layer achieved a significant increase in water flux.

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1. Introduction

Forward osmosis (FO) has received worldwide attention in recent years because it is an energy-effective, high rejections to contaminants, low pollution technology. FO technology has great potential applications in seawater/brackish desalination [1–3], wastewater treatment [4–6], food processing [7–9], power generation [10–12], and protein and pharmaceutical enrichment [3,13,14]. FO can produce pure water, and it is created by the osmotic pressure difference across a semi-permeable membrane from the feed solution (FS) side to the draw solution (DS) side. Previously, a great many studies have focused on the

Abbreviations: FO, forward osmosis; ICP, internal concentration polarization; HH-IPN-CNF, hydrophobic/hydrophilic interpenetrating network composite nanofibers; TFC, thin film composite; DI, deionized; FS, feed solution; DS, draw solution; TPSP, thin polyamide selective layer.

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fabrication of FO membranes. These efforts have resulted in the development of substantially improved membranes [15], which are tailored for the specific needs of FO. However, the number of successfully developed FO membranes for commercial use is limited [16,17], which restricts the full implementation of FO technology. To the present, commercial flat sheet asymmetric FO membranes made of cellulose triacetate (CTA) from Hydration Technologies Inc. are dominant, which are fabricated by non-solvent induced phase inversion [18]. Widespread developments of FO membranes have been in the form of asymmetric flat sheet or hollow fiber membranes; where both are produced by phase inversion technique [19,20]. These membranes have been widely used in various fields [21]; however, they have relatively low water flux particularly for osmotic seawater desalination, and they can be easily degraded by DS such as ammonium bicarbonate [22]. Thin film composite (TFC) membranes made by interfacial polymerization on porous support layers have advantages such as a higher water permeability, greater solute rejection, and non-biodegradability [16] in comparison to the conventional asymmetric membranes (CTA-based membranes). The traditional support layers of FO membranes are the phase separation-formed microporous polysulphone and polyethersulphone, which has either a dense finger or a sponge-shaped structure. The phase separation-formed support layer significantly increases resistance experienced by water and this results in a severe internal concentration polarization (ICP) problem. The ICP is a phenomenon inherent of the osmosis driven membrane system, as the result of the hindered diffusion of solutes within the membrane support layer. Generally, ICP acts to diminish the overall driving force across a membrane [23], and it is mainly responsible for water flux decline [24,25]. Most reported FO membranes encounter the ICP problem, and this occurs in phase inversion support layers. An ideal support layer for FO membrane should exhibit minimal thickness, minimal tortuosity, and a high porosity [26]. A novel nanocomposite FO membrane with a scaffold-like nanofiber support layer has been developed to mitigate ICP [27]. The electrospun ultrafine fibers for support layer have advantages as follows: (a) high porosity, (b) low tortuosity, and (c) thin thickness; hence, it is superior to support layer, which is formed by conventional phase inversion. The electrospun ultrafine fibers could provide a good solution for eliminating the ICP bottleneck [27].

Xiaoxiao Song et al. [27] polymerized active layer on top of hydrophobic polyethersulfone (PES) nanofiber support layer. This nanocomposite FO membrane exhibited water flux up close to 35LMH in the mode of the membrane active layer facing the DS using deionized (DI) water as FS and 0.5 M NaCl as DS. It is much higher than that of FO membrane with phase inversion support layer. To the best of our knowledge, it is the “best experiment result” among some available published literatures [18,20,21,24].

It has also been reported that ICP may be enhanced by the hydrophobic nature of TFC support layers [21]. The reduction in “wetted porosity” of the hydrophobic support layers reduces solute diffusivity and available pathways for water transport [28]. Kim [29] reported that the modification of the polysulfone support layer resulted in an increase of the water flux, salt rejection, and chlorine resistance. A novel HH-IPN-CNF support layer was designed in our research in order to enhance the hydrophilicity and the permeability of the support layer. The hydrophobic polyethylene terephthalate (PET) and hydrophilic polyvinyl alcohol (PVA) composite nanofibers were fabricated by electrospinning. The novel flat-sheet FO membrane was composed of HH-IPN-CNF support layer and a thin polyamide selective layer (TPSL). The TPSL was polymerized in situ via an interfacial polycondensation reaction. *m*-Phenylenediamine (MPD) and 1,3,5-trimesoylchloride (TMC) were employed as the monomers for the interfacial polymerization reaction to form a TPSL on top of HH-IPN-CNF support layer for the fabrication of FO membrane. The support layer and selective layer can be independently tailored.

In our paper, the objectives are (1) to design and fabricate HH-IPN-CNF support layer used for FO membrane and (2) to investigate the effects of HH-IPN-CNF support layer on the FO membrane performance.

It is postulated that the enhancement of water flux of the resultant FO membranes can be improved by forming HH-IPN-CNF support layer structure.

2. Materials and methods

2.1. Materials

Polyethylene terephthalate pellets (PET, viscosity of 0.8 ± 0.02) were supplied by Far Eastern Industry Ltd (Shanghai, China). Polyvinyl alcohol powder (PVA, $M_w = 77000$ g/mol, 98% hydrolyzed, degree of polymerization of 1750 ± 50) was purchased from Yili Chemicals Co. Ltd (Beijing, China). 1,3,5-Trimesoylchloride (TMC, 98%, urchem) and sodium chloride (NaCl, crystalline, $\geq 99.5\%$) were provided by National Medicine Group Chemical Reagent Co. Ltd (Shanghai, China). Glutaraldehyde (GA, 50% aqueous solution), trifluoroacetic acid (TFA, $\geq 99.0\%$), *n*-Hexane, and sodium hypochlorite (NaOCl, solution, and available chlorine 10%–15%) were obtained from Chengdu Kelon Chemical Reagent Factory (Sichuan, China). *n*-Hexane is utilized as the solvent for TMC.

Hydrochloric acid (HCl, 36%–38%), acid acceptor triethylamine (TEA, $\geq 99.0\%$), and sodium bisulfate (NaHSO₃, analytical reagent) were purchased from East Sichuan Chemical Co. Ltd (Chongqing, China). *m*-Phenylenediamine (MPD, 99%) was obtained from Recovery of Fine Chemical Engineering Research Institute (Tianjin, China). Deionized (DI) water was obtained from an Ultra Purified Water System (Molecular ΣH₂O®, China).

2.2. HH-IPN-CNF FO membrane fabrication

2.2.1. HH-IPN-CNF support layer fabrication

The preparation of HH-IPN-CNF support layer is given as follows:

- PET was dissolved in TFA, then the mixtures were stirred at ambient temperature for 12 h to obtain a 13.5 wt% homogeneous solution.
- PVA was dissolved in DI water. The solution was stirred at 90 °C for 3 h to obtain a 7 wt% homogeneous solution. Different ratios of these two polymer solutions were prepared for HH-IPN-CNF support layer fabricated by electrospinning, as displayed in Table 1.
- These two polymer solutions were added into different syringes and induced to flow out freely at the rate of 13.5 μl/min through a needle with an inner diameter of 0.7 mm. The composite fibers were deposited on a grounded rotating metallic (0.5 m²) under a 20 kV DC electric voltage. The deposition time was fixed in order to get a consistent HH-IPN-CNF thickness.
- The HH-IPN-CNF support layer was immersed in DI water for 10 min and then dipped into GA water/acetone solution for 2 h for cross-linking of PVA. The GA cross-linking solution was prepared by mixing water and acetone at ratios with water concentration in the range of 10 wt%. A given amount of GA was added into the water/acetone mixture and HCl solution was added to adjust the pH of the solution to 1.5.
- The resulting HH-IPN-CNF support layer was washed with DI water, then it was dried in a vacuum oven at 30 °C for 5 h and finally the samples were stored in a desiccator before use.

2.2.2. Polyamide selective layer fabrication

A salt-rejecting polyamide active layer was fabricated on top of HH-IPN-CNF support layer by the interfacial polymerization reaction between MPD and TMC, which was adopted from the previous work with some modifications [30]. The procedure was as follows: first, the HH-IPN-CNF support layer was immersed in an aqueous solution of 2–3.4 wt% MPD solution for 2 min. Thereafter, an absorbent paper was used to remove the water droplets on the support layer surface. After 2 min in the air, the saturated support membrane was dipped into a solution of 0.1 wt% TMC in *n*-hexane for 1 min; thereafter, TPSL was

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