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Middle support layer formation and structure in relation to performance of three-tier thin film composite forward osmosis membrane

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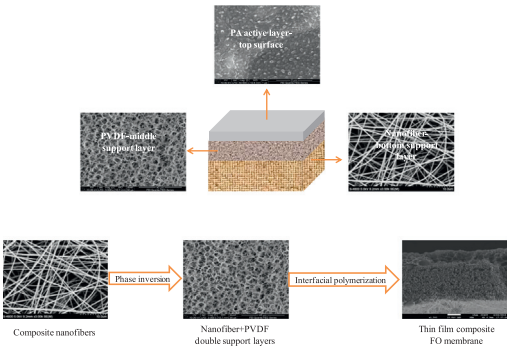
HIGHLIGHTS

- A novel three-tier TFC FO membrane was successfully designed and fabricated.
- Double support layers were produced by integrating electrospinning and phase inversion technique.
- The double support layers structure effectively reduced the ICP and enhanced the mechanical stability of TFC FO membrane.
- The casting parameters that affect the middle support layer formation and structure were investigated, respectively.
- A water flux of 30.62 LMH can be achieved with the PVDF middle support layer cast at 7 wt% concentration.

GRAPHICAL ABSTRACT



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ABSTRACT

In recent years, nanofibers have been directly used as the support layer for developing high-flux forward osmosis (FO) membrane due to their high porosity and low tortuosity. However, the rougher surface and larger pore size weaken the adhesion to the polyamide (PA) active layer. Conventional FO membrane made of phase inversion support layer exhibits favorable mechanical stability, however the water flux is poor due to severe internal concentration polarization (ICP). In order to simultaneously reduce the ICP and enhance the mechanical stability of FO membrane, a thin film composite (TFC) FO membrane composed of three layers was prepared, namely bottom electrospun hydrophobic/hydrophilic interpenetrating network composite nanofibers (HH-IPN-CNF) support layer, a phase separation formed microporous polyvinylidene fluoride (PVDF) middle support layer, and the top PA active layer formed by interfacial polymerization. The factors including polymer concentration, coagulation bath composition and the gate height of the casting blade that affect the mid-layer structure formation were investigated, respectively. With the mid-layer of FO membrane cast under different conditions, water flux ranged from 10.39 to 30.62 LMH, by using 0.5 M NaCl draw solution and deionized water feed solution. The TFC FO membrane with high structure integrity may be promising candidate for engineered osmosis.

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1. Introduction

Forward osmosis (FO) process is driven by the osmotic pressure difference between draw solution (DS) and feed solution (FS) across a permselective membrane. FO causes much less problem of fouling and cleaning [1], which is an emerging membrane process. The first key component of the FO unit is the semipermeable membrane, which only allows water to permeate through while blocking all the solutes in the DSs and FSs. An ideal FO membrane should present high water flux and low reverse solute flux in addition to good chemical and mechanical stability [2]. Commercial asymmetric cellulose triacetate (CTA) membranes from Hydration Technologies Inc. (HTI) are chemically stable only in a small pH window between 4 and 6, and especially, the water flux is relatively low [3]. To improve the water flux, most FO membranes are designed of asymmetric structure with a thin dense active layer on a porous support layer, i.e., thin film composite (TFC) FO membranes. In general, TFC polyamide (PA) membrane presents greater water flux than phase inversion membrane, CTA membrane for example. Furthermore, the support layer and active layer can be independently tailored. Therefore, it is relatively easy to optimize the overall TFC membrane with desirable separation performance.

Internal concentration polarization (ICP) is one of the serious problems in any FO process, which occurs inside the porous support layer. In the FO process, the severe reduction of water flux results from ICP, which is mainly influenced by the porosity, tortuosity and thickness of the support layer [4,5,24]. An ideal support layer for FO membrane should have low tortuosity, be highly porous, and have a very thin structure [4,5,7,24]. Therefore, the fabrication of an ideal FO membrane relies on mitigating the ICP problem in the support layer [8].

Some achievements in developing high performance FO membranes have been reported with the support layer produced via phase inversion methods [2,9,10,12,24]. However, these FO membranes all performed far below their potential due to severe ICP caused by their thick and dense phase inversion support layers [8]. Thus, in order to enhance FO membrane performance, it is essential to alleviate the ICP occurring in the support layer. According to the literatures [5–9], ICP can be largely reduced by forming a highly porous support layer.

Electrospun nanofibers exhibit an intrinsically high porosity and low tortuosity, which are promising candidate for TFC FO membrane support layer [11,13–20]. Recently, some studies have reported the fabrication of TFC FO membrane with an electrospun nanofibers support layer and a top active layer by interfacial polymerization [11,13–20]. The diffusive resistance was greatly reduced on account of the low tortuosity and open interconnected pore structure in the nanofibers support layer. Hence, the resulting FO membranes with lower ICP exhibited excellent permselectivity performance. Nevertheless, the nanofibers support surface possesses an open structure with large interconnected pores and high roughness [16], thus the adhesion is poor between the PA active layer and nanofibers support layer. Besides, the application of nanofibers as the support for TFC membrane is restricted due to their weak mechanical strength.

Several researches have been reported to overcome the aforementioned obstacles. Hot pressing was an effective method to reduce the pore size of the nanofibers support layer and surface roughness [17]. Results indicated that the mechanical integrity, the adhesion between the PA active layer and electrospun nanofibrous layer and backing material improved with the increase of pressure. Park et al. [18] presented that the open pore size of nanofibers could be reduced based on a post co-solvent treatment technique and the power density of the resultant TFC membrane increased significantly. Inorganic material was also investigated to modify the nanofibers performance. Obaid and the co-workers [19] incorporated amorphous silica nanoparticles into electrospun polyvinylidene fluoride (PVDF) nanofibers mats to change the porous structure and enhance mechanical property of the nanofibers. However, the active layer is still possible to delaminate from the nanofibers support layer at high cross-flow velocities and significant mechanical stress in pressure-retarded osmosis (PRO).

Nonsolvent induced phase separation technology combined with electrostatic spinning technology has been demonstrated to be an efficient method for the fabrication of FO membrane support layer tailored for PRO application [20]. Laura A. Hoover et al. [20] presented the first TFC membrane prepared with electrospun nanofibers and phase separation support layers. What's more, the resultant membrane exhibited enhanced mechanical robustness and integrity for engineered osmosis. Nonetheless, merely hydrophobic polyethylene terephthalate (PET) was used as electrospun material. In addition, identical microporous polysulfone layer structure was produced under the fixed phase separation condition. The effect of phase inversion process parameters on the resultant FO membrane properties is still unclear.

In our study, a flat-sheet TFC FO membrane with double support layers was designed and fabricated following a three-step procedure: electrospun hydrophobic/hydrophilic interpenetrating network composite nanofibers (HH-IPN-CNF) bottom support layer, casting of a PVDF middle support layer under different conditions onto the HH-IPN-CNF by phase inversion and finally polymerizing a PA active layer on top of the mid-layer through interfacial polymerization. Hydrophobic PET and hydrophilic polyvinyl alcohol (PVA) were used as electrospun materials to form HH-IPN-CNF, which has the characteristics of wetting performance and the water-transferring function. And this structure has been proved the superior support layer for high-flux FO membrane in our previous work [21]. PVDF was employed as the polymeric material for the fabrication of the mid-layer in view of its good mechanical property and strong chemical resistance [16].

The objectives of this study are (1) to design and fabricate double support layers by combining the electrostatic spinning technology with phase inversion technology; (2) to investigate the casting parameters for the PVDF middle support layer formation, such as polymer concentration, coagulation bath composition and the gate height of the casting blade; (3) to evaluate and discuss the relationship between middle support layer structural characteristics and the corresponding resultant FO membrane transport performances.

2. Materials and methods

2.1. Materials

Polyethylene terephthalate pellets (PET, viscosity of 0.8 ± 0.02) was supplied by Far Eastern Industry Co., Ltd. (Shanghai, China). Polyvinyl alcohol powder (PVA, $M_w = 77.000$ g/mol, 98% hydrolyzed, degree of polymerization of 1750 ± 50) was purchased from Yili Chemicals Co. Ltd. (Beijing, China). Commercial polymer polyvinylidene fluoride (PVDF, KYNAR®,761) was obtained from Xia Men Yan State Import and Export Co. Ltd. 1,3,5-trimesoylchloride (TMC, 98%, urchem) and sodium chloride (NaCl, crystalline, ≥99.5%) were provided by National Medicine Group Chemical Reagent Co. Ltd. Glutaraldehyde (GA, 50% aqueous solution), Trifluoracetic acid (TFA, ≥99.0%), Dimethyl acetamide (DMAc), isopropyl alcohol (IPA, >99.5%) and n-Hexane were obtained from Chengdu Kelon Chemical Reagent Factory. n-Hexane was utilized as the solvent for TMC and DMAc used as the solvent for PVDF.

Hydrochloric acid (HCl, 36–38%) and acid acceptor triethylamine (TEA, ≥99.0%) were purchased from Chongqing in East Sichuan Chemical Co. Ltd. M-phenylenediamine (MPD, 99%) was obtained from Recovery of Fine Chemical Engineering Research Institute in Tianjin. Deionized (DI) water was obtained from an Ultra Purified Water System (Molecular $\sum H_2O$®).

2.2. Synthesis of flat-sheet TFC FO membranes

2.2.1. Preparation of FO support layers

2.2.1.1. Electrospinning HH-IPN-CNF bottom support layer. Equal proportion of PET and PVA nanofibers were prepared for bottom support

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