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Double-blade casting technique for optimizing substrate membrane in thin-film composite forward osmosis membrane fabrication

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ABSTRACT

The thin-film composite (TFC) methodology has been explored in the last couple of years to fabricate forward osmosis (FO) membranes. Different tactics have been proposed to mitigate the internal concentration polarization (ICP) phenomenon in the substrate membrane layer. However, such modifications on the substrate membranes would likely alter their top surface morphology and this in turn, would profoundly influence the subsequent process of interfacial polymerization (IP) to form the active layer. In the current work, we presented a facile substrate membrane fabrication strategy – double-blade casting technique, to produce substrate membranes with enhanced structural features to mitigate ICP, yet retaining an ideal top surface for the formation of an intact and highly salt-rejecting active layer. A series of standard protocols have been utilized to characterize the substrate membranes and resultant TFC-FO membranes. Overall the resultant TFC-FO membranes based on the double-blade casted polysulfone substrate membranes showed improvement in water flux, J_v with reduced apparent structural parameter, S values and retained a relatively low reverse salt flux/water flux, J_s/J_v ratio. With a 1 M NaCl draw solution and DI water feed, the best TFC-FO membranes achieved a J_v of 31.1 LMH and a J_s of 8.5 gMH in the FO orientation, and a J_v of 60.3 LMH and a J_s of 17.6 gMH in the PRO orientation. This concept demonstration study may open up many new platforms for flat-sheet substrate membrane fabrication for FO membranes.

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1. Introduction

Forward osmosis (FO) is a remarkable concept that offers a viable prospect as the next generation water purification and seawater desalination technology [1,2]. FO operates based on the osmotic pressure differential across a semi-permeable membrane, rather than hydraulic pressure differential such as in the reverse osmosis (RO) and nano-filtration (NF) process, to extract clean water from the feed into the draw solution [3]. Compared to the traditional pressure driven processes, FO process displays several merits worthy of being further explored and capitalized on: (1) lower energy consumption and equipment costs and higher water recovery [1–3]; (2) less propensity towards fouling [4,5]; and (3) wide-ranging applications, such as power generation [6–9], juice or food concentration [10], and essential products and pharmaceuticals enrichment [11,12].

Nevertheless, FO process does have its inherent drawbacks as its performance is highly sensitive to osmotic pressure of the draw solution and concentration polarization (CP) [13–17]. Concentration

polarization at the membrane surface (external concentration polarization or ECP) or within the substrate membrane layer (internal concentration polarization or ICP) has been considered as the main reason for the lower-than-expected osmotic efficiency and thus, the poor water flux observed in most reported osmotic experiments. Especially ICP, resulted from the dilution of the draw solution (dilutive ICP) or the concentration of the feed (concentrative ICP) within the porous substrate membrane layer, has been proven to be the most aggravating factor in undermining the osmotic driving force across the membrane [5,18], since it cannot be effectively mitigated with changes in process hydrodynamics [13,14,19]. As a result, FO still remains in a status of laboratory study due to these critical technical challenges.

In order for FO to be applied in full-scale application, more concerted effort must be done in sourcing for: (1) suitable draw solutions that exert high osmotic pressure, can be recycled effectively with low energy consumption and display minimal reverse solute fluxes; and (2) high performance membranes with properties deliberated specially for osmotic processes, such as desirable characteristics in reducing the effect of ICP [1–3,14].

Numerous developments of membranes catered to FO processes have been carried out in the forms of asymmetric flat sheet or hollow fiber membranes, mostly involving phase inversion

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technique [3]. The current benchmark flat-sheet asymmetric FO membranes available in the market are made of cellulose triacetate (CTA). Although these membranes have been tested for broad applications, they have relatively low water permeability and salt rejection, particularly for application in seawater desalination [20]. The next generation of FO membranes was motivated by the thin-film composite (TFC) approach originally industrialized for RO membranes production and can be found in the form of both flat-sheets and hollow fibers. These membranes are typically fabricated in a two-step process: (1) a porous substrate membrane is formed by phase inversion technique with special features to enhance solute mass transfer and thus to reduce ICP and a second step where (2) a thin polyamide (PA) active layer is prepared by interfacial polymerization (IP) of amine and acid chloride on the substrate membrane surface [18,20–25]. For flat-sheet TFC-FO membranes, they generally consist of three layers: (1) a thin PA active layer; (2) a porous polymeric sub-layer as the intermediate; and (3) a woven or non-woven fabric support backing [18,20,26–28]. These TFC membranes are preferred over integral asymmetric membranes formed directly from phase inversion because apparently they possess superior separation properties and allow independent optimization of the active layer and substrate membrane layer [23,28,29].

It has been generally acknowledged among the FO membrane researchers that high performance TFC-FO membranes should possess the following characteristics [18,20,22,26,30]: (1) an active layer with high permeate water flux and low solute permeability; (2) a substrate membrane layer which is thin, highly porous/permeable and hydrophilic to reduce ICP and boost water/solutes transport; and (3) an overall structure capable of withstanding the mechanical stresses generated by the operating conditions.

The substrate membrane layer is of particular importance because it not only provides mechanical strength and flow pathways, but also governs the extent of ICP and hence the effective osmotic gradient for water flux in the final TFC-FO membranes [25]. In order to capitalize on the osmotic driving force more effectively, the improvement of substrate membrane plays a more significant role in TFC-FO membrane fabrication [18,26,27,31,32]. Although alternative fabricating methodologies have been proposed to create substrate membrane layer that reduces ICP [33,34], phase inversion techniques still remain as the conventional and most widely accepted and practiced techniques in the industry. Thus a bulk of current research for FO substrate membranes focuses on the optimization of membrane material property or the conditions during phase inversion process [2,18]. Although TFC-FO membranes allow independent optimization of the active layer and substrate membrane layer as mentioned earlier, the modifications on the substrate membrane would inevitably alter the substrate membrane top surface morphology and may inadvertently undermine the integrity of PA active layer developed during the subsequent IP process [22,23,31,34]. In other words, their individual optimizations cannot be totally de-coupled and a paradox exists where any amendment aiming at improving the substrate membrane structure may potentially compromise the PA active layer integrity. Consequently, it is imperative to find new methods to assure that structural changes to the substrate membrane, which are essential in minimizing ICP, must not compromise its critical role as a suitable surface for the formation of an intact active layer during IP process [33].

Co-casting technique is an effective one-step membrane casting technique which can realize a multilayered configuration in the membrane [35–37]. Pereira et al. has firstly demonstrated the application of double-blade in fabricating flat-sheet membranes with two different polymer solutions [37]. More recently, Hashemifard et al. [38] also presented a study demonstrating the co-casting technique with a dual-blade casting knife to produce

dual-layered flat-sheet membranes for gas separation. This simple fabrication strategy can be innovatively adapted in conventional flat-sheet substrate membrane fabrication (with non-woven mesh backing) and may provide an alternative method to produce a more permeable substrate membrane, yet retain its surface morphology suitable for PA active layer deposition. This could eventually bring about an enhancement of osmotic performance for the resultant TFC-FO membranes by maintaining the integrity of the PA active layer while reducing the ICP phenomenon in the substrate membrane. Earlier works have demonstrated that reducing the polymer concentration of casting solution may reduce the substrate membrane thickness and increase its porosity, ensuing an enhancement of water flux in resultant TFC-FO membranes during osmotic performance testing [26,39]. The current study will further investigate the effect of casting solution polymer concentration on substrate membrane surface morphology and performance of resultant TFC-FO membrane in terms of both water flux and reverse salt flux. More importantly, the current study shall demonstrate, with the polymer concentration in the casting solution as the varying parameter, the concept of double-blade casting technique as an innovative adaptation to conventional casting procedure. The protocol strives to produce flat-sheet substrate membrane especially tailored for synthesis of TFC-FO membranes. The outcome of current work aims to validate, with membrane characterization and performance evaluation, that the resultant TFC-FO membranes fabricated based on the double-blade casted substrate membranes are able to display reduced ICP with enhancement in osmotic water flux yet not retreating on reverse salt flux. To the best of our knowledge, it is the first time that the double-blade casting technique has been employed to fabricate flat-sheet substrate membranes for TFC-FO membranes.

2. Materials and methods

2.1. Chemicals and membrane materials

Unless otherwise specified, all chemicals were of analytical grade with purity over 99% and were used as received. Chemicals used for membrane substrate formation included Polysulfone (Psf) beads (M_n : 22,000 Da, Sigma-Aldrich Pte. Ltd, Singapore) and N,N-dimethylformamide (DMF, anhydrous, 99.8%, Fisher Scientific, Aik Moh Pte. Ltd, Singapore). Chemicals used for interfacial polymerization included M-phenylenediamine (MPD, > 99%, ACROS, Aik Moh Pte. Ltd, Singapore), Trimesoyl Chloride (TMC, 98%, Sigma-Aldrich Pte. Ltd, Singapore) and n-Hexane (Fisher Scientific, Aik Moh Pte. Ltd, Singapore). Polyethylene oxides with molecular weight ranging from 200,000 to 1,000,000 g mol⁻¹ (PEO, Sigma-Aldrich Pte. Ltd, and Singapore) were used as solutes in the single-solute rejection test. Ultrapure deionized (DI) water was supplied from a Milli-Q ultrapure water system (Millipore Singapore Pte Ltd) with a resistivity of 18.2 M Ω cm. Sodium chloride (NaCl, ACS, Aik Moh Pte. Ltd, Singapore) was used as the draw solutes in osmosis performance tests and salt permeability tests. A commercial polyester mesh (PET, 90 μ m (T) \times 100² μ m (S), see Fig. 2) was used as a backing layer for the Psf substrate membrane. Commercial FO membranes were cut from a Hydrowell module procured from Hydration Technologies Inc (HTI, US) and were used for comparison purpose.

2.2. Synthesis of flat-sheet TFC-FO membranes

The TFC-FO membranes were prepared via two steps: (1) a phase inversion step to form the substrate membrane; and (2) an interfacial polymerization step to form the polyamide active layer.

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