



Controlling membrane ionization with bifunctional alendronates to benefit desalination through forward osmosis



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ABSTRACT

Gradual ionizing forward osmosis (FO) membranes were synthesized through tailoring modifications with alendronate materials on polyamide thin film composite (TFC) membrane surfaces. The newly developed membranes have gradually ionized surfaces and are more hydrophilic after introducing amino, hydroxyl and phosphate groups derived from the alendronates. Significantly improved performance was achieved with the alendronate-modified membranes relative to the unmodified membrane. With DI water as the feed and 0.5 M MgCl_2 as the draw solution, the novel membranes yielded water fluxes 35–60% higher than the nascent one. When MgCl_2 (2.0 M) as the draw solution, alendronate tetrasodium (A-4Na) modified membrane had a water permeation rate of 17.3 LMH under FO mode (feed solution faced the membrane active layer) in seawater desalination, a 25% increase compared to that of the pristine membrane and surpasses many other recently-developed FO membranes, remarkably increasing water filtration efficiency.

1. Introduction

Clean water production via desalination offers a solution to the problems caused by freshwater scarcity [1–3]. Distillation and membrane technology are both commonly used for desalination [1,3–5]. However, these technologies, such as reverse osmosis (RO), consume intensive energy in desalination processes [4,5]. Problems of severe membrane fouling and low water filtration efficiency are also extensively present in RO processes [2,3,6]. Hence, alternative technologies with better energy efficiency and low fouling tendency are needed in desalination.

Forward osmosis (FO) is an osmotically-driven process [5–10]. Without applied pressure, FO exhibits characteristics of lower fouling tendency and higher water permeation relative to the RO process [6,8], and thus has been widely used for water treatment in recent years [8–10]. As a key element in an FO system, FO membrane is crucial to advance FO technology. Regardless of the noticeable advances in FO membrane [6–9], most of the existing FO membranes still have drawbacks of low water recovery efficiency, severe solute leakage and internal concentration polarization (ICP) [6–9]. To mitigate these problems, thin film composite polyamide (TFC-PA) membranes have developed rapidly currently due to their advantages of having relatively high performance with good stability at a broad pH range [6,8,11,12]. However, the PA-TFC layers of membranes still face problems caused

by mass transfer due to its insufficient hydrophilicity and “ridge-and-valley” structural feature, and hence frequently encountering a rapid decline in water filtration efficiency [6,8,9,12]. Chemical modification by introducing hydrophilic groups to the dense PA layer is a proven approach in improving FO performance [8]. Many FO membranes have been explored by this approach surfaces recently [13–16]. Various materials including porphyrin (Por) [13], N-aminoethyl piperazine propane sulfonate (AEPPS) [14], zeolite nanoparticles [15], and graphene oxide (GO) nanosheets [16] have been used to develop membranes with promoted FO performance. However, only a slight increase in water flux was achieved in most cases possibly due to increased mass transport resistance with an extra layer attached on the membrane surface after modification. Other issues including complicated modification processes or corrosive and explosive chemical involvement are also widely present [8,17]. These problems not only lower synthesis efficiency and increase cost, but also damage the membrane structure. Clearly, exploring an efficient approach to develop high-performance FO membranes is necessary.

Appropriate FO membranes should be synthesized easily, have an optimized surface with good water permeability and salt rejection [6,8,18]. Based on these principles, we aim to develop novel charged FO membranes with gradually increasing ionization degree controlled by a series of alendronates with different ionization fractions. The gradual ionizing property is essential to explore the factors influencing

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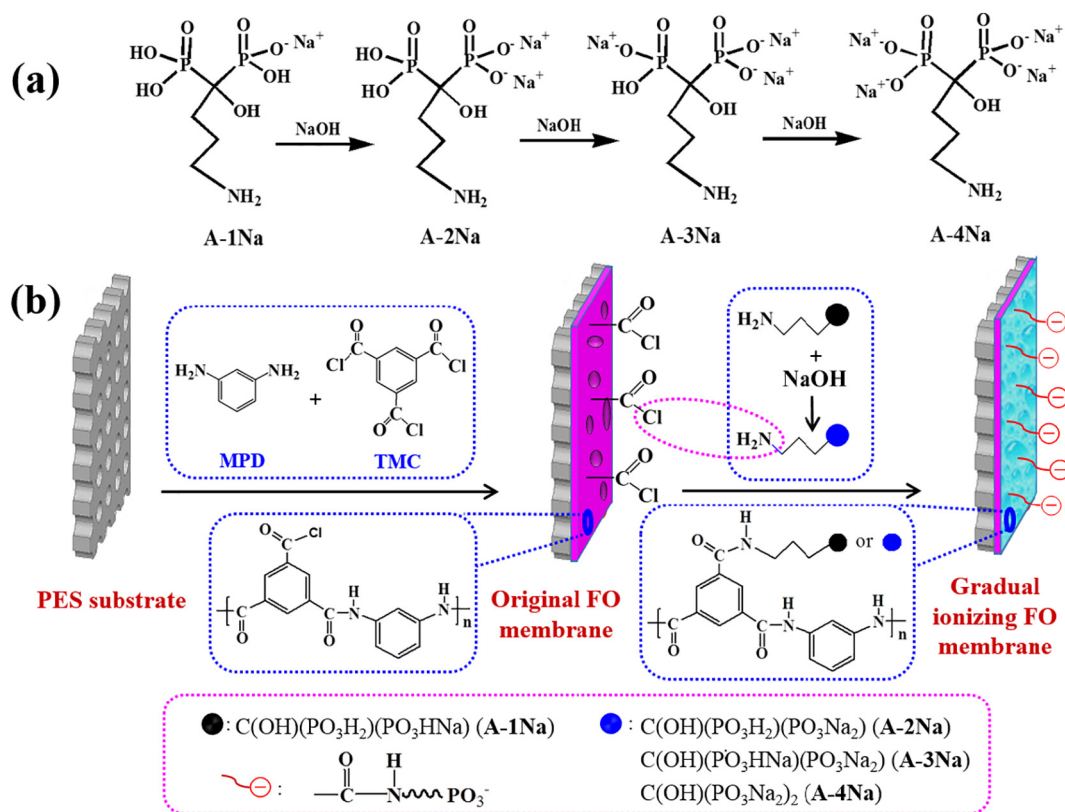


Fig. 1. The synthesis schemes of (a) alendronates of A-2Na, A-3Na and A-4Na, (b) the nascent and alendronate-modified membranes.

performance. The specific objectives are as follows: (1) to prepare amine-based bifunctional alendronates including alendronate monosodium (A-1Na), alendronate disodium (A-2Na), alendronate trisodium (A-3Na) and alendronate tetrasodium (A-4Na) (Fig. 1a); (2) to graft these bifunctional alendronates onto the PA layers of membranes using one-step reactions (Fig. 1b); (3) to assess the FO performance and explore applications of the charged membranes in desalination. Alendronates from A-1Na to A-4Na were deployed to develop novel FO membranes for the first time. Grafting alendronates onto membrane surfaces by covalent bonds via a one-step reaction is much simpler and more efficient than those with multistep reactions and avoids various chemicals including catalysts and initiators which are frequently involved in other modification approaches [8,19,20]. Amino, hydroxyl and phosphate groups grafted to the membrane surfaces would substantially increase the membrane hydrophilicity which benefits FO remarkably [8].

2. Materials and methods

2.1. Materials

Poly(ether sulfone) (PES) polymer from Solvay Advanced Polymer, L.L.C. was deployed to fabricate the substrates. n-Methyl-2-pyrrolidone (NMP, Aladdin) served as solvent and polyethylene glycol 400 (PEG 400, Fuchen) acted as pore former in PES dope preparation. m-Phenylenediamine (MPD, 99%, Macklin), 1, 3, 5-benzenetricarboxylic chloride (TMC, 98%, Micxy) and n-hexane (FuChen) were deployed to synthesize the PA layer coated on the subtracted surface. A-1Na (99%) obtained from Meryer was used as both the membrane modification reagent and a starting material of other alendronates. Small organic reagents of ethylene glycol, diethylene glycol, triethylene glycol and glucose supplied by Fuchen were employed to characterize membrane surface structural properties including MWCO, mean pore size and pore size distribution. NaOH (FuChen) was used as a starting material to

synthesize alendronates. NaCl (FuChen) and MgCl₂ (FuChen) were both used as draw solutes for FO processes. Ethanol (EtOH, 99.7%, Xilong Co. Ltd) was deployed to purify the modification reagents. Deionized (DI) water produced by a Millipore water system was used as solvent or feed solution.

2.2. Synthesis of alendronates and membranes

2.2.1. Preparation of alendronate materials

A-2Na, A-3Na, A-4Na were synthesized via a stepwise neutralization reaction between A-1Na and NaOH in different ratios. Briefly, A-2Na was synthesized as follows. A-1Na and NaOH in equimolar amount were added to a flask containing DI water. The mixture was reacted at 60 °C and a homogenous solution was formed after 6-h stirring. Precipitate was then obtained when cold EtOH was added to the solution and purified with H₂O/EtOH. White powders of A-2Na were obtained after vacuum dry with a quantitative yield (> 95%). A-3Na and A-4Na were synthesized similarly except that twofold and threefold amounts of NaOH were used, respectively.

2.2.2. Fabrication of substrate and FO membrane

A PES solution with composition 15.0/42.5/42.5 (wt%) PES/NMP/PEG 400 was prepared for the PES substrate fabrication, as established elsewhere [21]. The preparation details are as follows. Certain amounts of PES/NMP/PEG 400 according to the above proportion were loaded to a 250 mL three-neck flask. The mixture was stirred overnight at 60 °C to ensure the PES polymer dissolved completely. The resultant dope solution was then set aside 12 h to release air bubbles. Subsequently, The PES membrane substrate was fabricated following the procedures in the literature [21]. Briefly, PES dope solution was poured on a flat glass plate and a casting knife in 100-μm thickness was used to cast the substrate. The glass plate was then put into a coagulation bath of DI water. The substrate film was subsequently transferred to another coagulation bath for further solvent exchange and left for the follow-up

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