



Enhanced performance of cellulose triacetate membranes using binary mixed additives for forward osmosis desalination



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HIGHLIGHTS

- Single additive optimization experiments were conducted for the design of the binary mixed additives (BMA).
- Effectiveness of the BMA on the FO performance was extensively evaluated and confirmed.
- ZnCl₂-LA enhanced both water flux and salt rejection by ~51.3% and ~8.1% compared to HTI CTA membrane, respectively.
- BMA significantly improved the water flux more than three times compared to the membranes with single additives.
- Trade-off effect between the selectivity and permeability was partially broken for our membranes with BMA.

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ABSTRACT

In this study, on a basis of the single additive optimization experiments of the cellulose triacetate (CTA) forward osmosis (FO) membranes, the CTA FO membranes with binary mixed pore-forming additives were designed and fabricated to retrofit the membrane structure and separation performance for FO desalination. The binary mixed additives such as acetic acid-lactic acid (AA-LA), acetic acid-maleic acid (AA-MA) as well as zinc chloride-lactic acid (ZnCl₂-LA) were selected via combining a single additive with the capacity to improve the salt rejection with another single additive with the capacity to improve the water flux. The effectiveness of the binary mixed additives on the FO performance was extensively evaluated and confirmed. For example, with the ZnCl₂-LA as binary additive, the resulting CTA membrane exhibited a much improved water flux of ~11.5 L/m² h and an excellent salt rejection of ~98.3% under the fixed FO operating condition (0.1 M NaCl solution as feed, 2 M glucose solution as draw solution, FO mode, 2 h), which were increased by approximately 51.3% and 8.1% compared to the commercial CTA membrane from HTI, respectively. Compared to the single additives, the binary mixed additives could significantly enhance the water flux for the CTA membranes with an insignificant reduction in salt rejection, which might depend on the interaction between two additives as well as the interaction between additives and solvent during the phase inversion. It indicated that the trade-off effect between the selectivity and permeability of a polymer membrane was partially broken for our membranes with binary mixed additives. This study helps us to pave the way to design highly efficient additives for high-performance FO membranes.

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1. Introduction

Scarcity of water resources is troubling the life of mankind due to water pollution and overpopulation over the past few decades [1]. To address the severe water scarcity problem, membrane-based separation processes such as nanofiltration (NF) [2,3] and reverse osmosis (RO) [4,5] have attracted considerable attention as a promising technology to significantly reduce the cost of water treatment involving seawater desalination and wastewater reclamation [6]. Recently, forward osmosis

(FO) [7] as an alternative desalination and water purification process, offers the advantages of low energy consumption, high rejection of a wide range of ion contaminants and lower membrane fouling propensity, compared to the traditional pressure-driven membrane processes such as NF and RO [2–5]. Along with the fast development in polymeric membranes and separation processes in the past decades [8–10], FO has gained extensive concerns in the fields of food processing [11], power generation [12], pharmaceutical concentration [13] and desalination [14].

Although tremendous efforts have been made to successfully fabricate high-performance FO membranes with flat sheet and hollow fiber configurations [15,16], most of these FO membranes are in lab-scale and there is the only commercially available FO membrane composed

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of cellulose triacetate (CTA) coated on polyester mesh developed by HTI (Hydration Technology Innovations, Albany, USA) [17–19]. The commercial FO membranes still suffer from severe internal concentration polarization (ICP) [20–24] which greatly reduces the effective driving force and leads to a significant decline in permeation flux, and reverse diffusion of draw solutes which increases the economic losses to replenish the draw solution, complicates feed solution concentrate management and enhances membrane fouling potential [25–29]. Therefore, new explorations and developments in membrane material and formation process are urgently needed to fabricate appropriate membranes specifically for FO to put forward the industrial implementation of the FO technology.

Compared to the thin-film-composite membranes made by interfacial polymerization, cellulose acetate-based membranes with special advantages such as excellent chlorine tolerance and higher solute rejection are considered to be more suitable for extensive oxidizing wastewater reclamation [30]. CTA, a commercial HTI FO membrane material as above mentioned, is one of the cellulose acetate-based materials for the membrane fabrication and has been applied in various separation applications. Due to the unique advantages such as relative hydrophilicity, low fouling propensity, good mechanical strength and wide availability [31], it is worthwhile to further investigate the phase inversion of CTA polymers to explore new progress that might have been overlooked in the past and yet can be good supplement to the traditional fabricating process, and also to search for better design of membrane structure for FO process for the ultimate purpose of wastewater reclamation.

Generally, using pore-forming additives is an effective strategy to control and optimize the membrane structure such as pore tortuosity, membrane porosity and thickness so as to improve the separation performance [32–35]. Chung et al. [32] blended acetic acid as pore-forming additive into the CTA casting solution with dioxane/acetone solvent system and found that the resulting FO membrane had a significantly more porous and open-cell sublayer structure, leading to the water flux increased from 3.5 L/m² h to 22.7 L/m² h using 2 M NaCl solution and DI water as draw solution and feed solution, respectively. Xu et al. [36] used cis-trans isomers as pore-forming additives to fabricate cellulose acetate (CA) FO membranes and explored the underlying correlations between additive molecular structure and membrane properties. Moreover, the pore-forming mechanism of individual pore-forming additive including acetic acid, lactic acid and maleic acid was investigated and proposed [37,38], which might form complex with solvent such as 1,4-dioxane in the casting solution and the complex may release during the phase inversion. Inorganic pore-forming additives such as zinc chloride (ZnCl₂) might form complex with acetone [39]. However, so far, nearly all existing studies relevant to pore-forming additives focused on the single additive with various additive types and contents dosed into the casting solution. Much less attention has been paid to the mixed additives and their effects on the membrane structure and separation performance of FO membranes. Synergistic effects of mixed additives will work under the appropriate mixing conditions to improve the membrane structure and performance. For example, with the aid of the right mixed additives, the resulting membrane might have a dense top surface and a nano-porous bottom surface simultaneously, probably leading to both high water flux and salt rejection. Therefore, it would be worthwhile to investigate the effects of the mixed additives on FO membranes to achieve an improvement of separation performance.

In this paper, CTA was employed as the polymeric material with binary mixed pore-forming agents for the fabrication of superior membranes for FO desalination. The objective of this study is to explore if we can further enhance the FO performance of CTA membranes via the binary mixed additives including (1) acetic acid/lactic acid, (2) acetic acid/maleic acid, and (3) zinc chloride/lactic acid, which were selected as pairs composed of one additive leading to an excellent FO flux and the other leading to an outstanding salt rejection on a basis of the single additive experiments. We believe that our work will make a

contribution to the better design for FO membranes in the application of wastewater reclamation as well as seawater desalination.

2. Experimental

2.1. Materials and methods

Cellulose triacetate (CTA) with an averaged acetyl content of 43–44 wt% was purchased from Acros Organics (USA). 1,4-dioxane, acetone and lactic acid (LA) were purchased from Tianjin Fuyu Fine Chemicals Co., Ltd. (China). Acetic acid (AA), zinc chloride (ZnCl₂), maleic acid (MA) and sodium chloride (NaCl) were purchased from Sinopharm Chemical Reagent Co., Ltd. (China). Glucose was purchased from Shandong Xiwang Sugar Company (China). Deionized (DI) water with a resistivity of 18 M Ω cm used in these experiments was produced by a Milli-Q water purification system. Commercial FO membranes made from cellulose triacetate (CTA) were purchased from HTI (Albany, OR) for comparison purpose.

2.2. Membrane preparation and characterization

CTA was blended with various additives to prepare FO membranes via non-solvent induced phase inversion. The casting solution composed of CTA, mixed solvents of 1,4-dioxane and acetone (the mass ratio is 14:71:15), and various pore-forming additives, were stirred at 55 °C for 7 h and then degassed at 45 °C for 5 h to form a homogeneous solution. The compositions of various casting solutions are presented in Table 1. Afterwards, under the room temperature of 22–25 °C and relative humidity of 70% adjusted by a humidifier, the casting solution was casted onto a horizontal glass plate using a 150 μm casting knife and then exposed to air for 30 s followed by immediate immersion in DI water bath for 20 min. After peeled off from the glass plate, the CTA membrane was annealed in deionized water at 85 °C for 5 min and finally stored in deionized water before use. Surface and cross-sectional morphologies of the CTA membranes were examined using a scanning electron microscopy (SEM) (S-4800, Hitachi, Japan). Samples were freeze-dried by a freeze drier (ModulyoD, China) and then fractured in liquid nitrogen followed by gold coating before measurements.

2.3. FO performance measurements

FO performance in terms of water flux and salt rejection of the resulting CTA membranes was determined by FO test in FO mode with 0.1 M NaCl solution as feed and 2 M aqueous glucose solution as draw solution. Lab-scale cross-flow filtration cell with an effective membrane area of 57.42 cm² was used in this study, allowing the draw solution and feed solution to flow counter currently on each profile of the membrane with the same velocity of 8.5 cm/s. During the FO test, the dilution of the draw solution can be ignored because the ratio of the water flux to the volume of the draw solution was <2%. The water flux (J_v , L/m²h) was determined from the weight change of draw solution and can be calculated as below.

$$J_v = \frac{\Delta w}{\rho A t} \quad (1)$$

where Δw (kg) is the mass of water permeated across the membrane from the feed solution to the draw solution over a predetermined time interval t (h) in the duration of tests; A (m²) is the effective membrane surface area; ρ (kg/m³) is the water density. The weight change of draw solution was monitored by a balance (BSA323S, SSIL, Beijing).

The salt concentration in the draw solution was determined by measuring the conductivity using a conductivity meter (DDS-307a) based

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