



Alginate fouling reduction of functionalized carbon nanotube blended cellulose acetate membrane in forward osmosis



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HIGHLIGHTS

- The fCNT-CA membrane showed enhanced performance than the bare CA membrane in FO.
- The fCNT-CA membrane became more hydrophilic and negatively charged.
- Dominant alginate foulant–membrane interaction force was firstly investigated.
- The fCNT-CA membrane was more repulsive against alginate foulant via AFM analysis.

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ABSTRACT

Functionalized multi-walled carbon nanotube blended cellulose acetate (fCNT-CA) membranes were synthesized for forward osmosis (FO) through phase inversion. The membranes were characterized through SEM, FTIR, and water contact angle measurement. AFM was utilized to investigate alginate fouling mechanism on the membrane. It reveals that the fCNT contributes to advance alginate fouling resistance in FO (57% less normalized water flux decline for 1% fCNT-CA membrane was observed than that for bare CA membrane), due to enhanced electrostatic repulsion between the membrane and the alginate foulant. Furthermore, it was found that the fCNT-CA membranes became more hydrophilic due to carboxylic groups in functionalized carbon nanotube, resulting in approximately 50% higher water-permeated flux than bare CA membrane. This study presents not only the fabrication of fCNT-CA membrane and its application to FO, but also the quantification of the beneficial role of fCNT with respect to alginate fouling in FO.

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1. Introduction

As water scarcity and energy exhaustion become severe, a paradigm of water treatment aims at obtaining clean water with less energy. Membrane process is replacing conventional process in drinking water due to its excellent retention of pollutants (Kim and Lee, 2011). However, fouling is a major issue in membrane process operation, as it causes severe water flux decline and a shortened lifespan of the membrane. Hence, adequate cleaning is necessary, but this requires large energy inputs (Lee et al., 2010). Recently, new types of membranes have been introduced to overcome this issue. The convergence of nanotechnology and membrane science has been actively attempted to functionalize

membranes with discrete nano-material named “nano-enhanced membranes (NEMs)”, which improves membrane performances (Pendergast and Hoek, 2011). NEMs have been developed with nano-sized zeolite, titania, alumina, iron oxide, silver, and carbon nanotube, resulting in enhanced membrane performance with regard to water flux, rejection, fouling resistance, and microorganism inactivation (Kwak et al., 2001; Yan et al., 2005; Taurozzi et al., 2008; Lind et al., 2010; Celik et al., 2011b; Park and Choi, 2011).

Reverse osmosis (RO) has been widely utilized in both industrial applications and academic researches, due to its high rejection rate of target material or pollutant. In desalination, large quantities of fresh water can be obtained from seawater via RO with lower energy costs rather than via distillation. However, RO also results in severe membrane fouling due to the high hydraulic pressures required. Biofouling is particularly problematic, with biofilm formation on the membrane surface by variable bacteria, which is

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difficult to control. High-molecular weight compounds are secreted by bacteria in biofilm, named extracellular polymeric substance (EPS). EPS is mostly composed of polysaccharides and proteins. It constitutes 50–90% of the total organic matter in biofilm (Donlan and Costerton, 2002).

Recently, forward osmosis (FO) has been studied not only for the direct production of freshwater from seawater but also for utilizing as pretreatment prior to water treatment and desalination. It utilizes osmotic pressure gradients alone, having great potential due to its advantages – no hydraulic pressure required, low operating cost, and low fouling tendency. Draw solute development, process optimization, and FO membrane fabrication are considered as core research areas for the real application of FO (Cath et al., 2006; Zhao et al., 2012). Various natural and artificial draw solutes have been studied, including NH_4CO_3 , intracellular proteins, magnetic draw solutes, fertilizer, and nanoparticles (McCutcheon et al., 2006; Adham et al., 2007; Ling et al., 2010; Ling and Chung, 2011; Phuntsho et al., 2011). A draw solute for FO should have high osmotic pressure, be easy to separate, and cause no chemical or physical damage to membranes (Zhao et al., 2012).

The reverse solute diffusion phenomenon has been reported as a critical factor for consideration in the draw solute for FO (Cath et al., 2006; Phillip et al., 2010). Monovalent ions, such as sodium chloride, have the potential to penetrate nonporous membranes. The diffusivity of draw solute strongly affects thicker fouling layer formation, due to cake enhanced osmotic pressure (CEOP) caused by concentration polarization (Hoek and Elimelech, 2003). Past studies have attempted to investigate membrane fouling reversibility in FO. As no hydraulic pressure is required in FO, accumulated fouling layers are less compacted onto membrane surfaces. Therefore, no chemical cleaning is necessary and physical cleaning is an effective means of restoring water flux to initial levels. Commercial RO membranes have been utilized for FO research before a cellulose triacetate (CTA) based polymeric membrane was synthesized by HTI Inc. (Herron, 2006). It triggered FO membrane fabrication research. Cellulose-based asymmetric membranes (Su et al., 2010) and thin-film composite membranes (Yip et al., 2010) have been widely utilized. The critical aspects of membrane performance are water flux, ion rejection, and fouling resistance.

Several studies of NEM for FO application have been reported, using functionalized carbon nanotube (Amini et al., 2013; Dumée et al., 2013; Goh et al., 2013; El Badawi et al., 2014), vertically aligned carbon nanotube (Baek et al., 2014), zeolite (Emadzadeh et al., 2014), and silver nanoparticle (Liu et al., 2013). In the case of CNT, enhanced water flux and hydrophilicity were mostly reported. Organic foulant has been applied to investigate the fouling tendency of the CNT-based NEM for FO.

In this study, a functionalized CNT blended polymeric composite membrane was fabricated and characterized, and its performance was evaluated with a lab-scale FO system. A cellulose acetate was chosen as a membrane polymer due to its good chlorine tolerance and anti-biofouling property (Lee et al., 2011). Alginate was chosen as a hydrophilic organic EPS foulant surrogate in biofilm, and alginate fouling reduction on the NEM was evaluated in a lab-scale FO system. Interaction force measurement between the alginate foulant and the membrane surface was conducted by atomic force microscope (AFM) for the first investigation of the major mechanism of alginate fouling on the synthesized NEM.

2. Materials and methods

2.1. Chemicals

Cellulose acetate (CA, average M.W. 30,000), nitric acid (HNO_3 , 70%), sulfuric acid (H_2SO_4 , 98%), 1,4-dioxane, acetone, methanol,

lactic acid, sodium chloride (NaCl), and sodium alginate were purchased from Sigma–Aldrich. Multi-walled carbon nanotubes (MWCNTs) were purchased from Hanhwa Nanotech., and calcium sulfate (CaSO_4) was purchased from Junsei. All of the aqueous solutions for the experiments were prepared using deionized (DI) water from a water purification system (Synergy, Millipore, USA) which has a resistivity of 18.2 $\text{m}\Omega\text{ cm}$. A commercial asymmetric cellulose triacetate (HTI FO) membrane was provided by Hydration Technology Innovations, Albany, OR.

2.2. Carbon nanotube functionalization and membrane fabrication

MWCNTs were functionalized in a mixture of nitric and sulfuric acids, in order to increase dispersion in organic solvent (Liu et al., 1998). The MWCNTs were refluxed in a 1:3 (v/v) HNO_3 and H_2SO_4 mixture at 100 °C. Following this, the MWCNTs were washed with DI water until the solution pH reached 7.0, and then dried overnight at room temperature. The dried MWCNTs were ultrasonicated in a 1:3 (v/v) HNO_3 and H_2SO_4 mixture at 70 °C for nine hours. Next, the MWCNTs were again washed with DI water until the pH of the solution reached 7.0, and the functionalized MWCNTs were left to dry in a vacuum oven.

When dried, the functionalized MWCNTs blended cellulose acetate (fCNT-CA) membranes were synthesized through a phase inversion method. The functionalized MWCNTs were ultrasonicated in 1,4-dioxane for homogeneous dispersion. After dispersion, cellulose acetate and acetone were added to the MWCNT solution. Methanol and lactic acid were subsequently also added and carefully mixed into the MWCNT and polymer solution. The chemical composition of MWCNTs and polymer solution is shown in Table S2. The polymer solution was ultrasonicated and placed at room temperature to remove air bubbles and then casted with a casting knife onto glass plates with a 280- μm casting thickness. The casted polymer film was then allowed to evaporate for one minute and then dipped in DI water for coagulation, and the synthesized membranes were stored in DI water.

2.3. Membrane characterization

The morphologies of the functionalized MWCNTs and fCNT-CA membranes were observed by SEM (S-4700, Hitachi, Japan). Their functional groups were analyzed using FTIR spectrometry (Nicolet iS10, Thermo Scientific, USA). The hydrophilicity of the membrane surface was evaluated through the dynamic sessile drop method, using a contact angle goniometer (Model 100, Rame-Hart, USA). A minimum of seven contact angles were averaged to ensure reliable values.

2.4. Lab-scale forward osmosis test

A laboratory-scale FO system was operated using the synthesized fCNT-CA membrane and the HTI FO membrane, in order to evaluate water-permeated flux and reverse solute flux. To determine the water-permeated flux, 3 M NaCl aqueous solution was used as a draw solution and DI water was used as a feed solution. To determine the reverse solute flux, 1 M NaCl solution was used as a draw solution and DI water was used as a feed solution. A cross-flow membrane test unit was used, in which the temperature of draw and feed solutions was controlled at 20 ± 1 °C with a water-cooled chiller (DH-003BH, Daeho Auto Chiller, Korea). The solutions in the test unit were circulated with a peristaltic pump (BP-60601, Won Corp., Korea) with a 1000 cm^3/min crossflow rate and an effective membrane area of 20 cm^2 . The water-permeated flux was measured using a calculation in which the permeated volume rate was divided by the effective membrane area

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