



Effects of additives on dual-layer hydrophobic–hydrophilic PVDF hollow fiber membranes for membrane distillation and continuous performance

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ABSTRACT

The advantages of the implementation of dual-layer hydrophobic–hydrophilic hollow fiber membranes for membrane distillation (MD) have been highlighted in this work. The effects of incorporating methanol as a non-solvent additive and self-synthesized fluorinated silica (FSi) particles as a hydrophobic modifier on the resultant membrane morphology and MD performance were investigated. Employing a 3.5 wt% sodium chloride solution at 80 °C, the highest direct contact membrane distillation (DCMD) flux of 83.40 ± 3.66 kg/(m² h) and separation factor higher than 99.99% were attained for the membrane spun with methanol additive. Moreover, the stability of the dual-layer hydrophobic–hydrophilic hollow fiber membrane has been demonstrated through continuous DCMD experiments for 5 days. The separation factor was maintained higher than 99.99% for the membrane spun with methanol additive, verifying the suitability of the dual-layer hydrophobic–hydrophilic hollow fiber membrane configuration for desalination processes. The morphological transformation of the outer membrane surface from a porous agglomerated globule structure into a denser interconnected globule structure may be accounted for by the stability improvement of the membrane spun with methanol additive. On the other hand, it was found that an enhanced hydrophobicity of the membrane spun with FSi particles did not result in an improvement of the membrane stability. The existence of the hydrophilic hydroxyl group on the FSi particle surface may favor the occurrence of membrane wetting.

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1. Introduction

Water crisis, which is defined as shortage in the availability of fresh water resources relative to its demand, has been considered as one of the most important issues in this century. As the world population continually increases, water shortage is predicted to become more severe in the future (Service, 2006). Water crisis is also exacerbated by the limited availability of fresh water resources on earth. From the total amount of 1360 million km³ of water on earth, only about 37 km³ comprises fresh water. The amount of accessible fresh water is further reduced to around 0.126 km³ as the rest of the fresh water is stored in inaccessible ground water or locked in icecaps (Clarke, 1993). To overcome these problems, desalination processes have received great attention as an alternative route for fresh water production from saline water.

Nowadays, the common drawback of conventional thermal desalination processes, such as multi-stage flash (MSF) and multiple effect distillation (MED) is their high energy consumption (El-Dessouky et al., 1999; Fiorini and Sciuabba, 2007; Service,

2006; Van Der Bruggen and Vandecasteele, 2002). The highly fluctuating crude oil price towards higher prices further reduces their competitiveness. On the other hand, reverse osmosis (RO) process is limited by the saline water concentration and fouling (Mariah et al., 2006; Wirth and Cabassud, 2002). Among various attempts to develop more versatile desalination processes, MD process, an old technology that was firstly patented by Boddell (1963), stands out and receives worldwide attention. MD operability at a lower temperature and pressure has made it outstanding compared to conventional desalination processes (Al-Obaidania et al., 2008; Lawson and Lloyd, 1997). Furthermore, the feasibility of MD to be coupled with solar (Hogan et al., 1991; Koschikowski et al., 2003) and geothermal energy (Bouguecha and Dhabbi, 2003) or low-grade heat sources intensifies its attractiveness (Charcosset, 2009; Khayet, 2011).

The working principle of MD is the utilization of a porous hydrophobic membrane as a physical barrier between the hot feed side, where an aqueous solution is evaporated, and the distillate side, where the transported water vapor is condensed. Obviously, membrane hydrophobicity plays an important role in ensuring that only water vapor gets transported across the membrane while inhibiting the penetration of liquid feed. Based on this concept, a total rejection of non-volatile solutes can be theoretically achieved

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in MD (Burgoyne and Vahdati, 2000). In addition to water repellency property, MD membranes should also exhibit 2 main features: (1) low thermal conductivity to reduce conductive heat loss and (2) low transport resistance towards diffusion of vapor molecules to enhance permeation flux. These features require membrane scientists to exclusively design membranes with desirable morphological and transport properties.

Awareness of the coupling effect between mass and heat transfer phenomena is essential in designing membranes for the MD process (Ding et al., 2002; Gryta and Tomaszewska, 1998). A good membrane should maximize the extent of mass transport, yet minimize the extent of heat transport. Thus, compared to other membrane separation processes, the membrane parameters may have different effects on MD performance (flux and separation). Considering mass transfer alone, the relation between membrane parameters and the resultant MD flux can be written as follow (Lawson and Lloyd, 1997):

$$N \approx \frac{r^\alpha \varepsilon}{\delta_m \tau} \quad (1)$$

where N is the molar flux, r is the membrane mean pore size, α is a constant factor that equals 1 for the Knudsen diffusion and 2 for viscous flow, ε is the membrane porosity, δ_m is the membrane thickness, and τ is the membrane tortuosity. According to Eq. (1), a thinner membrane is favorable for mass transfer as it reduces resistance for the transport of water vapor. On the contrary, a thinner membrane increases conductive heat loss through the membrane matrix (El-Bourawi et al., 2006). It was reported that by modeling the competing effect between mass and heat transports, an optimum membrane thickness was estimated to be in the range of 30–60 μm (Lagana et al., 2000).

The hydrophobic–hydrophilic membrane configuration for MD was first introduced by Cheng and Wiersma (1982, 1983). Tailoring membrane thickness within the optimized range becomes possible with this configuration as the hydrophilic sub-layer beneath can provide mechanical support. Following this, there have been a number of publications on the fabrication of hydrophobic–hydrophilic membranes using various methods. Modification of cellulose acetate membranes by a radiation graft polymerization method was reported by Wu et al. (1992) while modification of cellulose nitrate membranes by a plasma polymerization method was reported by Kong et al. (1992). However, the additional modification step required by both approaches moderates their competitiveness. One-step fabrication of hydrophobic–hydrophilic composite membrane was proposed by Khayet and Matsuura (2003), Khayet et al. (2005), and Qtaishat et al. (2009b) in a series of publications. Enhancement of flat-sheet membrane surface hydrophobicity casted from hydrophilic materials was achieved by the incorporation of so-called surface modifying macromolecules (SMMs). Bonyadi and Chung (2007) were the first to propose the fabrication of hydrophobic–hydrophilic hollow fiber membranes by a dual-layer co-extrusion spinning technology. By increasing thermal conductivity properties of the hydrophilic sub-layer with the aid of graphite particles and multi-wall carbon nanotubes (MWNT), Su et al. (2010) enhanced the MD flux of the hydrophobic–hydrophilic hollow fiber membranes.

Depending on the degree of severity, membrane wetting was classified into four categories, i.e. non-wetting, surface wetting, partial wetting, and fully wetting (Gryta, 2007). In the case of surface wetting, the interface of the liquid and vapor is shifted inward of the membrane cross section. Nevertheless, distillate with a high purity can be produced as long as the gaseous gap between the feed and the distillate is maintained. As the MD operation continues, the feed and distillate solutions may penetrate deeper, resulting in a partial membrane wetting. In the case of partial wetting, the MD operation can be continued if the majority of the

pore channels are dried though a gradual decline of the distillate purity is normally observed. Membrane wetting has been regarded as one of the major barriers for the industrial implementation of MD process (El-Bourawi et al., 2006; Khayet, 2011). Thus, research efforts have been devoted to the development of MD membranes with better wetting resistance. However, at present, most of the developed membranes still suffer certain degree of wetting.

Several mechanisms were proposed to explain the wetting phenomenon: (1) membrane fouling and scaling (Gryta, 2007), (2) a large gap between hydraulic pressure at the opposing sides of the membrane (Gryta and Barancewicz, 2010), (3) formation of bigger membrane pores under high pressures (Gryta, 2005), and (4) oxidation of hydrophobic groups on the membrane surface to form hydrophilic chemical moieties, such as carbonyl and hydroxyl groups (Gryta et al., 2009). The longest MD operation of a single-layer polypropylene (PP) hollow fiber membrane in which RO permeate was employed as a feed was reported by Gryta (2005). It was found that during the 3 years' operation time, the membrane surfaces are firstly wetted and as the MD operation continues, the liquid penetrates deeper towards the membrane cross-section, resulting in the flux decay and separation performance deterioration.

According to previously explained wetting mechanisms, a thinner membrane may be more prone towards wetting and hence, questions may arise on the applicability of hydrophobic–hydrophilic membranes for MD application. Interestingly, based on the authors' best knowledge, the wetting propensity of hydrophobic–hydrophilic membranes under long-term MD operation has not been investigated by any researchers. Therefore, the objectives of this study are as follows: (1) to design the dual-layer hydrophobic–hydrophilic hollow fiber membranes with suitable morphological and transport properties by understanding the mechanism of phase inversion process; (2) to explore the effects of methanol as a weak non-solvent additive and fluorinated silica particle as a hydrophobic modifier on the membrane formation, membrane morphology, and the resultant MD performance; and (3) to investigate the stability and wetting resistance of the fabricated dual-layer hydrophobic–hydrophilic hollow fiber membranes under continuous DMCD operation.

2. Materials and methods

2.1. Materials

Commercial polyvinylidene fluoride (PVDF) HSV 900 was purchased from Arkema Inc. while commercial polyacrylonitrile (PAN) was kindly provided by Prof. Juin-Yih Lai and Prof. Hui-An Tsai from Chung Yuan Christian University of Taiwan. Both PVDF and PAN powders were dried at 60 °C overnight under vacuum before using. Hydrophilic cloisite NA^+ clay particles were purchased from Southern Clay Products. *N*-methyl pyrrolidone (NMP) as a solvent, methanol (MeOH) as a non-solvent, and ethylene glycol (EG) as a pore-forming additive were purchased from Merck. Heptadecafluoro-1,1,2,2-tetrahydrodecyltriethoxysilane (FOS) and tetraethyl orthosilicate (TEOS) for the synthesis of FSi particles were supplied by ABCR and Sigma Aldrich, respectively. Ethanol from Merck and aqueous ammonium hydroxide (28–30%) from Sigma Aldrich were used as a solvent and a catalyst for the synthesis of FSi particles, respectively. Sodium chloride was purchased from Merck and deionized (DI) water was produced by a Milli-Q unit from Millipore. All of the chemicals were of reagent grade and used as received.

2.2. Synthesis of the fluorinated silica (FSi) particles

The synthesis procedure of FSi particles was derived with modifications from the method described by Wang et al. (2008a,

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