

A novel dual-layer composite membrane with underwater-superoleophobic/hydrophobic asymmetric wettability for robust oil-fouling resistance in membrane distillation desalination

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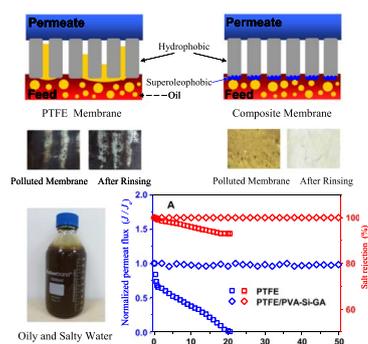
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GRAPHICAL ABSTRACT



ARTICLE INFO

Keywords:

Membrane distillation
Desalination
Membrane fouling
Composite membrane
Underwater superoleophobicity

ABSTRACT

In this study, we developed a new type of composite membrane to mitigate oil fouling in membrane distillation (MD). The composite membrane consists of a polytetrafluoroethylene (PTFE) hydrophobic substrate and a hydrophilic poly(vinyl alcohol)/silica nanoparticles (PVA-Si) hybrid fibrous coating prepared via sol-gel and electrospinning. We characterized the pristine PTFE membrane and the modified membrane using contact angle measurements and tensiometer-based oil probe force spectroscopy. While the hydrophilic coating presented excellent oil fouling resistance, the glutaraldehyde cross-linking treatment augmented the in-air hydrophilicity and underwater superoleophobicity of the electrospun coating surface. The cross-linking treatment also prevented the formation of PVA-Si hydrogels for the electrospun hybrid fibers, which made availability to utilize the water-soluble polymer to modify hydrophobic membrane for anti-oil-fouling MD. By comparing the performance of the composite membrane and the pristine PTFE membrane in MD desalination experiments using a saline emulsion with 1000 mg/L crude oil, it showed that the fabricated composite membrane was significantly more resistant to oil-fouling compared to the PTFE membrane. The results from this study suggest that underwater

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superoleophobic coating can effectively mitigate oil fouling in MD, and that the fabricated composite membrane can enable MD to desalinate hypersaline wastewater with high concentrations of hydrophobic contaminants.

1. Introduction

Membrane distillation (MD) is a non-isothermal membrane process through which water is separated in the form of vapor from an aqueous salty solution [1]. In a MD process, there is a thermally driven vapor transport through non-wetted hydrophobic membrane where the driving force is the partial vapor pressure difference across the two sides of membrane [2–4]. MD can operate at relatively low temperature and is thus able to tap into the vast amount of low-grade waste heat [5,6]. MD is also advantageous over conventional pressure-driven membrane processes, such as nanofiltration (NF) or reverse osmosis (RO), as its low operating pressure reduces the capital cost due to the absence of expensive components, such as high pressure pumps and vessels, as well as pressure exchangers [7–9]. Recently, there have been extensive studies on the application of MD for water desalination, wastewater treatment, recovery of valuable resources from wastewater, and treatment of radioactive wastes [10–15].

The hydrophobic membrane, which acts as a physical barrier and provides a liquid-gas interface for heat and mass exchanges, plays a crucial role in realizing the MD process [16]. Currently, most commercially available MD membranes are typically made of hydrophobic materials, such as polytetrafluoroethylene (PTFE), polyvinylidene fluoride (PVDF) and polypropylene (PP) [17–19]. However, hydrophobic membranes used for MD are inherently prone to fouling by hydrophobic organics such as hydrocarbons and natural organic matter due to the long-range hydrophobic-hydrophobic interaction [20,21]. The necessity of using hydrophobic membranes in MD and the abundance of hydrophobic contaminants in many saline waste streams limit the application of MD [22].

In recent years, material scientists have developed high-performance underwater anti-oil-adhesion surfaces inspired by natural surfaces of similar interfacial properties, such as those of fish skin, mussel shell, and sharkskin [23–26]. These biological interfaces highlight the importance of both surface morphology and interfacial tension in imparting excellent anti-adhesion or anti-wetting performance [27,28]. Following those principles, a wide variety of underwater oleophobic meshes and membranes have been prepared for oil-water separation [29–32].

Underwater oleophobic surfaces are typically in-air hydrophilic. Although the hydrophilic biomimetic solid surfaces can definitely offer new insights to fabricate anti-oil-fouling membranes, the underwater oleophobic membranes can not be directly applied in MD process, because hydrophobicity is necessary for the membrane used in MD to prevent liquid from permeating through the membrane pores [33]. Based on the above analysis, it has been clear that the key requirement for an anti-oil-fouling MD membrane is the construction of a composite membrane structure with a hydrophilic underwater oleophobic surface to repel oil fouling, and a hydrophobic substrate to serve as the liquid barrier and vapor gap. It essentially means that a composite membrane with underwater-oleophobic/hydrophobic asymmetric wettability is required.

Following this assumption, anti-fouling composite membranes have been developed using in-situ formation of a hydrophilic skin layer [34] or spraying of a hydrophilic nanoparticle-polymer composite on the top of a hydrophobic PVDF membrane [35]. Membranes with hydrophilic/hydrophobic composite structure have also been designed for the purpose of enhancing vapor flux, with the rationale that a thin hydrophobic layer can reduce the pathway for vapor transfer and thus enhance the vapor permeability of the membrane [36,37]. In other cases, composite materials have also been employed to fabricate

superhydrophobic and omniphobic membranes to enhance MD performance [38–40]. However, one challenge of applying a hydrophilic skin onto a hydrophobic substrate is the possibility of introducing wetting defects due to possible coating of the inner pores. If pores are rendered hydrophilic through the depth of the membrane even over very small percentage of membrane area, the salt rejection will decline to an unacceptable level [41]. A very effective approach to overcome this potential problem is to employ electrospinning to form a hydrophilic fibrous skin layer, as it is extremely unlikely for electrospun fibers with ultrahigh aspect ratios to penetrate through the tortuous pores.

Electrospinning is a promising and versatile approach for fabrication of nanometer- or submicrometer-sized fibrous networks under the application of a strong electric field to a polymer or biopolymer liquid solution [42–45]. Electrospun fibrous networks have high porosity and interconnected open structure [46], which can minimize the additional mass transport resistance to the underlying hydrophobic membrane. Previous studies have demonstrated successful preparation of electrospun fibrous membranes for microfiltration (MF), ultrafiltration (UF) and NF [47–50]. Here, we explore the possibility and effectiveness of using electrospinning to prepare composite membrane with underwater-oleophobic/hydrophobic asymmetric wettability for anti-oil-fouling MD.

In this study, we fabricated a novel composite MD membrane with exceptional oil-fouling resistance. The composite membrane has a PTFE hydrophobic substrate and an underwater superoleophobic poly(vinyl alcohol)/silica nanoparticles hybrid fibrous coating formed by sol-gel technology and electrospinning method. The morphology and wettability of the composite membrane and the pristine PTFE hydrophobic membrane were investigated. The interactions between oil droplet and membrane surface were measured to compare the propensities of oil adhesion to different membrane surfaces. Finally, direct contact MD (DCMD) experiments with saline feed solution containing a relatively high concentration of crude oil (1000 mg/L) were carried out to show the drastically different anti-fouling behaviors of the pristine PTFE and the composite membrane.

2. Materials and methods

2.1. Materials and reagents

PTFE hydrophobic flat sheet membrane used polyester fabric as support was supplied by Sano Membrane Technology Engineering Co., Ltd. Poly(vinyl alcohol) (PVA) polymer (99% hydrolyzed, Mw = 89,000–98,000), tetraethyl orthosilicate (TEOS), dimethyl sulfoxide (DMSO), glutaraldehyde (GA), NaCl and HCl were provided by Sigma-Aldrich. The crude oil, as the fouling agent, was acquired from Daqing Oil Field of China National Petroleum Corporation. Deionized (DI) water was from a Milli-Q ultrapure water purification system (Millipore, Billerica, MA).

2.2. Preparation of PVA/silica sol

At first, PVA (10.0 g) was dissolved in 90 mL DI water under magnetic stirring at 85 °C for 5 h to prepare aqueous 10 wt% PVA solution. After complete dissolution of the PVA polymer, 5.0 g DMSO and 0.3 g HCl were added into 32.0 g PVA solution to improve the electrical conductivity of the polymer solution and stirred continuously for 2 h at room temperature. To prepare PVA/Silica sol, 3.0 g TEOS was dropped slowly into the mixture solution and stirred for 3 h at 30 °C to reach completion of the hydrolysis reaction of the TEOS.

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