



Amphiphobic surface modification of electrospun nanofibrous membranes for anti-wetting performance in membrane distillation

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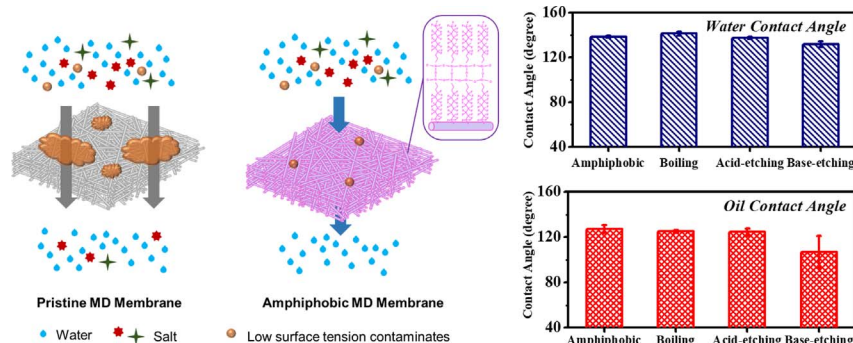
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GRAPHICAL ABSTRACT



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ABSTRACT

Membrane distillation (MD) works as a promising alternative of RO to desalinate hypersaline water owing to its unique advantages including high concentration brine tolerance, 100% theoretical solute rejection, and low operating pressure. Hydrophobic MD membranes face critical challenge to treat industrial wastewaters containing low surface tension substances because these substances led to membrane wetting with loss of membrane permeability and rejection. In this study, we report a simple and facile approach to fabricate amphiphobic polyvinylidene fluoride-co-hexafluoropropylene (PVDF-HFP) electrospun nanofibrous membranes with anti-wetting property. 1H,1H,2H,2H-perfluorodecyltriethoxysilane (FAS) was used to fluorinate PVDF-HFP fibers and was then crosslinked to form a network upon dealcoholization under thermal treatment. The FAS-coated PVDF-HFP nanofibrous membranes exhibited great stable amphiphobicity with high contact angles against both water and oil, even challenged under critical conditions including boiling water, and strong base and acid etchings. Surface fluorination of PVDF-HFP nanofibrous membrane with the FAS coating did not sacrifice its permeate flux and salt rejection much. The fabricated amphiphobic nanofibrous membrane exhibited robust MD performance in the long-term operation and also in the presence of surfactant sodium dodecyl sulfate in the feed saline solution.

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1. Introduction

Membrane-based water purification technologies have been demonstrated as effective and efficient approaches to produce fresh water from seawater or wastewater [1,2]. In particular, reverse osmosis (RO) works as a state-of-the-art membrane process to remove salt from water [3]. However, RO faces great challenges to treat hypersaline water because the required operation pressure to overcome the osmotic pressure of hypersaline water is far beyond the tolerance of RO membrane [4]. Recently, membrane distillation (MD) has been developed as a superior alternative of RO to desalinate hypersaline water owing to its unique advantages including high concentration brine tolerance, 100% theoretical solute rejection, and low operating pressure [5,6]. MD is a hybrid thermal/membrane separation process, where only water vapors pass through a hydrophobic microporous membrane driven by vapor pressure gradient [7,8], and salt is rejected independently of salt concentration. However, MD membranes are often easily wetted by low surface tension organic contaminants which are ubiquitous in hypersaline industrial wastewaters such as oil, alcohol, and surfactants [9,10], and thus lose their rejection properties. Therefore, it is highly desired to develop robust anti-wetting MD membranes for treating the challenging wastewater sources from major industries, including textile, paper, printing, drugs, cosmetics, coal-fired plant, oil and gas industry [7,11,12].

Recently, amphiphobic surfaces, which have a contact angle (CA) greater than 90° to both water and low surface tension liquids, have been found to exhibit an excellent wetting resistance against both water and low surface tension liquids [13,14]. The most important affecting parameters on surface amphiphobicity are surface roughness and surface energy [15]. Amphiphobic surfaces having high surface roughness and low surface energy are beneficial for completely repelling low surface tension liquids. The creation of micro/nano hierarchical structures on surfaces can effectively improve surface roughness and provide multilevel re-entrant structures to facilitate a metastable Cassie-Baxter state for the solution-membrane interfacial contact, preventing the fully wetting Wenzel state for low surface tension liquids [14]. Plasma etching, photolithography, and electrochemical treatment have been applied to create surfaces having a re-entrant structure, and to achieve the superamphiphobicity [16,17]. Surface energy can be reduced through surface fluorination via silanization and coating of fluoroalkyl molecules. For example, coating of fluorinated-decyl polyhedral oligomeric silsesquioxane (FD-POSS) and fluoroalkyl silane (FAS) on polyester fabrics dramatically improved the contact angles of water, hexadecane, and tetradecane to 171° , 155° , and 152° , respectively [18]. However, most of these surface construction strategies were developed for improving amphiphobicity for textile fabric materials or impermeable substrates such as silicon [19], glass [20], metal [21] and others [22,23], which might not be applicable to porous membrane surfaces since the pore structure or pore sizes would be damaged or affected during those treatments.

Till very recently, amphiphobic porous surfaces have been fabricated successfully for robust anti-wetting membrane distillation application. Generally, nanoparticles such as silica nanoparticles (SiNPs) [9,10,24–26] and TiO_2 [27] nanoparticles were used to coat porous membrane surfaces for creating multilevel re-entrant structures, and fluoroalkanes were engineered on membrane surface for lowering surface energy. To immobilize nanoparticles on porous membrane surface, it generally requires pre-treatment of substrates to create charges or functional groups promoting strong interaction with nanoparticles [9,10,25,26]. For example, in order to bind SiNPs on membrane surface, the phase-inversion PVDF membrane was treated with alkaline (7.5 M NaOH solution) at 70°C for 3 h to generate hydroxyl groups and then grafted with 3-aminopropyl triethoxysilane (APTES) for positive surface charges, and finally immobilized negative-charged SiNPs on membrane surface via electrostatic attraction, followed by coating SiNPs with perfluorodecyltrichlorosilane (FDTS) via vapor-phase

silanization [28]. Although the SiNPs-coated PVDF membrane achieved excellent wetting resistance against low surface tension liquids, densely populated SiNPs severely blocked membrane surface pores to decrease membrane permeability by more than 30%. Furthermore, these amphiphobic surface engineering approaches usually involve cost-expensive chemicals and time-consuming multiple steps such as pre-functionalization of pristine porous membrane, nanoparticle synthesis and immobilization, surface fluorination and thermal treatment. Numerous surface treatment procedures often led to a significant water flux decline of the fabricated amphiphobic membranes.

Herein, we develop a simple and facile method to fabricate amphiphobic nanofibrous membranes and to demonstrate their anti-wetting properties during dynamic membrane distillation operation. The advantage of electrospun nanofibrous membrane was taken of its intrinsic reentrant geometry, and its excellent amphiphobicity was achieved with low surface energy material coating. Therefore, we first prepared poly (vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) nanofibrous membrane through electrospinning, and then coated 1H,1H,2H,2H-perfluorodecyltriethoxysilane (FAS) on membrane surface through a simple dip-coating process, and finally improved the membrane amphiphobicity through thermal treatment. We also investigated the surface chemistry, morphology, stability and wetting-properties of the fabricated nanofibrous membrane, and examined the impacts of surface modification on membrane performance in the DCMD process. Furthermore, we challenged the amphiphobic nanofibrous membrane with sodium dodecyl sulfate (SDS, a model surfactant)-containing feed saline solution to promote wetting, and investigated its stability and performance in the DCMD process. Our research provides new insights on fabricating robust amphiphobic nanofibrous membrane for anti-wetting performance in membrane distillation.

2. Experiments

2.1. Materials and chemicals

Poly (vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP, M_w : 455,000 g/mol) and 1H,1H,2H,2H-perfluorodecyltriethoxysilane (FAS, 97%) were purchased from Sigma-Aldrich (St. Louis, MO, USA). All other solvents and reagents, including sodium chloride (NaCl), acetone, *N,N*-dimethylacetamide (DMAc) and *n*-hexane were analytical grade and purchased from Shanghai Sinopharm Chemical reagent CO. LTD (China). All chemicals were used as received.

2.2. Fabrication of PVDF-HFP nanofibrous membrane

PVDF-HFP nanofibers were electrospun following the reported conditions [29] from 10 wt% PVDF-HFP solution in a mixture of acetone/*N,N*-dimethylacetamide (DMAc) (7:3, w/w) and collected on the rotating drum with naturally accumulating into a nanofibrous membrane. During the electrospinning, the PVDF-HFP solution was fed to the spinneret at 1.0 mL/h, and 20 kV DC voltage was applied between the spinneret and the rotating drum, which were 15 cm apart in parallel.

2.3. Heat-press treatment of PVDF-HFP nanofibrous membrane

The fabricated electrospun PVDF-HFP nanofibrous membrane was sandwiched between two nonwoven PET fabrics and pressed for 10 s under the pressure of 0.7 kPa in a thermal stage (IKA, German) at the temperature varying from 100°C , 120°C , 130°C to 140°C , respectively. In addition, the heat-press time of nanofibrous membranes varying from 10 s, 20 s, 60 s, 120 s to 300 s was also investigated under the pressure of 0.7 kPa at 130°C .

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