



Contents lists available at ScienceDirect

Separation and Purification Technology

journal homepage: www.elsevier.com/locate/seppur

A simple coating method to prepare superhydrophobic layers on ceramic alumina for vacuum membrane distillation

Chun-Yung Huang, Chia-Chieh Ko, Liang-Hsun Chen, Ching-Ting Huang, Kuo-Lun Tung, Ying-Chih Liao *

Department of Chemical Engineering, National Taiwan University, Taipei, Taiwan

ARTICLE INFO

Article history:

Received 23 August 2016

Received in revised form 27 October 2016

Accepted 6 December 2016

Available online xxxx

Keywords:

Vacuum membrane distillation

Ceramic membrane

Superhydrophobic surface

ABSTRACT

In this work, a simple coating method was developed to create a superhydrophobic solid layer on a ceramic alumina substrate for vacuum membrane distillation without sintering. A silica/alumina nanoparticle mixture was formulated to create a gel solution for hydrophobic coating. The oxide nanoparticles were first modified with a fluorinated monolayer, and the solution was stirred overnight for gelation. The gel was coated over an alumina disk and dried to form a superhydrophobic coating with an extremely high water contact angle of 158° and an average pore size of $0.4\ \mu\text{m}$ to prevent water leakage under vacuum conditions. The vacuum desalination performance of the coated membrane was tested by exposing the coated membrane to salt solutions. The influence of operational parameters, such as NaCl concentrations, flow rate, and temperature of feed solutions, were also examined carefully. A high flux of $29.3\ \text{L m}^{-2}\ \text{h}^{-1}$ with rejection rate of 99.9% can be obtained for 3.5 wt% NaCl solution at 70°C . With the simple preparation process without sintering and excellent desalination performance, the modification process for ceramic membrane in this work shows great promise in practice and can be further extended for industrial applications.

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1. Introduction

The demand for fresh water has increased immensely over the past decades, and thus investigations for new water treatment processes are rising dramatically. Among these processes, desalination is known as a clean process with stable resources and low pollutions [1,2]. However, traditional desalination methods, such as reverse osmosis (RO) and thermal evaporation, usually need high energy supply. Alternatively, membrane distillation (MD), a highly efficient membrane separation process, provides a vital technology to practical industries with low energy needs [3–5]. MD is a thermally driven separation process in which water vapor molecules evaporate at the hot side of the membrane, pass through the pores of membrane by vapor pressure differences, and condense into water at the other side. This liquid/gas phase separation process can yield in a theoretical 100% salt rejection rate [6,7]. Besides, MD is also an energy-saving process: the temperature gradient across the membrane can be obtained from low-grade heat sources, such as waste heat, solar energy or geothermal energy [4]. However, to maintain the separation of liquid and gas phases,

the membrane must be non-wetted. Otherwise the feed salt solutions will fill or pass through the pores, and reduce the separation efficiency. As a result, a porous hydrophobic membrane must be prepared to prevent penetration of aqueous feed.

Challenges remain unresolved in the fabrication of the hydrophobic membranes for MD. Intrinsic hydrophobic material, such as polypropylene (PP), polytetrafluoroethylene (PTFE), polyvinylidene fluoride (PVDF), are commonly used in MD process [8–14]. To further increase the hydrophobicity of polymer membrane, the polymer surfaces can be coated with a fluorinated silica layer to form a superhydrophobic composite membrane [15]. However, the high temperature sensitivity or swelling phenomenon of polymeric membrane results in short working life. Compared to polymeric membranes, ceramic membranes usually possess high mechanical strength, thermal resistance, and chemical tolerance, which are desirable for MD. However, ceramics are usually hydrophilic due to the hydroxyl groups of the metal oxide surface. To make hydrophobic ceramic membranes, fluoroalkylsilane (FAS) are regularly as a surface modifier to promote the membrane hydrophobicity for MD applications [16–26]. In these researches, phase inversion and sintering methods are regularly used to prepare ceramic substrates with sub-micron pores, following with a hydrophobic surface modification with FAS. The hydrophobic character of the sufficiently small pores provides a liquid/gas

* Corresponding author.

E-mail addresses: r04524033@ntu.edu.tw (C.-Y. Huang), jackko0213@gmail.com (C.-C. Ko), b00504041@ntu.edu.tw (L.-H. Chen), amy800327@gmail.com (C.-T. Huang), kltung@ntu.edu.tw (K.-L. Tung), liaoy@ntu.edu.tw (Y.-C. Liao).

separation barrier and results in a better permeate flux for MD. However, the pore size control of the modified ceramic membrane is solely determined by the sintering process, which usually needs high temperature or pressure, and therefore it is relatively difficult to achieve the hydrophobic modification simultaneously with good pore-size control.

In this study, we aim to develop a practical approach, which can yield in small pore size and great hydrophobicity at the same time without sintering, to create a superhydrophobic solid layer on ceramic alumina substrates for vacuum membrane distillation (VMD). Silica/alumina nanoparticles with a FAS monolayer are first formulated properly to create a gel solution. After gel coating on the ceramic membrane and drying, a thin solid layer is formed and the nanoparticle stacking leads to sub-micron pore passages with superhydrophobicity. With this thin superhydrophobic coating as a water leakage barrier, the porous alumina membrane can still maintain its originally great permeability for a high permeate flux. The hydrophobicity, morphology, pore size distribution as well as MD desalination properties of the coated membrane will be carefully examined to investigate the feasibility of this coating method.

2. Experiments

2.1. Materials and gel formulation

A commercial colloidal silica solution (RS-30, Rongxiang Ltd., Taiwan) was used. The aqueous silica solution contains 0.6 wt% Na₂O (pH = 9) as colloidal stabilizer and 30 wt% silica particle (mean diameter ~10 nm). Alumina powder (Advanced ceramics nanotech) with an average particle size of 100 nm was added into the silica solution to form a solution with alumina/silica ratio of 1:10 in weight. 1H,1H,2H,2H-Perfluorodecyltriethoxysilane (FAS, >97%, Alfa-Aesar, USA) was diluted in ethanol (>99.8%, Sigma-Aldrich, USA) to form a 2 wt% FAS solution. The alumina/silica and the FAS/ethanol solutions were mixed at various ratio in weight at room temperature and stirred vigorously. After stirred for 24 h, the mixture became an opaque gel. Alumina disks

(2.4 mm thickness and 47 mm diameter, Kinik Company, Taiwan) with an average 2.4 μm pore size and 42.8% porosity were used as the coating substrate. To enhance the adhesion between the alumina substrate and the as-prepared gel, the alumina disks were immersed in silica solution for 30 min and dried. Then, the alumina/silica gel was deposited by a brush evenly on the alumina disk and dried in ambient air overnight. After the coating/drying process, a composite oxide membrane disk with hydrophobic top layer was prepared with a net weight increase of ~0.6 g.

2.2. Characterization

Fourier transform infrared (FT-IR) spectroscopy (Perkin Elmer Spectrum 100) was used to identify the chemical binding in the gelation process with a resolution of 4 cm^{-1} . The gel samples were dried in oven for 24 h to form powder type and compressed to tablets before measurements. The contact angles were observed with an in-house goniometer by depositing 3 μL deionized water or various concentration of sodium chloride (99.5%, Acros, USA) droplets on the membrane surfaces. Surface morphology of the membrane was examined by using scanning electron microscope (Nova NanoSEM, FEI, USA). Mercury Porosimeter (micromeritics AutoPore® IV 9520) was further used to analyze the pore size distribution of the modified membranes. A home-made instrument was used to measure the liquid entry pressure. The membrane sample was fixed in a module. The coated side of the membrane was in contact with deionized water, and the other side connected to a collector. Pressurized nitrogen was fed into the cylinder and exerted on the water. Once the water penetrated the membrane and flowed into the collector, the value of pressurized was recorded.

2.3. Membrane performance

Membrane distillation performance was conducted by using a home-made MD setup (Fig. 1). The circular alumina membrane was fixed with a thermally stable silicone rubber ring. Sodium chloride ($\geq 99.0\%$, Seedchem, Australia) aqueous solutions with

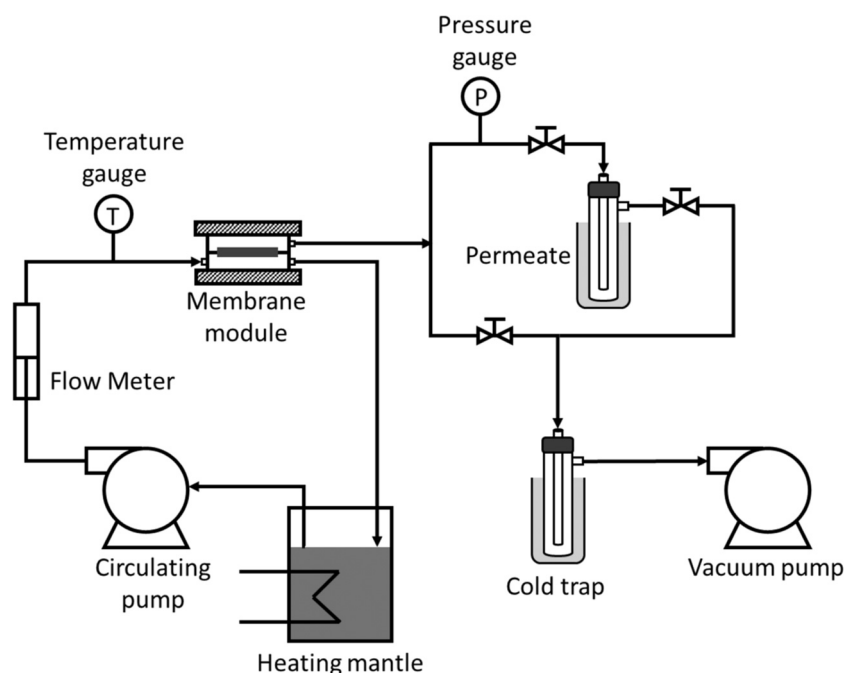


Fig. 1. Schematic illustration of vacuum membrane distillation setup.

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