

## Superhydrophobic polytetrafluoroethylene hollow fiber membrane based on fluoroalkylsilanes for vacuum membrane distillation

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### ABSTRACT

Membrane material plays an important role in the membrane distillation technology. However, traditional membrane materials such as polytetrafluoroethylene (PTFE), poly(vinylidene fluoride) and PP have existing membrane pollution and membrane infiltration problems, which limit further development of the membrane distillation technology. To solve these problems, the 1H,1H,2H,2H-perfluorodecyltriethoxysilane (PFDTES) has been added to the PTFE solution to create a novel super-hydrophobic PFDTES-PTFE membrane. A series of characterizations and characterization techniques have accordingly been used to study the structure and performance of the prepared membranes, such as atomic force microscope (AFM), water contact angle (WCA) measurements, liquid entry pressure (LEP) tests, vacuum membrane distillation (VMD) experiments, etc. The AFM analysis indicated a decreased roughness of the PFDTES-PTFE membrane. Moreover, the WCA of the membrane was increased from 131.3° to 137.5°, and the LEP increased by 33.3%, which indicated that the PFDTES-PTFE membrane showed excellent hydrophobicity and anti-wetting performances. What is more, the mean pore size was reduced from 269.6 to 205.0 nm, but there was a minor difference in film thickness. Furthermore, the VMD tests were implemented to evaluate the performance of the original and modified membranes with aspect to wetting. Compared to the pristine membrane, the modified PTFE membrane showed a slight decrease in membrane flux in the VMD process. However, the modified membrane kept a stable membrane flux about 0.932 L/(m<sup>2</sup>·h) with a salt rejection values of 99.8% after distillation for 3 h. In a word, the present study has proposed a new method to extend the membrane life, in favor of further developments of the membrane technology for islands and reef fields.

**Keywords:** Desalination; Vacuum membrane distillation; Super-hydrophobic PTFE membrane; Anti-wetting performance

### 1. Introduction

In recent years, the lack of fresh water has become one of the most serious issues of human society. An increasing number of scientists have regarded the seawater desalination technology as the most effective solution [1,2]. Among

the numerous desalination techniques, membrane separation technologies are considered as the most promising methods. They include reverse osmosis (RO), forward osmosis (FO), and membrane distillation (MD) [3]. The MD technology is a more feasible separation method compared to RO and FO, since it has several main advantages, such

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as low working pressure, nearly 100% of salt rejection, mild operating conditions, high permeation flux, and utilization of renewable energy sources [4–7]. It is divided into four types according to different condensation types: direct contact membrane distillation (DCMD), vacuum membrane distillation (VMD), air gap membrane distillation (AGMD), and sweep gas membrane distillation (SGMD). Nowadays, it is widely recognized that VMD is the most promising technology, which is due to a lower heat loss and higher permeation flux [8–12].

The MD is a thermally driven process, which utilizes the vapor pressure difference between the bulk feed solution and the cooling solution. The bulk feed solution induces the evaporation of water, and the evaporated water is condensed in the cooling solution by a hollow fiber membrane [13]. Thus, the heat is transferred from the bulk-feed solution to the membrane-feed interface and, thereafter, permeates through the membrane. It finally detaches to the downstream side in a vapor state finally [14]. Thus, the hollow fiber membrane must have excellent selectivity to prevent any unnecessary substances to pass through the membrane. It must also have an excellent anti-wetting property. Common hollow fiber membranes include polytetrafluoroethylene (PTFE), polyetherimide (PEI), and poly(vinylidene fluoride) (PVDF) [15–17]. However, due to fouling or wetting issues, the traditional membranes can easily fail after a long-time operation process, especially for the situation of a higher permeate flux, which reduces the separation efficiency and limit further successful applications. Therefore, it is necessary to find an effective method by which it is possible to control the membrane wetting problems.

In order to solve these problems, many efforts have been made to create superhydrophobic membranes by using the sol-gel method [18], electro-spinning method [19,20], plasma surface treatment technology [21], blending modification method, and surface modification method. The blending modification method and the surface modification method are the most common methods for the preparation of superhydrophobic membranes, which is due to an easy operation condition and lower cost. The blending modification method refers to the mixing of polymers with inorganic nanoparticles in the preparation of membranes. The mixing can increase the water flux and stability because of an improved porosity and hydrophobicity. Wang et al. [22] prepared a PVDF membrane that was modified with ZnO nanorods-PFDTES (1H,1H,2H,2H-perfluorodecyltriethoxysilane). It exhibited a stable super-hydrophobicity, easy-cleaning property, and excellent thermal and mechanical stabilities. Yadav et al. [23] fabricated super-hydrophobic Si@PVDF membranes by using PVDF solutions with silica nanoparticles and an indirect modification way. The water contact angle (WCA) was increased up to  $154.6^\circ \pm 2.2^\circ$ , and the VMD test showed a modified membrane with strong wetting resistance and excellent salt rejection. Alghamdi et al. [24] utilized a polyvinyl chloride (PVC) that was modified with magnetite nanoparticles (MNPs) by using a phase inversion method. Compared to the PVC membrane, the nanocomposite membrane showed excellent water flux, solute rejection, and anti-fouling properties. Furthermore, Zhai et al. [25] prepared a honeycomb-like polyelectrolyte multilayer surface

that was overcoated with silica nanoparticles. It demonstrated an excellent superhydrophobicity.

The surface modification method refers to the formation of a new membrane surface with low surface energy by using deposition, copolymerization, surface coating, or other ways. Yang et al. [26] prepared super-hydrophobic ceramic membranes based on 1H,1H,2H,2H-perfluorooctyltrichlorosilane (PFAS) and 1H,1H,2H,2H-perfluorodecyltriethoxysilane (PFDS) under ultraviolet irradiation conditions. The modified membrane showed a high membrane flux, excellent hydrophobicity, and an almost 99.99% salt rejection rate. Moreover, Ding et al. [27] used polydimethylsilane and polyvinylidene fluoride to modify the polypropylene membrane by using the filter coating method and phase inversion. The results showed that the membrane can keep a stable flux of over 12.2 kg/(m<sup>2</sup>·h) in a 120 h VMD process. Furthermore, Yu et al. [28] fabricated a composite membrane with a Nafion layer on a PTFE substrate membrane. The contact angle test showed that the modified membrane had an underwater super-oilphobic property.

A coating with fluorinated silica layer is considered to be an effective method for preparing hydrophobic membranes [29], and the fluoroalkylsilanes are regarded to be a common modifier for the improvement of the hydrophobic properties [30,31]. Marczak et al. [32] investigated the influence of alkylsilane and fluoroalkylsilane modifiers on the hydrophobic properties. The results showed that the hydrophobic properties increased with an increase in modifier chain length, and the fluoroalkylsilanes had the lowest surface free energy. Bystrzycka et al. [33] studied the influence of 1H,1H,2H,2H-perfluorodecyltrichlorosilane (FDTS) and 3,3,3-trifluoropropyltrichlorosilane (FPTS) on the wettability of the Si-DLC coatings by using vapor phase deposition. The results showed that the modified DLC coatings had a lower value of the friction coefficient, high hydrophobicity, and low surface free energy.

In this paper, PTFE has been used as a hollow fiber membrane for VMD. Also, PFDTES was chosen as a modifier due to its low surface energy and high hydrophobicity [21]. Moreover, atomic force microscope (AFM), WCA and LEP were used to characterize the structure and properties of the super-hydrophobic PTFE composite membrane. In addition, VMD tests of 3.2% NaCl solution were carried out in order to evaluate the composite membrane performance. The results showed that the modification membrane had an excellent anti-wetting performance and good stability, which indicated that the membrane was suitable for applications in the MD engineering field.

## 2. Experiments

### 2.1. Materials

PTFE membranes were purchased from Nanjing Zhongke bidun New Membrane Technology Co., Ltd., China. 1H,1H,2H,2H-perfluorodecyltriethoxysilane (PFDTES, 96%) and n-hexane (AR) were provided by Shanghai Aladdin Biochemical Technology Co., Ltd., China and Tianjin Kermel Chemical Reagent Co., Ltd., China, respectively.

## 2.2. Preparation of the PFDTES-PTFE composite membrane

The PFDTES-PTFE composite membranes were prepared as follows: Firstly, the dimension of the PTFE membrane was 6 cm (length) × 5 cm (width) with an effective membrane semi-diameter of 4.5 cm according to the size of the membrane cell. This membrane was immersed in ethanol and underwent an ultrasound cleaning for 0.5 h. Then, 1 mL PFDTES was added to 100 mL n-hexane to obtain a PFDTES solution. The dried PTFE membrane was immersed in the PFDTES solution for 24 h at the room temperature. Finally, the soaked PTFE membrane was washed with n-hexane to remove unreacted PFDTES, and dried in vacuum at 130°C for 2 h. The resulting membrane named as PFDTES-PTFE was then stored in a dry and clean environment.

## 2.3. Characterization

The membrane surface morphology and roughness were characterized by using scanning electron microscopy (SEM, HITACHI S-4800, Japan) and atomic force microscope (AFM, NT-MDT, Russia). Furthermore, the hydrophobicity of the membranes was analyzed by using water contact angle measurements (WCA, DSA30S, Krüss GmbH Germany). In addition, the pore sizes of the membranes were measured by using a capillary flow porometer (3H-2000PB, China) and the bubble pressure method. Moreover, the salt rejection was studied by using a conductivity meter (HQ30d, Hach, USA). Also, the membrane liquid permeability (LEP) test was performed by using a laboratory homemade membrane device.

## 2.4. Vacuum membrane distillation experiment

In order to evaluate the permeability and anti-wetting of the modified membrane, the performances of the PTFE membrane and PFDTES-PTFE composite membrane were analyzed by using a home-made VMD system as shown in Fig. 1.

Firstly, the modification membrane was placed in the circular membrane cell. Thereafter, the feed solution including

3.2% of NaCl was heated to 80°C in a water bath, where it was kept during the whole experiment. As the next step, the peristaltic pump was opened (301.13 mL/min) to control the flow velocity, and to keep a stable recycling of feed solution between the water bath and the membrane cell. Next, the vacuum pump was turned on and kept at 0.08 MPa continuously. The steam went through the membrane pores from the hot side of the membrane to the cold side and was collected by a conical flask. The duration of the experiments was at least 3 h, and they were repeated three times.

The permeate flux  $J$  (L/(m<sup>2</sup>·h)) and salt rejection  $R$  (%) were used to estimate the membrane performances, and they were calculated by using Eqs. (1) and (2), respectively.

$$J = \frac{m}{A \cdot t} \quad (1)$$

where  $m$  is the mass of the permeate,  $A$  is the effective membrane area of the hollow fiber membranes, and  $t$  is the operation time.

$$R = \frac{C_f - C_p}{C_f} \times 100\% \quad (2)$$

where  $c_f$  is the concentration of the feed solution, and  $c_p$  is the concentration of the permeate.

## 3. Results and discussion

### 3.1. Membrane morphology

Figs. 2 and 3 present the SEM and AFM of the original and modified membranes. It is obvious that the original membrane differs from the modified membrane. As shown in Fig. 2, the surface of the original membrane had a typical melting and tensile morphology, as well as a relatively uniform pore structure.

The AFM 2D images of the PTFE membrane and PFDTES-PTFE composite membrane are shown in Fig. 3. As compared with the original membrane (with a roughness of 0.193 μm), the roughness of the modified membrane surface was somewhat decreased, with an average

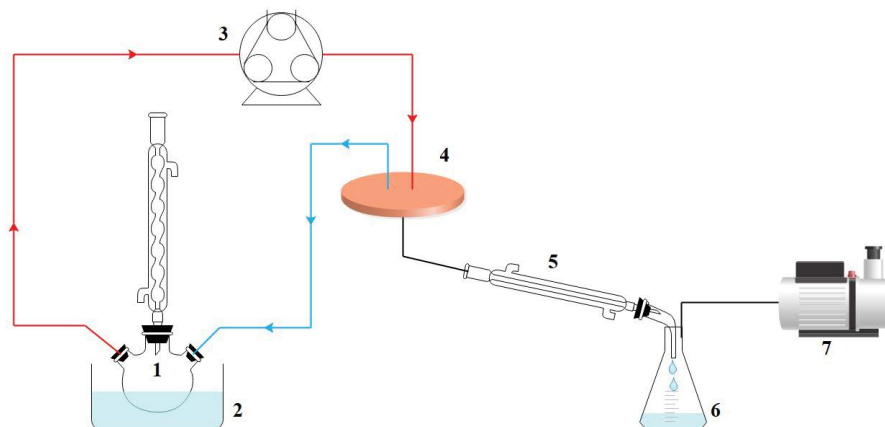


Fig. 1. Schematic of the VMD experiment (1) feed solution; (2) water bath kettle; (3) peristaltic pump; (4) membrane cell; (5) condenser; (6) conical flask; (7) vacuum pump.

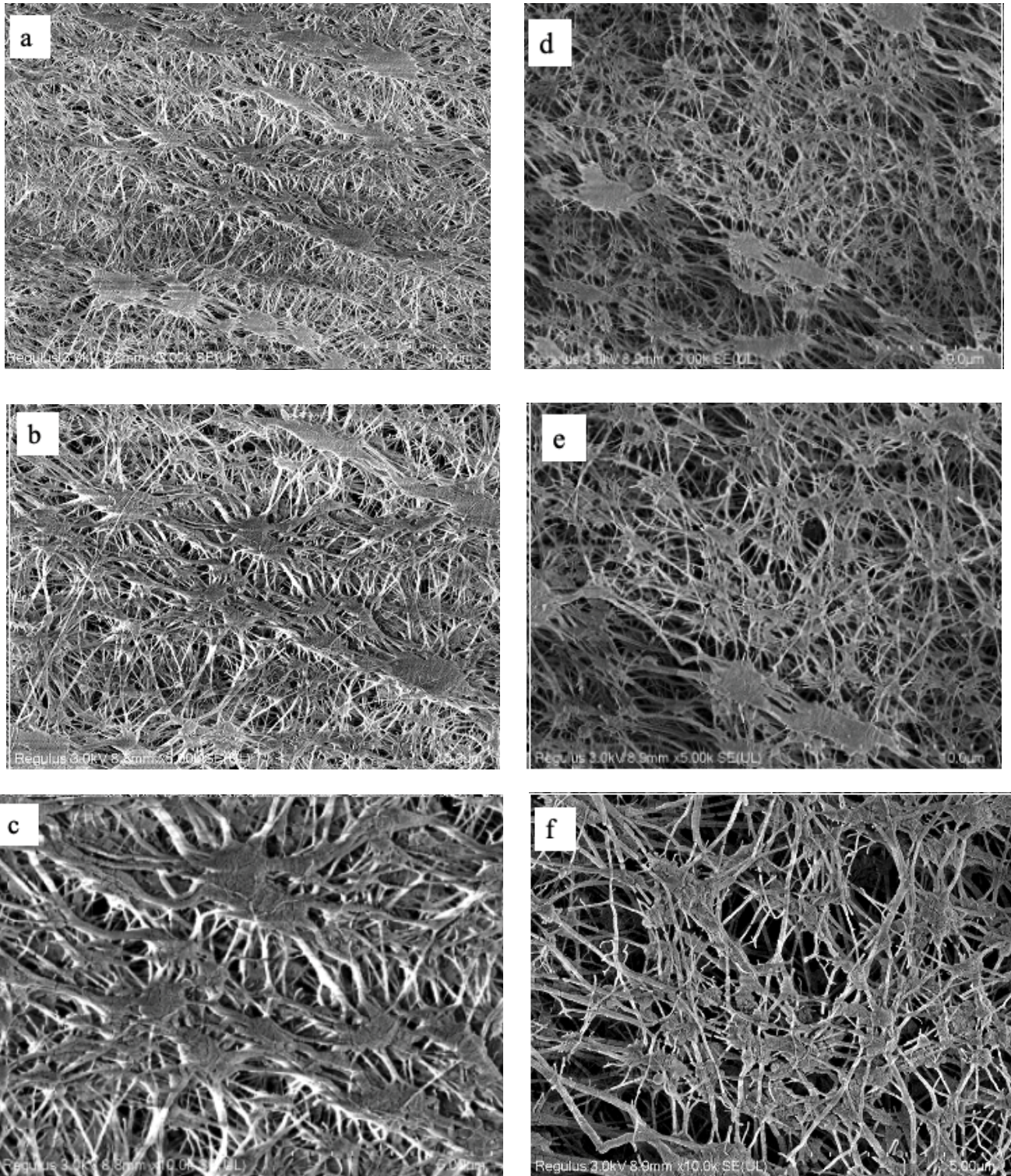


Fig. 2. SEM of the original PTFE membrane (a–c) and the modified PFDTES-PTFE composite membrane (d–f) at 1,000 magnification times (a,d), 3,000 magnification times (b,e), and 5,000 magnification times (c,f).

roughness of 0.165  $\mu\text{m}$ . This is because some surface morphology inhomogeneities have been eliminated in the modified PFDTES.

3.2. Membrane hydrophobicity

The super-hydrophobic hollow fiber membrane can achieve a high selectivity permeability in the VMD experiment. Theoretically, the hydrophobicity of the membrane depends on the geometrical structures and surface energy of the material [34,35]. It is also well known that the PFDTES possess a lower surface energy, so it has a higher hydrophobicity. As compared with the original membrane, the WCA of the composite membrane was increased from 131.3° to 137.5°, which indicated that the modified membrane had a good hydrophobicity (as shown in Fig. 4). The reason is that PFDTES had a lower surface energy due to the fluorinated ( $-\text{CF}_3$ ) groups on the surface.

3.3. Pore size, porosity, and membrane thickness

Pore size and porosity are crucial factors for the VMD separation performances. Generally, the higher the porosity of the membrane, the higher the permeability. The mean pore size, smallest pore size, and membrane thickness of the original and modified membranes are summarized in Table 1. The mean pore size was reduced from 269.6 to 205.0 nm upon modification. This can be explained by

a partial coverage of the pores by the PFDTES compound layer, and an occupation of the PTFE membrane gap. Compared to the original membrane, the thickness of the composite membrane was almost identical. However, the results suggest that the modified process had a significant effect on the membrane pore size and porosity.

3.4. LEP measurements

The liquid entry pressure (LEP) is an important parameter by which it is possible to evaluate the hydrophobicity of the membrane. It can be influenced by the membrane pore size, the surface energy of the liquid, the properties of the membrane material, etc. It can also be calculated by using [Eq. (3)] (i.e., the Laplacian equation).

$$\text{LEP} = \frac{-2B\gamma_L \cos\theta}{r_{\text{max}}} \tag{3}$$

where  $B$  is the geometric factor which can be determined by the membrane pore,  $\gamma_L$  is the feed liquid surface tension,  $\theta$  is the liquid-solid contact angle, and  $r_{\text{max}}$  is the largest pore radius in the membrane.

The membrane liquid permeability test was performed by using a homemade membrane device. The membrane was cut into a circular membrane with a diameter of 30 mm, and then placed in a membrane pool with the front side pointing upwards. Thereafter, the power supply of the

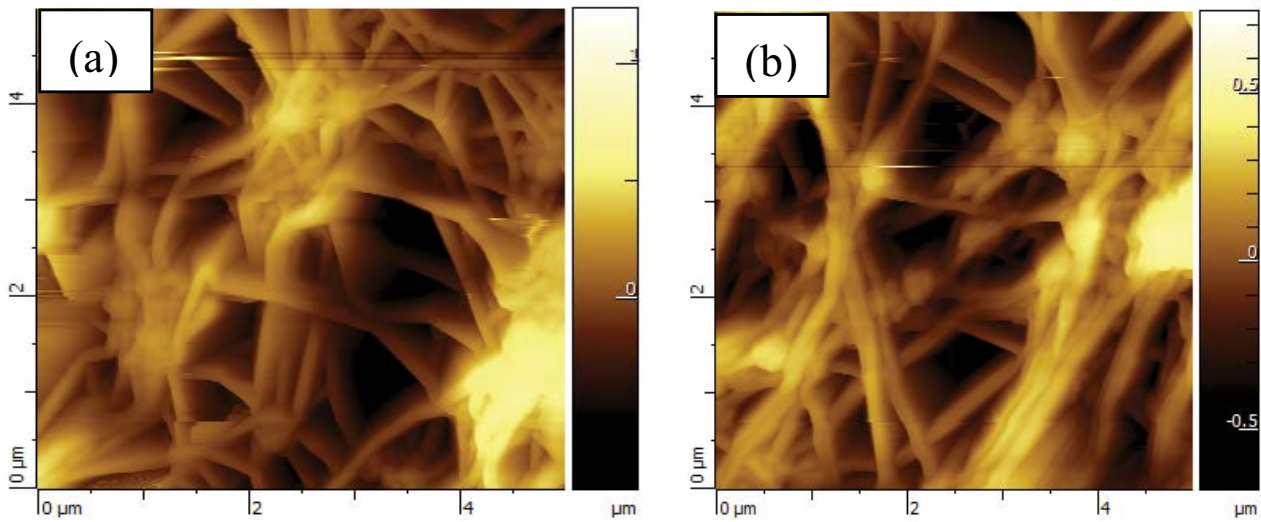


Fig. 3. AFM of PTFE membrane (a) and PFDTES-PTFE composite membrane (b).

Table 1  
Pore size, porosity and membrane thickness of PTFE and PFDTES-PTFE

Membrane	Mean pore size (nm)	Minimum pore size (nm)	Membrane thickness ( $\mu\text{m}$ )	Porosity (%)
PTFE	269.6	229.2	64	74.4
PFDTES-PTFE	205.0	155.0	65	70.3

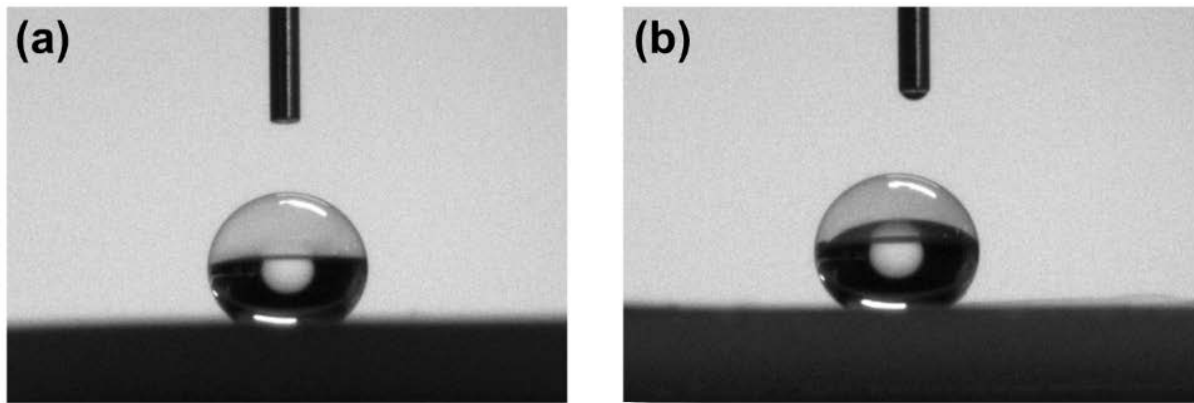


Fig. 4. WCA of PTFE membrane (a) and PFDTES-PTFE composite membrane (b).

device was turned on and the valve was fully open. After the liquid in the pipeline became stable (i.e., with no bubbles), the valve was gradually closed while the automatic pressure recorder was turned on. The pressure on the first drop of liquid on the membrane surface is the osmotic pressure of the liquid via the membrane. Five samples were selected from each membrane for testing, and an average of measured data was calculated after three distinct measurements.

The LEP of the pristine membrane and of the hydrophobic membrane were 0.36 and 0.48 MPa, respectively. After super-hydrophobic modifications, the  $\cos\theta$  was increased due to the improvement of the hydrophobicity of the membrane surface, which corresponded with an increase in WCA. However, the  $r_{\max}$  was kept almost unchanged.

### 3.5. VMD tests

VMD experiments were conducted to investigate the performances of the modified membrane. Fig. 5 shows the flux of the original and modified membranes at different conditions. It is well known that the permeate flux of the PFDTES-PTFE composite membrane generally decreases as compared with the original membrane, which is a result of a decreased pore sizes and increased mass transfer resistances. Thus, the membrane flux is related to both pore structure and roughness [36].

Based on the WCA and LEP data, the composite membrane showed an excellent hydrophobicity. Although the membrane flux of the composite membrane was somewhat lower than the flux of the original membrane, the membrane stability did also play a vital role in the VMD tests. Therefore, VMD experiments were also conducted to investigate the stability of the PFDTES-PTFE composite membrane.

Fig. 6 shows the membrane flux and salt rejection of both the original membrane and the composite membrane. Pollutants including surfactants, microorganism, organic acid, etc. are easily absorbed on the surface and pores size of the PTFE membrane. This causes an increase in surface tension and a reduction of the hydrophobic property of the membrane, thereby causing serious membrane fouling. It will even result in a failure of the vacuum membrane distillation. For example, Field et al. obtained a decrease in

flux when using Ceramesh and PVDF membranes with surfactant adsorbates [37].

In the present study, the membrane flux of a composite membrane (0.923 L/(m<sup>2</sup>·h)) was at first found to be slightly lower than the flux of an original membrane (1.038 L/(m<sup>2</sup>·h)). After 1 h, the addition of 0.1 mmol/L sodium dodecyl sulfate (SDS) resulted in a gradual increase in the flux of the PTFE membrane (from 1.045 to 1.550 L/(m<sup>2</sup>·h)). In addition, the salt rejection decreased rapidly from 99.9% to 98.0%, which indicated that the membrane surface occurred wetting phenomenon, which was due to the low surface tension of the SDS. When adding 0.2 mmol/L SDS, the original membrane became immediately completely wet, and the SDS passed the membrane together with water. This phenomenon indicated that the membrane had completely failed. However, the flux of the composite membrane was kept stable for 3 h, and the salt rejection had not decreased basically in the same condition. As compared with the PFDTES-PTFE membrane, the reason is that SDS is more easily adsorbed on the surface and on the pores of the PTFE membrane, which may cause serious membrane pollutions. This can increase the tension of the membrane surface and cause hydrophobic reduction of the membrane surface and pore diameter. It can even increase the hydrophilicity of the PTFE membrane, and cause a failure of the membrane distillation. Thus, the flux of the PTFE membrane has increased, and the salt rejection has decreased [38]. Although the PFDTES-PTFE membrane can be loaded with a surfactant, it is difficult to adsorb pollutants on the surface, and on the pores due to its large hydrophobic angle. Also, the hydrophobicity of the PFDTES-PTFE membrane has been improved, which is due to the lower surface energy of the PFDTES coating. In addition, the membranes can resist the membrane wetting that is caused by SDS to some extents [24]. The results have shown that PFDTES-PTFE composite membranes have a good hydrophobicity and can provide the necessary pre-conditions for interesting and important applications.

## 4. Conclusions

In this article, the modified PFDTES-PTFE membrane was constructed by using a simple method, which clearly

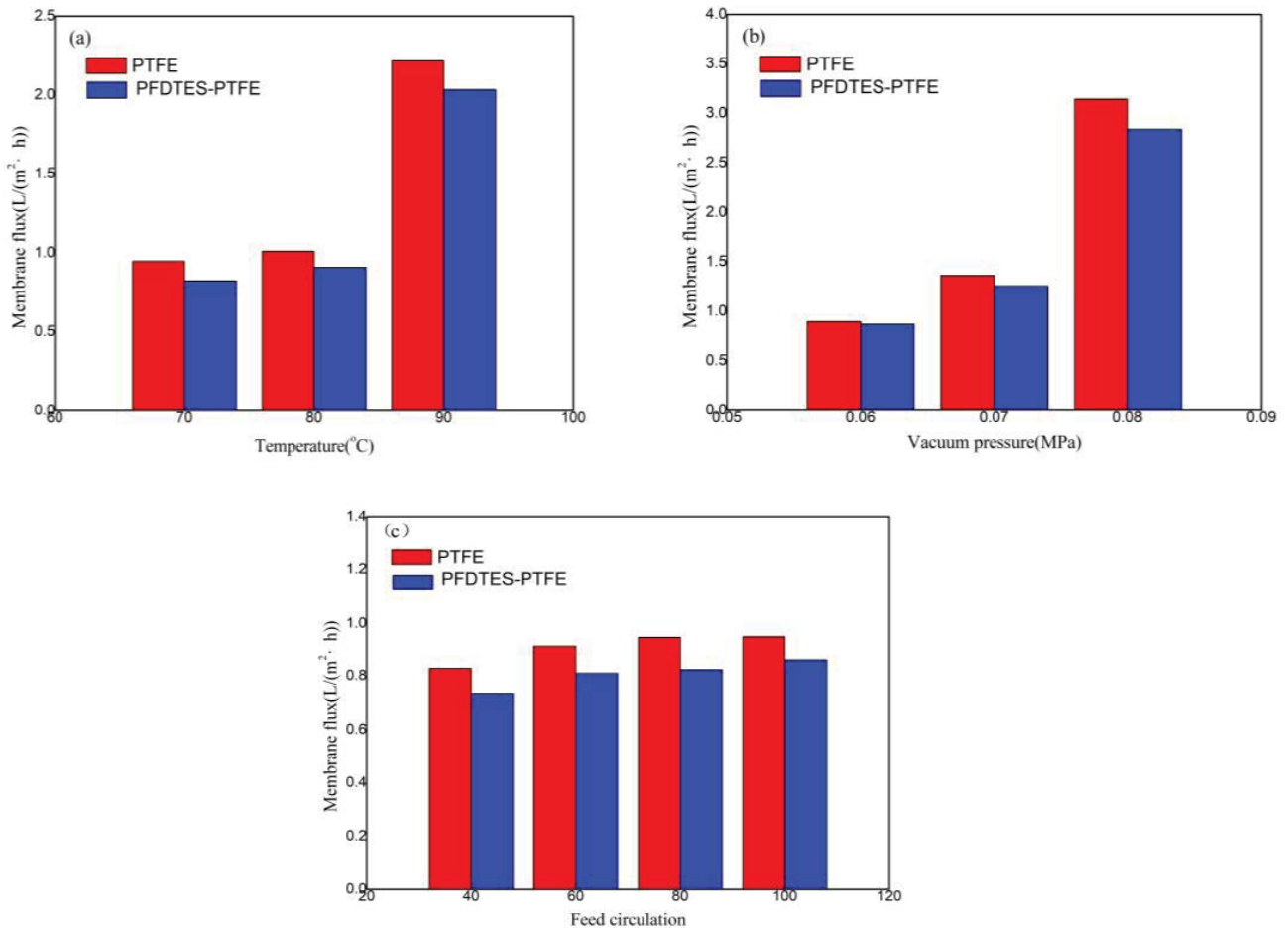


Fig. 5. The change trend of membrane flux with temperature at 80 flow rate of 0.08 MPa (a), change trend of membrane flux with vacuum pressure at 100 flow rate of 90°C (b) and change trend of membrane flux with feed circulation at 90°C of 0.08 MPa (c).

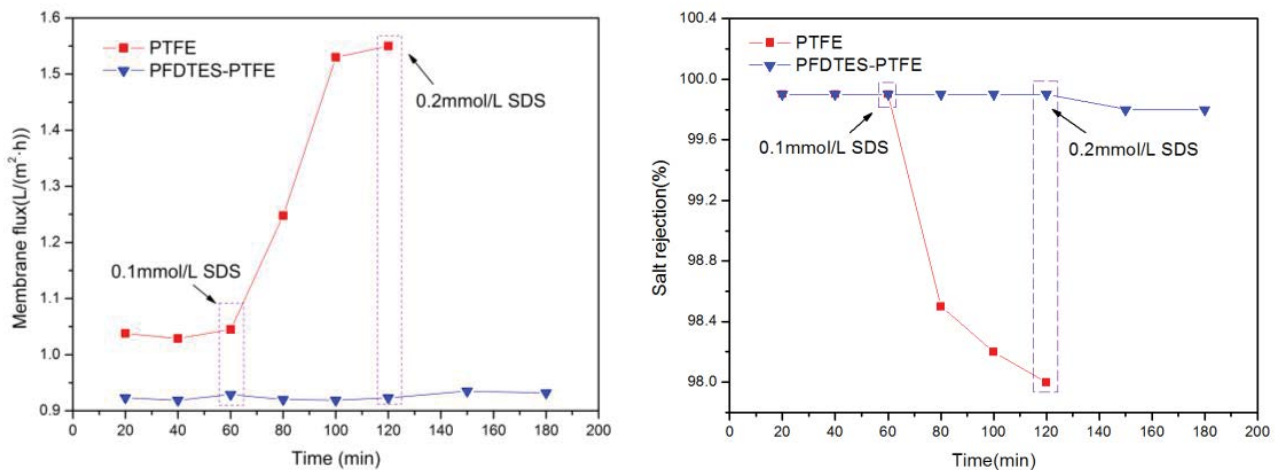


Fig. 6. The anti-wetting performances of the original membrane and composite membrane.

improved the surface hydrophobicity. A series of characterizations were performed on the membrane, such as SEM, AFM, WCA, and LEP tests. The AFM, WCA, and LEP tests showed changes in roughness and hydrophobicity of the PFDTES-PTFE membrane. Furthermore, the WCA of the

composite membrane was increased from 131.3° to 137.5°, which was caused by the lower surface energy of the PFDTES. There was also an increase in the hydrophobicity. Correspondingly, the LEP of the original membrane and of the hydrophobic membrane became 0.36 and 0.48 MPa,

respectively. However, since there was a decrease in porosity and meaning pore size, the flux of the PFDTES-PTFE membrane was also slightly reduced. However, the membrane flux of the PFDTES-PTFE membrane was 0.932 L/(m<sup>2</sup>·h) with a salt rejection of 99.8% after 3 h in a vacuum membrane distillation. Thus, the results have shown that the PFDTES-PTFE membrane has an excellent hydrophobicity and a stable service life, which can provide a guideline for further studies of the membrane distillation technology of applications concerning far-sea reeves.

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