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Hydrophobic modification conditions of Al_2O_3 ceramic membrane and application in seawater desalination

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Abstract. 1*H*,1*H*,2*H*,2*H*-perfluorodecytriethoxysilane ($C_{16}H_{19}F_{17}O_3Si$) be successfully applied to the hydrophobic modification of Al_2O_3 tubular ceramic membrane. Taking the concentration of modification solution, modification time, and modification temperature as factors, orthogonal experiments were designed to study the hydrophobicity of the composite membranes. The experiments showed that the modification time had the greatest impact on the experimental results, followed by the modification temperature, and the modification solution concentration had the smallest impact. Concentration of the modified solution 0.012 $mol \cdot L^{-1}$, modification temperature 30 ℃ and modification time 24 h were considered optimal hydrophobic modification conditions. And the pure water flux reached 274.80 $kg \cdot m^{-2} \cdot h^{-1}$ at 0.1MPa before hydrophobic modification, whereas the modified membrane completely blocked liquid water permeation at pressures less than 0.1MPa. Air gap membrane distillation experiments were conducted for NaCl (2wt%) solution, and the maximum flux reached 4.20 $kg \cdot m^{-2} \cdot h^{-1}$, while the retention rate remained above 99.8%. Given the scarcity of freshwater resources in coastal areas, the article proposed a system for seawater desalination using air conditioning waste heat, and conducted preliminary research on its freshwater production performance using Aspen Plus. Finally, the proposed system achieved a freshwater production capacity of 0.61 $kg \cdot m^{-2} \cdot h^{-1}$.
Keywords: Al_2O_3; ceramic membrane; hydrophobic modification; membrane distillation; seawater desalination

1. Introduction

Approximately 4 billion people in the world live in areas where freshwater security may be threatened. And climate change, population growth, and human activities will further exacerbate this situation. Therefore, one of the main environmental issues of the 21st century may be the sustainable management of global water resources (Padaki *et al.* 2015). Salt water resources represented by seawater account for nearly 97.5% of the total global water resources. Moreover, there are data shows that over 70% of the world's population lives within a range of 70 kilometers from the seaside. Therefore, seawater desalination is considered the most practical method for providing a sustainable source of freshwater. At present, the research of seawater desalination mainly involves multi-stage flash evaporation (MSF), multiple effect distillation (MED), reverse osmosis (RO), forward osmosis (FO) and membrane distillation (MD). Among them, RO, MSF, and MED are currently the main commercial technologies, while other methods are mostly in the laboratory or pilot testing stage, with almost no commercial engineering applications (Saleh *et al.* 2019, Lau *et al.* 2014, Bundschuh *et al.* 2021, Mabrouk and Fath 2015).

MD is a membrane separation process driven by thermal gradients, which allows water vapor to pass through hydrophobic micro-porous membranes and condense on the other side of the membrane at very low operating pressures and temperatures, achieving brine separation (Ali *et al.* 2018, Lin *et al.* 2023, Sandid *et al.* 2019) The advantages of MD lie in the small impact of raw water properties on water flux and water quality. Research reports had pointed out that when the salinity of raw water reaches around 100 g/L, the salt content of MD produced water can be controlled within 10 mg/L (Xu *et al.* 2016). At the same time, compared with pressure driven processes, the risk of membrane pollution is lower, so it has broad development prospects in the field of water treatment. At present, the high cost, low membrane flux, and unstable operation of MD are the main factors restricting its large-scale application.

In order to achieve high retention and flux, the membrane used in MD process needs to have two basic characteristics: hydrophobicity and porosity. At present, hydrophobic membranes commonly used in MD process are made of high molecular polymers, such as polytetrafluoroethylene (PTFE), polyvinylidene fluoride (PVDF), polypropylene (PP), etc. (Wang and Chung 2015, Yadav *et al.* 2022, Gao *et al.* 2018). But they are inferior to inorganic ceramic membrane in terms of high temperature resistance, chemical corrosion resistance, high mechanical strength, antibacterial, service life, etc (Varela-Corredor and Bandini 2020). Therefore, it is of positive significance to study the MD technology of ceramic membrane in the harsh physical and chemical environment of seawater desalination. In general, there are a large number of hydrophilic groups on the surface of ceramic particles, and water can easily pass through. Therefore, the surface function of ceramic membranes used for seawater desalination must be changed.

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How to construct high-precision functional coatings and engineering materials has always been a theme of concern for many authors. For instance, Lee *et al*. (2007) utilized dopamine self polymerization to form a surface adhered polydopamine film on the surface of ceramics through simple dip-coating. Geng *et al*. (2022) focused on the preparation of functional materials by forming metal-organic complexes. Yang *et al*. (2020, 2021) studied the preparation of high-performance separation membranes by atomic layer deposition for water treatment, which provides a reference for the preparation of functional materials. Wieszczycka *et al*. (2021) discussed the latest trends in the field of surface functionalization of nano-materials, which are formed by various structures such as polymers, silica, inorganic nanoparticles, and hybrids.

Fluorosilane is a new type of organic silane, which is an organic combination of fluorine containing organic compounds and silicon containing organic compounds. Due to the weak interaction between F-C chains, fluorine elements aggregate on the surface of the material, resulting in a rapid decrease in surface energy and high hydrophobicity. At present, the preparation of hydrophobic composite membrane by grafting fluorinated hydrophobic molecular chains in fluorosilane onto hydrophilic ceramic membrane has attracted the attention of scholars all over the world. Picard *et al.* (2001) grafted $C_6F_{13}C_2H_4Si(OMe)_4$ and $C_8F_{17}C_2H_4Si(OEt)_3$ as hydrophobic modifiers onto the surface of ZrO_2-$KTiOPO_4$ composite ceramic membrane, and the hydrophobic angle of ceramic membrane surface reached 145° after modification. Gazagnes *et al.* (2007) used 1*H*,1*H*,2*H*,2*H*-perfluorodecyltriethoxysilane to chemically modify ceramic membrane (ZrO_2, Al_2O_3 and Al_2SiO_5) with different properties and pore diameters of 50 nm, 200 nm, 400 nm and 800 nm respectively. The hydrophobic ZrO_2 membrane (50nm) had the highest retention rate and flux (>95%) for NaCl solution in air gap membrane distillation process. Tubular and planar TiO_2 ceramic membrane were modified by grafting perfluoroalkylsilane (C_6&C_{12}). The results show that the proposed grafting method effectively modified the hydrophilic surface of TiO_2 ceramic membrane. The contact angles of C_6 and C_{12} grafted planar membranes were 130° and 140°, respectively (Kujawa *et al.* 2013). Fluorosilane with different chain lengths (C_6~C_{12}) were used as modifier to hydrophobic modify ceramic membrane such as TiO_2, Al_2O_3, ZrO_2 (Kujawa *et al.* 2017, Kujawa *et al.* 2020).

It is not difficult to find that ceramic membrane such as Al_2O_3, ZrO_2 and TiO_2 have been used for hydrophobic modification in MD process, but there are few systematic studies on hydrophobic modification conditions of ceramic membrane. In this paper, tubular Al_2O_3 ceramic membrane was selected as the base membrane, 1*H*,1*H*,2*H*,2*H*-perfluorodecytriethoxysilane was used as the modifier, and the concentration of the modifier, the modification time and the modification temperature were taken as factors. Three factors and three levels orthogonal experiments were designed to systematically explore the hydrophobic modification conditions of Al_2O_3 ceramic membrane. Considering the small freshwater production of MD technology, which is mainly limited by the current characteristics of MD materials, it is difficult to achieve a fundamental breakthrough in the short term. This article proposes to use the waste heat generated by lithium bromide absorption refrigeration air conditioning equipped in island factories or large island tourist hotels for seawater desalination. The soft of Aspen Plus was used to calculate the proposed system and study its water production capacity.

2. Experimental procedures

2.1 Materials

Commercially available Al_2O_3 tubular ceramic membrane (ϕ 12×2 mm, length 150 mm, average pore diameter 50 nm, Nanjing Jiuwu High Tech Co., Ltd, China), 1*H*,1*H*,2*H*,2*H*- perfluorodecytriethoxysilane ($C_{16}H_{19}F_{17}O_3Si$) (modifier), concentrated hydrochloric acid, ethanol, deionized water, isopropanol and sodium chloride. The chemicals listed above are analytically pure.

2.2 Instruments

The main instruments and equipment include membrane reactor (self-made), membrane distillation module (self-made), electrothermal constant temperature drying oven (202-00), ultrasonic cleaner (KH5200E), precision electronic balance (FA1204B), peristaltic pump (BT-100), flowmeter (LZB-10), conductivity meter (DDS-307), infrared analyzer (IRPrestige-21), Optical contact angle measuring instrument (OCAH200), the field emission scanning electron microscopy (TM 3030 plus).

2.3 Methods

Al_2O_3 ceramic membrane was pretreated with dilute hydrochloric acid (5wt%), and then the required concentration of the modifier-isopropanol solution was prepared in a dry environment for standby. Placed the pretreated membrane tube (wrapped with tape on the outer surface) in the membrane reactor, poured in the prepared modified solution, reacted at the set temperature for a certain time, and then removed it. Cleaned it twice with ethanol water solution ultrasonic for 15 minutes each time, and dried it in a drying oven to obtain Al_2O_3 tubular composite film with hydrophobic surface. The specific process is shown in Fig. 1.

2.4 Performance testing

Firstly, pure water was used as the experimental material to test the flux of the Al_2O_3 composite film. Then, an optical contact angle measuring instrument was used to analyze the hydrophobic properties of the inner surface of the Al_2O_3 composite film prepared. In addition, to evaluate the desalination capacity of composite membranes, membrane distillation experiments were conducted using NaCl solution as the experimental medium. The experimental setup is shown in Fig. 2.

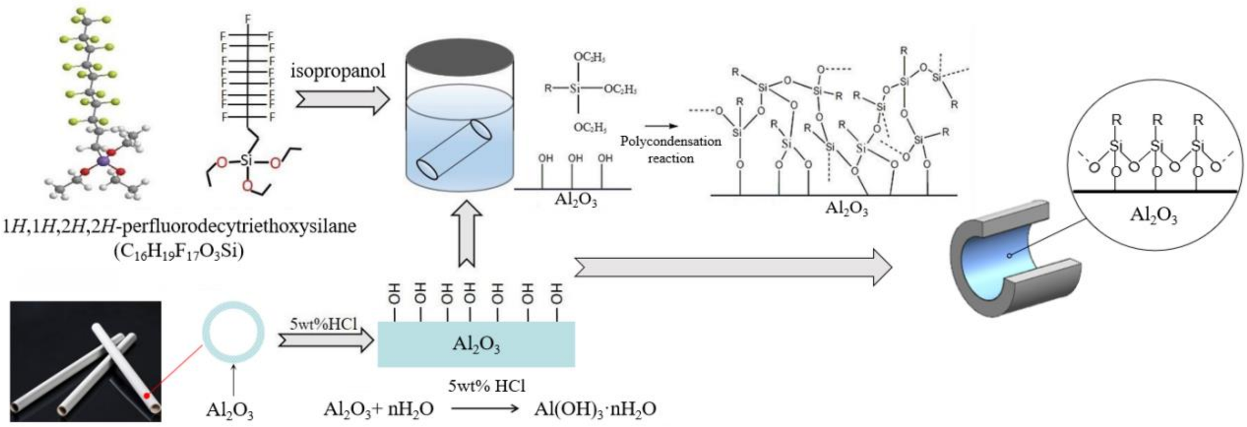

Fig. 1 Hydrophobic modification process of Al_2O_3 ceramic membrane

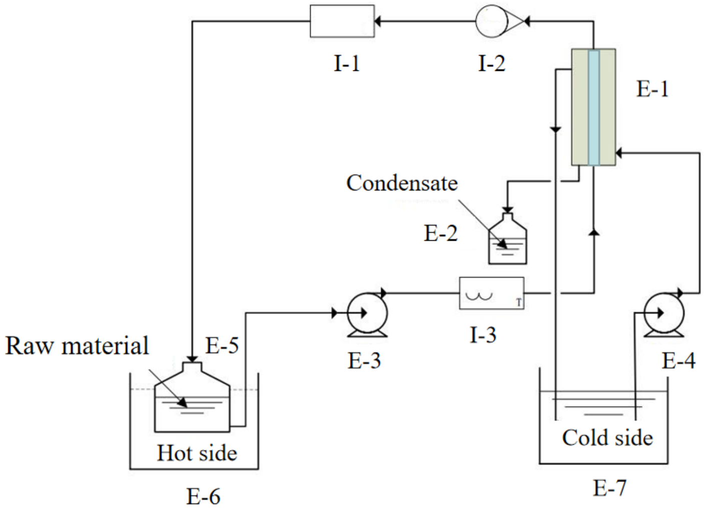

Fig. 2 Schematic diagram of experimental device: Air gap membrane distillation module (E-1); Liquid collection bottle (E-2); Peristaltic pump (E-3&E-4); Raw material bottle (E-5); Constant temperature bath (E-6); Cooling water tank (E-7); Pressure gauge (I-1); Glass rotor flowmeter (I-2); thermometer (I-3)

2.5 Seawater desalination system

At present, the application of MD technology in seawater desalination is still in the demonstration project stage, mainly due to the small freshwater production obtained by MD technology. Although MD technology has low requirements for operating conditions (temperature, pressure, etc.) and low energy consumption, considering the overall freshwater production, the cost-effectiveness is still not high enough.

Given that there are many places in coastal areas that require refrigeration, such as pirate factories, large hotels, etc, this paper proposes a system for seawater desalination using air conditioning waste heat (Fig. 3). The utility model comprises a Lithium bromide absorption refrigerator waste heat utilization system and a seawater desalination system connected with the system. The Lithium bromide Absorption refrigerator waste heat utilization system comprises a generator, a condenser, an evaporator, an absorber, a solution heat exchanger, a solution pump and a throttle valve. The seawater desalination system includes a seawater source, seawater pump, air gap membrane distillation component, heat exchanger, and freshwater collection device.The seawater desalination system consists of the following cyclic processes. The cold side outlet of the air gap membrane distillation component is connected to the cooling liquid inlet of the absorber, the cooling liquid outlet of the absorber is connected to the cooling liquid inlet of the condenser, and the cooling liquid outlet of the condenser is connected to the hot side inlet of the air gap membrane distillation component. After heat exchange between low-temperature seawater and high temperature seawater in the heat exchanger through the seawater pump, they pass through the cold side of the air gap membrane distillation module, then enter the absorber of the Lithium bromide Absorption refrigerator system, take away the heat released in the absorption process, arrive at the condenser, and then enter the hot side of the air gap membrane distillation module after further heating. The seawater desalination process is completed by concentrating in the air gap membrane distillation component and returning to the seawater source through a heat exchanger.

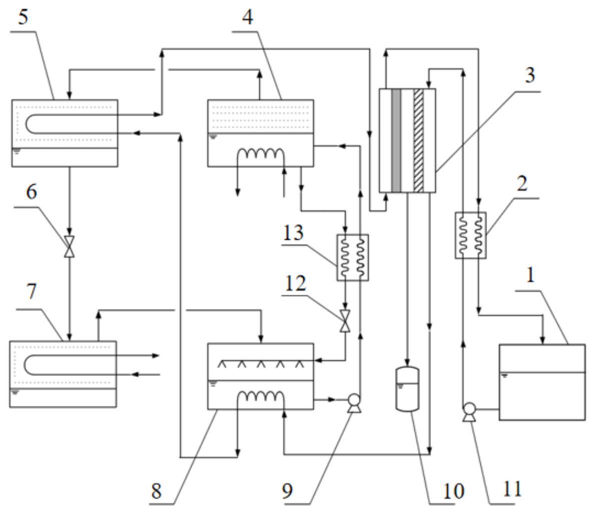

Fig. 3 System for seawater desalination by air conditioning waste heat: Raw material solution (1); Heat exchanger (2&13); Air gap membrane distillation module (3); Generator (4); Condenser (5), Throttle valve (6&12); Evaporator (7); Absorber (8); Solution pump (9); Fresh water collection device (10); Seawater pump (11)

Table 1 Factor level table

Level	Factor		
	Concentration of the modified solution/mol·L^{-1}	Modification temperature/℃	Modification time/h
1	0.008	30	24
2	0.010	40	48
3	0.012	50	72

3. Results and discussion

3.1 Pure water flux

The related studies show that the main factors affecting the hydrophobic modification of ceramic membrane are the concentration of the modified solution, the modification temperature and the modification time (Subramanian *et al.* 2017, Chen *et al.* 2018, Kujawa *et al.* 2020). Therefore, this paper designed a three-factor and three-level orthogonal experiment, and the factor level table is shown in Table 1. Nine experimental schemes were obtained from Table 1, and flux testing was conducted using pure water at room temperature. The results are shown in Table 2 and Fig.4. Obviously, the difference in pure water flux under different conditions reached 11.64 kg·m^{-2}·h^{-1}, indicating the necessity of studying hydrophobic modification conditions. The range number $R_3 > R_2 > R_1$, so the modification time had the greatest impact on the experimental results, followed by the modification temperature, and the modification solution concentration had the smallest impact. In addition, the corresponding $k_3 < k_2 < k_1$ below the concentration of the modified solution indicates that a concentration of 0.012 mol·L^{-1} was better. Similarly, a modification temperature of 30 ℃ and a modification time of 24 h were better. However, the optimal group was not included in the nine experiments.

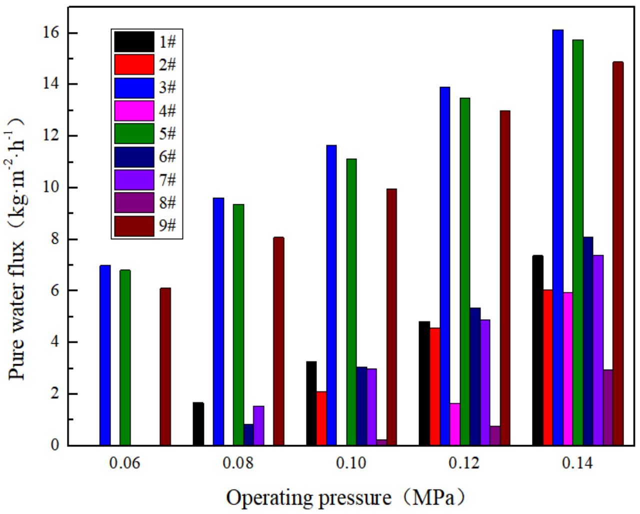

Fig. 4 Pure water flux of each group under different operating pressures

The pure water flux of the prepared Al_2O_3 composite film 10# (Optimal group) were observed (Fig. 5). The pure water flux of unmodified membrane reached 274.80 kg·m^{-2}·h^{-1} at 0.1MPa, whereas the modified membrane completely blocked liquid water permeation at pressures less than 0.1MPa. Clearly, significant changes in pure water flux before and after hydrophobic modification, indicates that hydrophobic modification was very successful.

3.2 Characterization

The FT-IR of the inner surface of the membrane before and after hydrophobic modification were shown in Fig. 6 (10#). Apparently a new C-F bond stretching peak appeared at 1155 cm^{-1} and 1180 cm^{-1}. The bands around 1455 cm^{-1} were related to C-H asymmetric deformation and -CH_2- scissors vibration. The absorption bands at at 1315 cm^{-1} were assigned to asymmetric Si-O stretching vibration originating. The absorption peak near 2960 cm^{-1} was considered to be the vibrational peak of -CH_2-. These evidences indicate that the modifier had been successfully grafted onto the surface of Al_2O_3 ceramic membrane (Jeong *et al.* 2001, Monde *et al.* 1999).

According to Fig. 7, the particle of Al_2O_3 ceramic membrane after modification became smoother than before, possibly due to its surface had been covered with a layer of organic matter, which reduced the pore size of the modified membrane.

Taking sample 10 # as an example, performed water contact angle testing on the membrane tube, and the results were shown in Fig. 8. The water contact angle showed an obtuse angle (~137°) after modification, while for the unmodified ceramic membrane, this value was only about 52°. This indicates that the hydrophobic modification of the 10 # membrane tube had been completed.

3.3 Membrane distillation experiments

The desalination performance and anti fouling ability of the membrane are important parameters for the membrane.

Table 2 Orthogonal test plan and results: Operating pressure 0.10 MPa

Factor / No.	Concentration of the modified solution/mol·L^{-1}	Modification temperature/°C	Modification time/h	Pure water flux/kg·m^{-2}·h^{-1}
1#	0.008	30	24	3.28
2#	0.008	40	48	2.11
3#	0.008	50	72	11.64
4#	0.010	30	48	0.00
5#	0.010	40	72	11.11
6#	0.010	50	24	3.07
7#	0.012	30	72	2.98
8#	0.012	40	24	0.24
9#	0.012	50	48	9.97
K_1	17.03	6.26	6.59	
K_2	14.18	13.46	12.08	
K_3	13.19	24.68	25.73	
k_1	5.68	2.09	2.20	—
k_2	4.73	4.49	4.03	
k_3	4.40	8.23	8.58	
R	1.28	6.14	6.38	

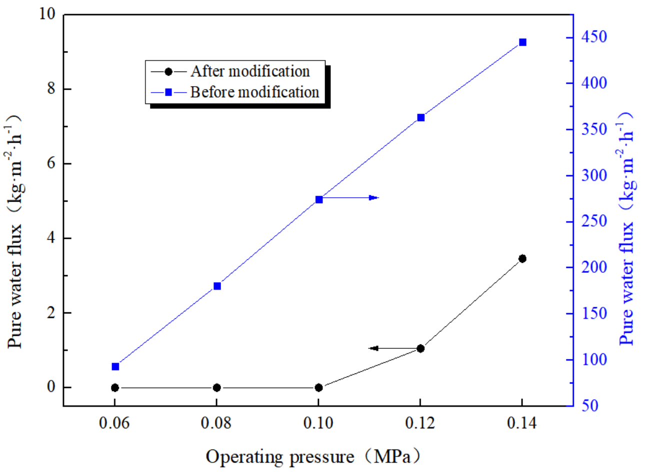

Fig. 5 Pure water flux of the optimal group under different operating pressures

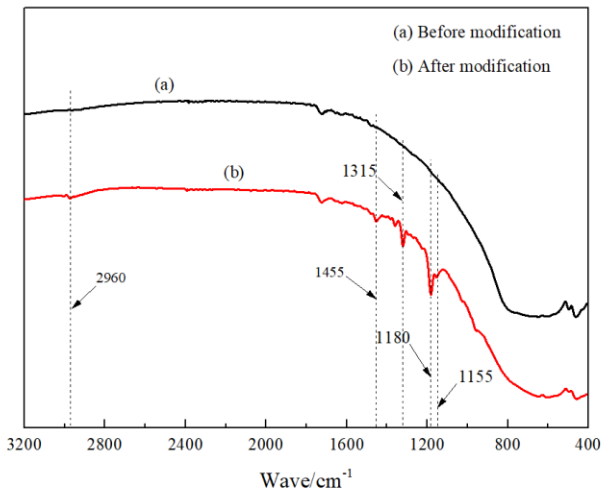

Fig. 6 FT-IR of the membrane before and after modification

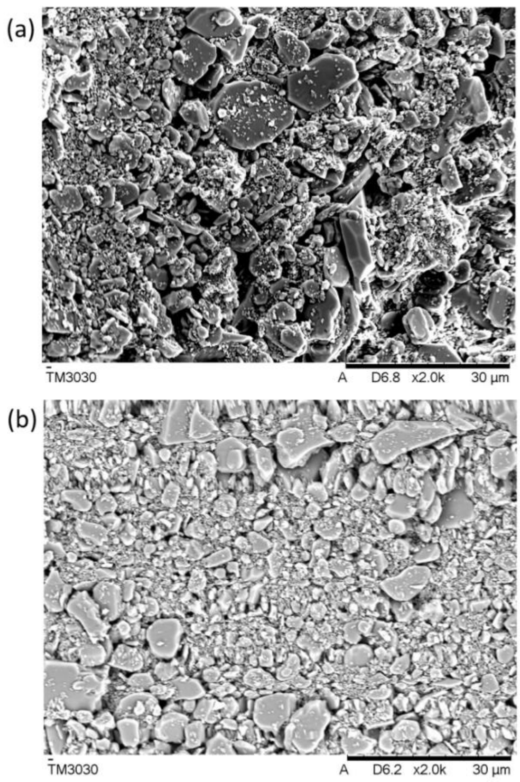

Fig. 7 SEM image of the membrane surface: (a) before modification and (b) after modification

The membrane distillation experiments were conducted using NaCl (2wt%) solution as the experimental material, and the results were shown in Figs. 9-10 (10#). The results show that the membrane distillation flux increased exponentially with the increased of temperature, up to 4.20 kg·m^{-2}·h^{-1} at 75 ℃, and the retention rate remained

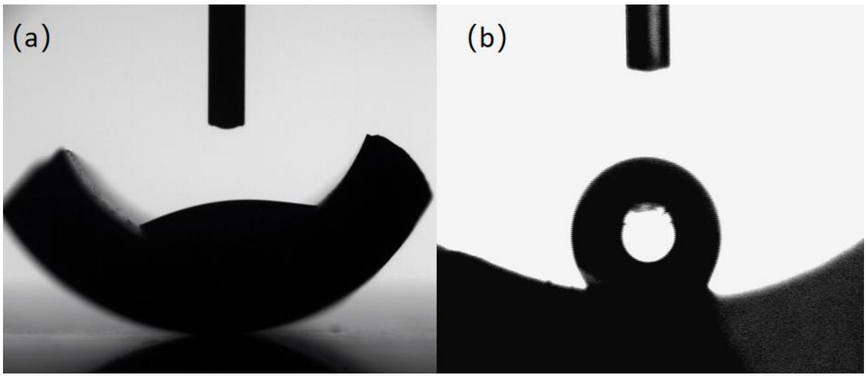

Fig. 8 The surface water contact angle of the membrane: (a) before modification. and (b) after modification

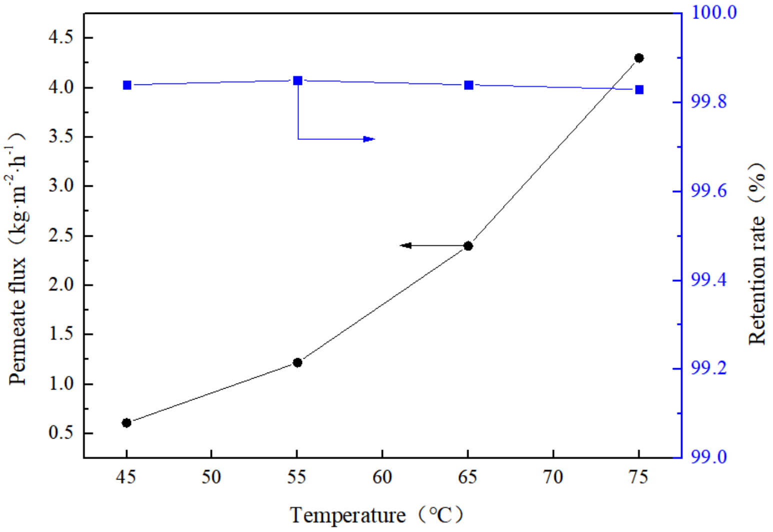

Fig. 9 Membrane distillation flux at different temperatures: Cold side: 15 ℃&50 L/h; Hot side: 25 L/h; Operating pressure 0.10 MPa

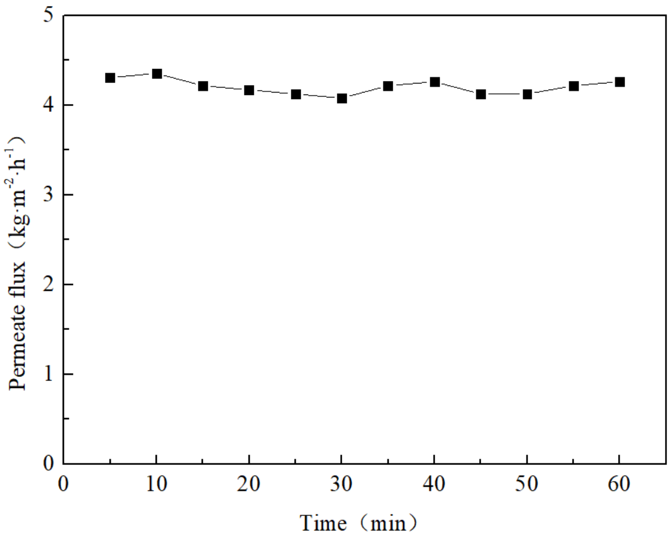

Fig. 10 Changes in membrane distillation flux over time: Cold side: 15 ℃&50 L/h; Hot side: 75 ℃&25 L/h; Operating pressure 0.10 MPa

consistently above 99.8% in the process. The membrane distillation flux was measured multiple times within 60 minutes and it was found to maintain around 4.20 $kg \cdot m^{-2} \cdot h^{-1}$, demonstrating good membrane fouling resistance.

Table 3 Comparison of pure water permeability

Materials of membrane	Pure water permeability /kg·m^{-2}·h^{-1}·MPa^{-1}	Liquid entry pressure/MPa	Membrane shape	References
Yttria-stabilizedzirconia	$1.4 \pm 0.1 \times 10^3$	0.22	Planar	Liu *et al.* (2017)
Porous alumina	1.25×10^3	~ 0.15	Hollow fiber	Fang *et al.* (2012)
α-Al_2O_3	$5.75 \pm 0.78 \times 10^3$	＞0.05	Planar	Miao *et al.* (2022)
Al_2O_3	4.40×10^3	~ 0.10	Tubular	This paper

Table 4 Comparison of ceramic membranes performance in AGMD process

Materials of membrane	Permeate flux/kg·m^{-2}·h^{-1}	Retention rate (%)	Feed temperature /℃	Membrane shape	Other conditions	References
ZrO_2	2.71	99.8	75	Tubular	0.5mol/L NaCl; Cold side temperature 5 ℃	Cerneaux *et al.* (2009)
	3.50	99.5	85			
	4.70	99.8	95			
Tunisian clay	3.40	98.5	75	Tubular	Seawater；Cold side temperature 5 ℃	Khemakhem and Ben Amar (2011)
	4.17	98.6	85			
	7.10	99	95			
TiO_2	0.56	92.3	70	Tubular	0.5 mol/L NaCl；Cold side temperature 5 ℃	Kujawa *et al.* (2013)
	1.92	99.5	80			
	2.41	99.8	90			
Al_2O_3	3.70	99.0~99.5	90	Tubular	0.25wt% NaCl; Cold side temperature 5 ℃	Kujawa *et al.* (2017)
TiO_2	1.90	99.5~99.6				
Al_2O_3	32.2	> 99.99	80	Hollow fiber	1.2 wt% NaCl; Cold side temperature 20 ℃	Garcia-Fernandez *et al.* (2017)
ZrO_2	3.15	~99	90	Tubular	0.5 mol/L NaCl; Cold side temperature 5 ℃	Kujawa *et al.* (2020)
Al_2O_3	0.61	99.87	45℃	Tubular	2wt% NaCl；Cold side temperature 15 ℃	This paper
	1.22	99.85	55℃			
	2.40	99.84	65℃			
	4.20	99.83	75℃			

3.4 Performance comparison

For the permeability of pure water, some related research results were compared together, as shown in Table 3. Related research mostly focused on planar membranes, while research on tube membranes was relatively rare. Compared with relevant literature data, the tubular membrane used in this article had good water permeability and higher liquid entry pressure after modification, which demonstrated excellent separation performance form the memebrane.

Table 4 lists some research results of air gap membrane distillation (AGMD) experiments by modified ceramic membrane in recent years. Compared with the literature data, the performance of the tubular membrane had been improved in this paper. For example, Cerneaux *et al.* (2009) tested the performance of ZrO_2 membranes, with a maximum flux of 4.70 kg·m^{-2}·h^{-1}. However, the temperature difference on both sides of the membrane was about 50% higher than in our case. Kujawa *et al.* (2017) used an Al_2O_3 membranes to desalinate NaCl (0.25wt%) in AGMD at the temperature difference of 90 ℃ on both sides of the membrane. The reported value of permeation flux was approximately 3.70 kg·m^{-2}·h^{-1}, and the retention rate during this process was 99%~99.5%. It is worth mentioning that the maximum flux in this article reached 4.20 kg·m^{-2}·h^{-1} when the temperature difference was 60 ℃, and the retention rate remained consistently above 99.8% in the process.

3.5 Performance of seawater desalination system

Taking a single effect Lithium bromide absorption refrigerator system as the research object, its capacity for seawater desalination in the system shown in Fig. 3 was investigated. The parameters of the refrigeration system are shown in Table 5. Aspen Plus has a complete physical property database, users can build models according to their needs and simulate the process. Therefore, Aspen Plus was used to simulate the system to evaluate the performance in this paper. According to Fig. 3, the heat load of the absorber and condenser were used to heat the raw material used in the AGMD process in Aspen Plus. After calculation, the refrigeration system could heat the NaCl (2wt%) to about 45 ℃. Based on Fig. 8, the proposed seawater desalination system can produce 0.61 kg·m^{-2}·h^{-1} of fresh water. Although the water production of the system is not very high, considering its lower cost, this study still has positive significance for coastal water shortage areas.

Table 5 Parameters about the refrigeration system

Parameter	Value
Refrigeration capacity	1744.5 kW
Inlet temperature of refrigerant water	15℃
Outlet temperature of refrigerant water	4 ℃
Cooling water inlet temperature	32 ℃
Absorber thermal load	~2318 kW
Condenser heat load	~1874 .1kW

4. Conclusions

1*H*,1*H*,2*H*,2*H*-perfluorodecytriethoxysilane be successfully applied to the hydrophobic modification of Al_2O_3 tubular ceramic membrane. Orthogonal experiments were designed to test the membrane permeability by pure water, and it was found that modification time had the greatest impact on permeability, followed by modification temperature, and the effect of modifier concentration was the smallest. Concentration of the modified solution 0.012 $mol \cdot L^{-1}$, modification temperature 30 ℃ and modification time 24 h were considered optimal conditions. The membrane penetration pressure from the optimum design was greater than 0.10MPa. The membrane distillation performance were tested by NaCl (2wt%) solution for the optimal group. It was found that with the increase of feed temperature, the permeation flux increased rapidly, up to 4.20 $kg \cdot m^{-2} \cdot h^{-1}$ at 75 ℃, the rejection rate of NaCl by the membrane remained above 99.8% in this process. The membrane distillation flux was measured multiple times within 60 minutes and it was found to maintain around 4.20 $kg \cdot m^{-2} \cdot h^{-1}$, demonstrating good membrane fouling resistance. The modified membrane was used in the seawater desalination system and calculated in Aspen Plus. It was found that the solution feed temperature could be heated to about 45 ℃, and the system achieved a water yield of 0.61 $kg \cdot m^{-2} \cdot h^{-1}$, while the refrigeration system was operating normally. Under lower driving force, the prepared membrane achieved higher permeation flux and retention rate. Although the water production of the refrigeration system is not high, considering its low cost, this plan is still feasible.

Acknowledgments

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