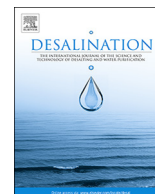




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Performance of ceramic membrane in vacuum membrane distillation and in vacuum membrane crystallization

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ABSTRACT

Membrane crystallization (MCR) is emerging as an interesting candidate to extract additional freshwater and raw materials from high-concentrated solutions. Traditionally, MCR has been carried out by using polymeric membranes that have limited chemical and mechanical stability. These shortcomings can be overcome by using ceramic membranes. The current study describes the preparation and testing of two hydrophobic ceramic membranes synthesized through sol-gel process, and combined phase-inversion and sintering method. The first membrane (CM-L) was synthesized by coating hydrophobic polymethylsilsesquioxane aerogels on alumina membrane supports via a sol-gel process. The membrane showed stable hydrophobic character in membrane distillation and crystallization tests but very low flux. To obtain high flux, a second type (CM-S) membrane was prepared by applying Fluoroalkylsilanes (FAS) (1H, 1H, 2H, 2H-perfluorooctyltriethoxysilane) hydrophobic agent at the relatively thin and more porous as-sintered alumina hollow fibers. The suitability of both membranes for MCR process was analyzed by crystallizing NaCl and LiCl. By using 1 M NaCl and 13 M LiCl aqueous solutions, and under the same operative conditions, CM-S membrane exhibited average flux higher than CM-L membrane. The performance of both the membranes, in terms of hydrophobic character, remained stable throughout the performed tests.

1. Introduction

Membrane Distillation (MD) and its extension Membrane Crystallization (MCR) are relatively less-explored membrane processes with potential to recover freshwater and minerals from highly concentrated liquid streams. The partial pressure gradient induced across a microporous hydrophobic membrane acts as the driving force for MD/MCR. The hydrophobic nature of the membrane allows the passage of vapors only and stops the liquid intrusion into the membrane pores. Some of the main advantages of MD are:

- (i) lower operating temperatures with respect to those usually used in conventional distillation column thus allowing the utilization of low-grade heat streams such as alternative energy sources (solar, wind, or geothermal),
- (ii) theoretically 100% rejection of all non-volatiles and

- (iii) the possibility to reach higher water recovery with respect to RO and conventional thermal process thus reducing the amount of brine discharged into the environment [1,2]. The latter advantage is exploited in MCR to achieve solution saturation and thus crystallization.

Principally, MCR can be operated in all four well-known configurations of MD including direct contact, vacuum, sweep gas and air gap MD. In fact, direct contact membrane distillation (DCMD) is the simplest to operate and the best suited for applications such as desalination or the concentration of aqueous solutions in which water is the major permeate component. On the other hand, one of the main benefits of vacuum membrane distillation (VMD), relative to the other MD configurations, is that conductive heat loss through the membrane is lower which helps in maintaining the high vapor pressure gradient and, therefore, high mass flux across the membrane. A recent study [3] has

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Table 1
MD performance for ceramic membranes published recently.

Membrane material	Membrane morphology	Hydrophobize technique	Configuration	NaCl feed solution	Feed temp (°C)	Permeate side	MD permeate flux (L m ⁻² h ⁻¹)	Ref
Titania	Tubular	C12-FAS	DCMD	0.8 M	90	5 °C pure water	2.5	[20]
Al ₂ O ₃	disk	C8-FAS	DCMD	0.5 M	53	18 °C water	9.0	[17]
Al ₂ O ₃	disk	C6-FAS	DCMD	4.0 wt%	80	20 °C distilled water	17.0	[21]
Al ₂ O ₃ /ZrO ₂	Tubular	C6-FAS	DCMD	1.0 M	95	5 °C water	6.9	[22]
Si ₃ N ₄	Hollow fiber	C6-FAS	DCMD	4.0 wt%	80	20 °C water	10.4	[23]
β-Sialon	Hollow fiber	C6-FAS	DCMD	4.0 wt%	80	20 °C water	6.7	[24]
ZrO ₂ /Ti	Tubular	C8-FAS	DCMD	0.5 M	95	5 °C water	3.8	[25]
TiO ₂	Nanofiber	C6-FAS	DCMD	3.5 wt%	80	20 °C pure water	11.9	[26]
ZrO ₂ /Ti	Tubular	C6-FAS	VMD	0.5 M	40	Vacuum 0.02 bar	10.8	[24]
Si ₃ N ₄	Hollow fiber	C6-FAS	VMD	4.0 wt%	80	Vacuum 0.02 bar	27.5	[23]
Al ₂ O ₃	disk	C6-FAS	VMD	4.0 wt%	80	Vacuum 0.02 bar	10.4	[24]
Al ₂ O ₃	Hollow fiber	C6-FAS	VMD	4.0 wt%	80	Vacuum 0.04 bar	42.9	[15]
Zeolite/Al ₂ O ₃	Tubular	MFI zeolite	VMD	3.5 wt%	60	Vacuum 0.0045 bar	12.0	[27]
Si ₃ N ₄	Hollow fiber	C6-FAS	VMD	4.0 wt%	70	Vacuum 0.02 bar	22.2	[28]
Al ₂ O ₃	disk	C8-FAS	VMD	3.5 wt%	70	Vacuum 0.03 bar	37.1	[29]
Al ₂ O ₃	Hollow fiber	C6-FAS	VMD	3.5 wt%	70	Vacuum 0.03 bar	60.0	[30]
ZrO ₂ /Al ₂ O ₃	Tubular	C8-FAS	AGMD	1.0 M	95	10 mm air gap/stain less surface	6.9	[12]
Al ₂ O ₃ /ZrO ₂	Tubular	C12-FAS	AGMD	1.0 M	95	Air gap/5 °C cold surface	5.0	[11]
ZrO ₂	Tubular	C8-FAS	AGMD	0.9 wt%	70	10 mm air gap/cold surface	7.0	[37]
Titania	Tubular	C12-FAS	AGMD	0.8 M	90	Air gap/5 °C cold	2.8	[20]
ZrO ₂ /Ti	Tubular	C8-FAS	AGMD	0.5 M	95	Air gap/5 °C cold	4.7	[25]
Al ₂ O ₃	Disk	C8-FAS	SGMD	4.0 wt%	90	N ₂ gas (337 L/h, 20 °C)	19.8	[38]
Si ₃ N ₄	Planar	SiNCO nano-particle	SGMD	4.0 wt%	75	N ₂ gas	6.7	[39]

shown that the direct contact configuration of MCr is not effective in crystallizing highly soluble salts from their solutions and has suggested the use of vacuum configuration for this purpose. However, the main drawback of vacuum configuration is that special care must be taken to prevent membrane wetting which occurs when the liquid penetrates into the membrane pores. Once the wetting occurs, the membrane must be completely dried and cleaned before the wetted pores can once again support a vapor-liquid interface [4].

Anti-wetting characteristic can be incorporated into the membrane through hydrophobicity of the membrane material and by tuning the membrane pore size. Additionally, membranes for MD/MCr applications should exhibit high overall porosity, sharp pore size distribution and low thermal conductivity. The membranes for MD should also possess excellent thermal, chemical and mechanical strength. Many attempts have been made to fabricate the membranes for MD with the required characteristics [5–7]. Polymeric materials have been used in most of the traditional efforts. However, the polymeric membranes have several drawbacks including the low thermal stability, low resistance to strong solvents such as acids, low mechanical strength and consequently relatively shorter lifetime. These issues can be addressed by using ceramic membranes as already being practiced in other similar applications such as microfiltration and ultrafiltration [8]. However, the ceramic membranes have high thermal conductivity and are inherently hydrophilic in nature and therefore, in principle, not suitable for MD applications.

For what concerns the high thermal conductivity of ceramic membranes, which will lead to low thermal efficiency in MD, the latter can be controlled and improved either reducing membrane thickness or increasing trans-membrane flux according to Al-Obaidani et al. [45]. Regarding the hydrophilic nature of the ceramic membranes, some attempts have been made to alter it. Leger et al. [9] have reported the hydrophobic surface modification of alumina membrane for pervaporation applications by using polydimethylsiloxane oil. Picard et al. [10] have reported the application of different fluorinated silanes to render hydrophobic character to various hydrophilic microfiltration and ultrafiltration membranes. Similar attempts were reported by

several other authors [11–15]. Dafinov et al. [16] used alcohol adsorption to modify the hydrophilic surface of commercial γ -alumina membrane. Effectiveness of different salines for hydrophobic surface modification of ceramic membranes has also been performed [17,18]. Lin et al. [19] coated hydrophobic polymethylsilsesquioxane (PMSQ) aerogels on alumina membrane supports via a sol-gel process. The hydrophobic PMSQ aerogel membranes were demonstrated to be both durable and reusable, which showed great promise for application in membrane contactors. A summary of state-of-the-art on fabricating hydrophobic ceramic membranes and their performance in MD applications has been provided in Table 1.

The current study describes the fabrication of hydrophobic PMSQ tubular aerogel membranes (CM-L) via a sol-gel process and hydrophobic alumina hollow fiber (CM-S) membranes via phase-inversion and sintering method for MCr applications. The PMSQ tubular aerogel membrane applied in this study is prepared by the same method as described elsewhere [19,31] but with different alumina substrate: tubular substrate rather than flat sheet which offers packing density. Thickness and overall porosity of CM-S have been tuned to achieve high flux compared to CM-L. The performance of both membranes has been analyzed in MD/MCr tests by using aqueous solutions of NaCl and LiCl.

2. Materials and methods

2.1. Membrane preparation

2.1.1. PMSQ tubular aerogel membranes: CM-L

PMSQ tubular aerogel membranes were prepared via a sol-gel process according to the various steps shown in Fig. 1. First, the PMSQ precursor, here methyltrimethoxysilane (MTMS) was chosen, ethanol (EtOH) and hydrochloric acid (HCl) were mixed with deionized water and stirred at 25 °C for 90 min. The molar ratio of MTMS, EtOH, HCl and water was 1:3:6.9 $\times 10^{-4}$:1. Subsequently, EtOH and aqueous ammonia (NH₄OH) were further added with the molar ratio of EtOH (amount of second adding), NH₄OH and water being 1:0.223:2.12. After stirring the solution at 25 °C for 30 min, a tubular ceramic

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