



# Morphological design of alumina hollow fiber membranes for desalination by air gap membrane distillation



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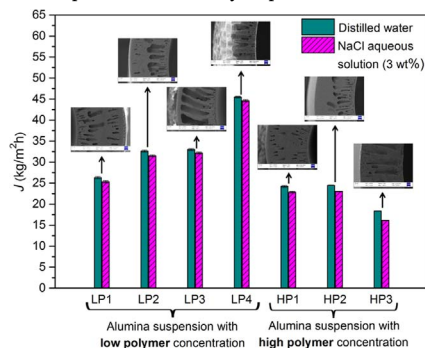
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## GRAPHICAL ABSTRACT

AGMD permeate flux of hydrophobic alumina hollow fiber membranes and the corresponding SEM images of their cross-section.



## ARTICLE INFO

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## ABSTRACT

Alumina hollow fiber membranes were prepared by the phase inversion technique under different spinning conditions in order to induce various types of structural morphologies. In the membrane fabrication process, the studied parameters were the polymer concentration in the inorganic suspension and its flow rate, the gap distance, the bore liquid, the outer coagulant composition and their flow rates. After sintering, the hollow fibers were chemically modified by grafting (1H,1H,2H,2H-perfluorodecyltriethoxysilane) rendering them hydrophobic for their use in membrane distillation (MD) process. The effects of the membrane morphology on the obtained MD membrane characteristics and on air gap membrane distillation (AGMD) desalination performance were studied in order to figure out the most promising structure for MD. The suitability of alumina hollow fibers for AGMD was confirmed by various membrane characterization techniques. In general, the membranes prepared with lower polymer concentration in the inorganic suspension exhibited higher AGMD performance (i.e. higher permeate flux with a smaller flux reduction factor and a good salt rejection factor). Among all prepared hollow fiber membranes prepared in this study, the one with the largest micro-channel structure exhibited the best AGMD performance, even better than all hydrophobic ceramic membranes used so far in desalination by AGMD and DCMD.

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## 1. Introduction

Water desalination by membrane distillation (MD) is an environmentally-friendly alternative able to tackle the global water scarcity issue in combination with other water treatment techniques [1–3]. MD process can be used to produce not only distilled water but also ultrapure water, and stands out for its capability to treat concentrated salt aqueous solutions [3–5]. Porous hydrophobic membranes are used in this non-isothermal process in which the driving force for the mass transfer is the transmembrane vapor pressure.

The majority of MD studies were performed with membranes made from hydrophobic polymers such as polyvinylidene fluoride (PVDF), polypropylene (PP) and polytetrafluoroethylene (PTFE) [5]. Ceramic membranes can endure harsh environments, opening up the possibility of treating a broader variety of feed solutions and operating conditions [6–10]. It is well known that the superior structural, thermal and chemical characteristics of these membranes, facilitate membrane cleaning process after fouling without reducing the membrane properties and maintain its lifetime as consequence reducing therefore the membrane's replacement cost [11]. In spite of the excellent thermochemical stability of ceramic membranes, these are seldom explored for MD applications, mainly due to their hydrophilic nature. In other words, ceramic membranes cannot be used directly in MD, but are rendered hydrophobic by surface modification using diverse agents and techniques as described elsewhere [12]. This is one of the main limitations of using ceramic membranes in MD applications.

Different inorganic membranes of metal oxides (i.e. alumina, zirconia and titania) and non-oxides (i.e. silicon nitride) were used in desalination by MD. Research studies with commercial ceramic membranes have been carried out analyzing different properties such as the pore diameter of the zirconia layer on the microporous alumina tubular support [13], the grafting efficiency in terms of hydrophobic stability of the tubular titania membrane with time [14] and the effect of modifying agent type on alumina anodisc™ membranes [15]. Other research studies have been devoted to the preparation of ceramic membranes with different inorganic suspension compositions in order to perform the most promising one in MD [8,16,17]. Das et al. [16] studied the effect of the clay/alumina suspension proportions on the characteristics of the capillary membranes. By means of rheological properties, Zhang et al. [8] and Wang et al. [17] analyzed first the stability of silicon nitride and  $\beta$ -Sialon suspensions, respectively. Then, other parameters such as the powder/polymer binder ratio of the silicon nitride hollow fiber membranes and the  $\beta$ -Sialon hollow fiber composition and sintering temperature were investigated looking for the suitable properties of an MD membrane.

Other researches using ceramic membranes focused on the study of the effects of the MD operation parameters (i.e. sodium chloride concentration of the feed aqueous solution, feed and permeate temperatures and flow rates) [13,18] and the different MD configurations (i.e. air gap membrane distillation (AGMD), direct contact membrane distillation (DCMD) and vacuum membrane distillation (VMD)) [8,17,19] on the desalination efficiency. In all cases, compared to the other MD configuration the VMD permeate flux was the highest. Zhang et al. [8] found a significant difference between VMD and DCMD permeate fluxes of the grafted silicon nitride hollow fiber membranes mainly due to the temperature polarization effect. Wang et al. [17] managed to reduce this difference by using  $\beta$ -Sialon ceramic membranes with a lower thermal conductivity, but VMD flux remained the highest. In VMD configuration, the mass transfer resistance is reduced due to the air removal from the membrane pores and the conductive heat loss through the membrane is the smallest compared to the other MD configurations. However, this configuration is not very attractive from an industrial point of view since external condensers are necessary to collect the distillate outside the membrane module, complicating in this way the system assembly and increasing the operational cost [4]. Cerneaux et al. [19] also compared the VMD and DCMD permeate fluxes of the zirconia

tubular membranes with those of AGMD configuration. In AGMD configuration, as a consequence of the high thermal conductivity of ceramic materials, the temperature at the permeate side of the membrane is higher than that registered when using polymeric membranes with lower thermal conductivity. However, owing to the low thermal conductivity of the air gap, the temperature of the condensing surface is kept low, leading to a higher effective temperature gradient than that observed in DCMD when using ceramic membranes and comparatively better water production rates are obtained in AGMD. In addition to the lower heat transfer by conduction through the membrane in AGMD than in DCMD [4], other advantage of AGMD is the less risk of membrane pore wetting [13]. Nevertheless, the ceramic membranes used so far in MD showed very low AGMD permeate fluxes [13,14,16,18,19].

It is worth quoting that very few MD studies have been performed in desalination by ceramic hollow fiber membranes. However, it is important to emphasize the competitive interest of this membrane configuration against tubular, capillary or flat-sheet due to its higher packing density in modules (i.e. up to 9000 m<sup>2</sup>/m<sup>3</sup>) [20,21]. Among the proposed ceramic membrane materials, alumina is the most commonly used one because of its chemical and mechanical stability as well as its wide availability and low cost.

The majority of ceramic membranes used so far in desalination by MD are not hollow fibers and their MD performance is usually lower than that of polymeric membranes [12,22]. Therefore, attempts have been made in this study to prepare ceramic hollow fiber membranes with different morphologies. The morphological characteristics of the membranes play a key role in MD performance [23–25]. However, the effect of the membrane morphology on MD performance has not been thoroughly studied yet for ceramic membranes. As such, this work is focused on the preparation of alumina hollow fiber membranes with different morphological designs in order to figure out the suitable structure inducing better AGMD performance. The study of the effects of the alumina hollow fiber membrane morphology on the MD membrane characteristics and on the AGMD performance may give important new insights into the structure performance correlation in the MD field. It will be shown through proper structural morphologies that ceramic hollow fiber membranes have the potential to obtain high permeation flux competitive in desalination when using AGMD process.

## 2. Experimental

### 2.1. Materials

Aluminium oxide powders (Al<sub>2</sub>O<sub>3</sub> Powder, Ultra-Pure Grade, 99.99%) with an average particle size of 0.5–1.0  $\mu$ m were purchased from Inframat® Advanced Materials™. The polymeric binder was polyethersulfone (PESf, Radel A-300, Solvay Advanced Polymers GmbH, Dusseldorf, Germany). The additive was Arlcel P135 (polyethylene glycol 30-dipolyhydroxystearate, Uniqema, Wilton, UK). The solvent N-Methyl-2-pyrrolidone (NMP, GPR RECTAPUR, VWR Chemicals), ethanol (VWR Chemicals) and deionized (DI) water were used as coagulants. The grafting solution consists of 1H,1H,2H,2H-Perfluorodecyltriethoxysilane (97%, Sigma-Aldrich) and methanol (HiPerSolv CHROMANORM, VWR Chemicals). POREFIL® (Porometer) and isopropyl alcohol (IPA, Sigma-Aldrich Chemical) were the wetting liquids used for the gas-liquid displacement test and the void volume fraction measurement, respectively. The feed salt aqueous solutions of AGMD experiments were composed by sodium chloride (NaCl), which was purchased from Scharlab.

### 2.2. Preparation of alumina hollow fiber membranes

First, the spinning suspensions were prepared by dissolving 0.42 wt % of the dispersant (Arlcel P135) in 35.75 wt% of solvent (NMP) and then, the 63.83 wt% of aluminium oxide powder was added to the mixture. In order to obtain a good inorganic dispersion, the mixture was ball milled (SFM-1 Desk Top Planetary Ball Miller, MTI Corporation)

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