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## Green silica-based ceramic hollow fiber membrane for seawater desalination via direct contact membrane distillation

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### ABSTRACT

In this work, green silica-based ceramic hollow fiber membranes (CHFMs) derived from rice husk ash waste were successfully prepared via phase inversion and sintering technique. Prior to the fabrication, rice husk waste was converted into amorphous and crystalline silica-based rice husk ash (ARHA and CRHA). The surface of CHFMs could be easily modified to become hydrophobic by grafting with a fluoroalkylsilane (FAS) agent. The CHFMs were characterized before and after grafting using scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), measurement of contact angle and liquid entry pressure of water (LEPw), and three-point bending test. The pore size and the porosity of the membrane surface were also evaluated using mercury intrusion porosimetry (MIP). Lotus-leaf structure formed at the surface of CHFMs by grafting, leading to a high contact angle of  $> 150^\circ$ . Direct contact membrane distillation (DCMD) performance of membrane after grafting was conducted for 200 h using the synthetic seawater of various NaCl concentrations at different feed temperatures. The experimental results show that the permeate flux decreased with the increasing feed concentration, whereas it was enhanced with the increasing feed temperature. A high water flux of  $38.2 \text{ kg/m}^2 \text{ h}$  and salt rejection up to 99.9% were obtained by CHFMs prepared from CRHA (CHFMs/CRHA).

### 1. Introduction

“Fresh water is the world’s first and foremost medicine.” Unfortunately, fresh water is scarce and more than one billion people in the world, especially from the developing countries, do not have access to it. According to the United Nations World Water Development Report [1], around 780 million people lack access to drinking water while the demand for processed water is expected to increase by 400% between 2000 and 2050 globally. Due to this reason, seawater desalination nowadays receives attention since seawater constitutes  $> 97\%$  of the total water on the earth [2]. Accordingly, many investigations on seawater desalination technologies have been reported in the literature [3–5]. An early investigation on seawater desalination was carried out by Riley et al. by reverse osmosis (RO), where a spiral-wound thin film composite membrane was used to achieve more than 99% of salt rejection [6]. In 2012, a review was written by Peñate and García-Rodríguez on the current trends and future prospects in the design of seawater reverse osmosis desalination technology [7]. It was reported that reverse osmosis is the most globally widespread technology for

seawater desalination. However, desalination using RO is an energy-intensive process. Furthermore, RO process is not able to reject boron satisfactorily in its boric acid form in seawater. In addition, the water treated through RO may not contain precious minerals such as calcium and magnesium which are necessary for maintaining good health [8].

Membrane distillation (MD) has emerged as a viable option for addressing current problem of seawater desalination. MD is a thermal process that uses a hydrophobic porous membrane, allowing water vapor, instead of liquid feed, to pass through its pores [9]. This technology possesses four configurations with respect to the cold side arrangement, namely (1) direct contact membrane distillation (DCMD), (2) vacuum membrane distillation (VMD), (3) sweeping gas membrane distillation (SGMD), and (4) air gap membrane distillation [10]. Among all configurations, DCMD is considered the one most commonly applied for seawater desalination, due to the least cost of equipment and the simplest design and operation [11]. Kharraz et al. conducted an investigation of real seawater desalination from the Arabian Gulf using DCMD process with polyvinylidene fluoride (PVDF) membrane by running it for 103 h [12]. A high flux ( $10 \text{ L/m}^2\text{h}$ ) and a high salt

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rejection (99.9%) could be achieved at the feed and permeate temperatures of 65 and 25 °C, respectively, with stable DCMD performance. Whereas, Munirasu et al. prepared superhydrophobic PVDF membrane by phase inversion for synthetic seawater desalination, where they used 35 g/L of NaCl solution with a conductivity of 15  $\mu$ S as the feed [13]. As a result, high water vapor flux of up to 90 L/m<sup>2</sup>h at feed the temperature of 60 °C was achieved with the salt rejection of > 99.99%.

However, the use of polymeric membranes, like PVDF, is severely restricted for long-term operations at elevated temperatures or drastic chemical conditions. Thus, ceramic membranes offer several advantages over polymeric membranes especially due to their non-swelling behavior and thermal stability, which are desirable in MD application [14–17]. However, the limitation of most commercial ceramic membranes such as alumina, titania, and zirconia is that they are hydrophilic in nature. Accordingly, modification of hydrophilic ceramic membrane to be hydrophobic using organic groups such as fluoroalkylsilanes (FAS) has been attempted. In 2004, Larbot et al. pioneered this work by successfully modifying the hydrophilic membrane to be hydrophobic with the contact angle of around 150° for desalination application [18]. A 100% salt rejection was obtained in their study, proving that ceramic membranes can be used instead of polymeric membranes for membrane distillation. However, ceramic membranes are made from alumina, zirconia, and titania whose costs are very high and their fabrication requires high sintering temperature, making them extremely expensive.

Nowadays, low cost ceramic membranes made from clay and waste materials are receiving much attention to be used for various water treatment applications. In a study by Das et al. on the development of low cost hydrophobic clay–alumina-based capillary membrane for MD desalination [19], a salt rejection of 99.96% with a vapor permeate flux of 98.66 L/m<sup>2</sup> day was successfully obtained at a feed temperature of 60 °C. In our previous work [20], low cost, green silica-based ceramic hollow fiber membrane was successfully prepared from waste rice husk through combined phase inversion and sintering technique for water filtration. In fact, we found that burning the rice husk at 600 and 1000 °C produced amorphous and crystalline silica-based rice husk ash (ARHA and CRHA), respectively, thus affecting the membrane properties such as pore size, porosity, and mechanical strength.

Therefore, the aim of this work was to investigate the feasibility of low cost, green silica-based ceramic hollow fiber membrane (CHFMs) for seawater desalination via DCMD. In this study, CHFMs from ARHA and CRHA sintered at 1200 °C were chosen, denoted in this manuscript as CHFMs/ARHA and CHFMs/CRHA, respectively. To turn the hydrophilic surface hydrophobic, the CHFMs were grafted with FAS. In addition, various techniques such as scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), and contact angle measurements were employed to investigate the membrane properties before and after the modification. The membrane performance in DCMD was studied using synthetic seawater (NaCl solution) as the feed. The sustainability of the modified CHFMs in a high salinity feed solution containing 6 g/L of NaCl was also evaluated for 200 h.

## 2. Materials and methods

### 2.1. Materials

The agricultural waste RH was obtained from North Jelapang rice mill, Muar, Johor, Malaysia, washed with 0.1 M of NaOH solution, rinsed with distilled water, dried in an oven, burned at 600 and 1000 °C to produce ARHA and CRHA, respectively, ball-milled, and sieved. Polyethersulfone (PESf, Radel A-300, Ameco Performance, USA), *N*-methyl-2-pyrrolidone (NMP, QRëC™), and Arlcel P135 (polyethyleneglycol 30-dipolyhydroxystearate, CRODA) were used as polymer binder, solvent, and dispersant, respectively, for the preparation of the spinning suspension. Tap water was used as bore fluid and

**Table 1**

Dope composition and spinning condition.

Dope composition (wt.%)	
Silica based rice husk ash	37.5
<i>N</i> -methyl-2-pyrrolidone (NMP)	56.5
Polyethersulfone (PESf)	5.0
Arlcel	1.0
Spinning parameters	
Bore fluid	Tap water
Coagulant fluid	Tap water
Bore fluid flow rate (mL/min)	10
Dope extrusion flow rate (mL/min)	10
Air gap (cm)	5
Spinneret: Outer diameter (mm)	2
Spinneret: Inner diameter (mm)	1

coagulant bath during the hollow fiber spinning. 1H,1H,2H,2H-Perfluorodecyltriethoxysilane (97%), with a chemical formula of C<sub>8</sub>F<sub>17</sub>C<sub>2</sub>H<sub>4</sub>Si(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>, was used as the grafting agent (Sigma Aldrich, USA). Ethanol (C<sub>2</sub>H<sub>5</sub>OH) and sodium chloride (NaCl) were purchased from Merck, Germany.

### 2.2. Preparation and modification of low cost, green silica-based ceramic hollow fiber membrane

Silica-based ceramic hollow fiber membrane was prepared from rice husk ash via phase inversion and sintering technique, as reported in our previous work [20,21,34]. Arlcel P135 (1 wt%) was added to NMP (56.5 wt%) under gentle stirring prior to the addition of RHA powder (37.5 wt%). The mixture was then ball-milled at 150 rpm for 48 h before the addition of PESf (5 wt%) and the ball milling was continued for another 48 h. Then, the suspension was degassed for at least 1 h and immediately transferred into a stainless steel syringe. The extrusion process was completed based on the fabrication conditions listed in Table 1. After the extrusion, the CHFMs precursors were soaked in water overnight to remove the residual solvent, dried in an oven, and sintered in air in a high-temperature tubular furnace (XY-1700 MAGNA). The temperature was increased at a heating rate of 2 °C/min from room temperature to 600 °C, at which the temperature was kept constant for 2 h to remove the polymer binder. The temperature was then increased to 1200 °C at a rate of 3 °C/min and held at 1200 °C for 3 h. Finally, the temperature was brought down to room temperature at a rate of 5 °C/min. Grafting of CHFMs was conducted first by rinsing with a mixture of distilled water and ethanol in a ratio of 2:1 and then drying in an oven at 100 °C. Prior to the grafting process, epoxy resin was used to seal both end of the CHFMs to avoid the FAS solution to penetrate into the bore side. Afterwards, the CHFMs was immersed in a grafting solution containing 2 wt% of FAS and 98 wt% of ethanol for 24 h, followed by rinsing with distilled water and drying in an oven before being assembled into a membrane module. The grafted CHFMs were denoted as CHFMs/ARHA and CHFMs/CRHA, depending on the temperature of HR burning either 600 or 1000 °C, respectively.

### 2.3. Membrane characterization

The SEM images of the CHFMs/ARHA and CHFMs/CRHA before and after grafting were taken using a JEOL JSM 6360 scanning electron microscopy (SEM). To prepare the samples, the CHFMs were snapped to obtain cross-sectional images. The outer surface images were obtained by placing a piece of CHFMs on a metal holder. All samples were sputtered by gold under vacuum. The CHFMs surface roughness was investigated by atomic force microscopy (AFM, Park XE-100). Similar to SEM, a piece of CHFMs was adhered to the metal holder. The membrane outer surface was then scanned in the size of 10 × 10  $\mu$ m. The surface roughness of the membrane was expressed in terms of mean surface roughness (R<sub>a</sub>).

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