CERAMIC HOLLOW FIBER MEMBRANE DERIVED FROM PALM OIL FUEL ASH FOR MEMBRANE DISTILLATION

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy

School of Chemical and Energy Engineering
Faculty of Engineering
Universiti Teknologi Malaysia

FEBRUARY 2021

DEDICATION

This thesis is dedicated to my beloved parents, who inculcated me the importance of learning new knowledge to equip myself to be a better and successful person in life. I would also like to dedicate my work to Koo Khong Nee, who has always been there with me through the ups and downs of my Ph.D. journey.

ACKNOWLEDGEMENT

I am grateful to have met many knowledgeable and helpful people who have guided and inspired me throughout my Ph.D. study. First, I would like to extend my utmost gratitude to my supervisor, Assoc. Prof. Ts. Dr. Mohd Hafiz Dzarfan Othman for his invaluable guidance and support. His selfless sharing of knowledge and willingness to give the best assistance in any form has been my greatest motivation to overcome the toughest challenge during this journey. He has also given me numerous opportunities that have widened my insights beyond the research works in the laboratory, such as the opportunities to participate in national and international exhibitions. I am also thankful to my co-supervisor, Assoc. Prof. Dr. Mohd Irfan Hatim Mohammed Dzahir for providing constructive feedback on one of my manuscripts.

A special thanks to Koo Khong Nee for her unconditional assistance and motivation that deeply inspired me to get through the hardships in the process of completing my study. I am also indebted to my friends from our research center, Dr. Siti Khadijah Hubadillah, Dr. Wong Kar Chun, Mohd Ariff Azali, Dr. Ng Be Cheer, Dr. Mohd Ridhwan Adam, Dr. Nur Hashimah Alias, Nurul Jannah Ismail, and Jeganes Ravi. Their valuable experience, knowledge, and technical support have undoubtedly given a boost to the success of my research. Without their continuous support, the completion of this thesis would have been impossible.

Furthermore, I also owe a big thanks to my family for their encouragement and motivation that helped me to get over the ups and downs throughout my Ph.D. journey. Last but not least, I also offer my wholehearted regards to those who have given supports in any aspect throughout the whole process of my research.

ABSTRACT

Membrane distillation (MD) is an emerging desalination technology which separates vaporized solutes from the feed solution using a hydrophobic membrane at fairly high temperature. Ceramic membranes are known to have excellent chemical and thermal stability. However, their application in MD has not been well received as compared to their polymeric counterparts due to high cost and intrinsic hydrophilicity. This study focused on the development of novel cost-effective hydrophobic ceramic hollow fiber membranes (CHFMs) from an industrial waste, palm oil fuel ash (POFA), for direct contact MD (DCMD). POFA has high silica and potassium oxide content that could endow it with lower sintering temperatures during membrane fabrication than that of the widely used alumina membranes. However, POFA also exhibits a significant amount of carbon and organic impurities that could be detrimental to the mechanical properties of CHFM. Hence, in the first stage of the study, POFA was subjected to thermal pre-treatment at temperatures of 500–1,000˚C, and the effect of pre-treatment temperature on the chemical and physical properties of POFA was correlated. It was found that the carbon content of POFA was eliminated after being pre-treated at ≥600˚C, whereas the silica content was improved to >70 wt%. Moreover, the physical properties of POFA changed with increasing pre-treatment temperatures. In the second stage of the study, the high-strength POFA-derived CHFMs were fabricated through combined phase inversion/sintering technique. It was found that the pre-treatment temperature of POFA, POFA loading, phase inversion parameters (i.e.: air gap distance, bore fluid flow rate), and sintering temperature had substantial influences on the morphology and mechanical properties of CHFM. A high-strength CHFM (98.1 MPa) was acquired at the following conditions: 700˚C POFA pre-treatment temperature; 55 wt% POFA suspension loading; 5 cm air gap distance; 9 mL/min bore fluid flow rate; 1,050˚C sintering temperature. To attain hydrophobic properties, the surface of the CHFM was modified via dip-coating with polymethylhydrosiloxane/tetraethylorthosilicate (PMHS/TEOS) hybrid in the third stage of the study. A novel post-coating spinning technique has been developed to facilitate the pore formation on the coating layer. The effect of the number of coating layer on the morphology of the CHFM was studied. The concentrations of ethanol and PMHS were also found to affect the surface morphology and hydrophobicity of the CHFM. High water contact angle (WCA) of 108.2˚ and liquid entry pressure with water (LEP_w) of 1.0 bar was achieved by the CHFM modified with the following conditions: TEOS/ethanol molar ratio: 1:45; PMHS/TEOS mass ratio: 1:10; the number of coating layer: 2; with post-coating spinning. An excellent DCMD desalination performance was achieved with a salt rejection of >99.98% and flux of 4.8 L/m^2h at the feed salinity of 35,000 ppm. The outcomes of this study suggest that the hydrophobic POFA-derived CHFM could be an excellent low-cost alternative for MD desalination applications.

ABSTRAK

Penyulingan membran (MD) merupakan teknologi penyahgaraman yang sedang berkembang dengan memisahkan zat terlarut yang teruap dari larutan suapan menggunakan membran hidrofobik pada suhu yang agak tinggi. Membran seramik mempunyai kestabilan kimia dan haba yang hebat, tetapi kurang mendapat perhatian dalam MD berbanding dengan membran polimer disebabkan kos yang tinggi dan sifatnya yang hidrofilik intrinsik. Kajian ini memfokuskan pada pembangunan membran gentian geronggang seramik hidrofobik (CHFM) baharu yang menjimatkan kos dari sisa industri, iaitu abu kelapa sawit (POFA) untuk penyulingan membran sentuhan langsung (DCMD). POFA mempunyai kandungan silika dan kalium oksida yang tinggi yang dapat menyumbang kepada penurunan suhu pensinteran semasa fabrikasi membran berbanding dengan alumina yang digunakan secara meluas. Namun begitu, POFA juga mengandungi karbon dan kekotoran organik yang boleh memudaratkan sifat mekanik CHFM. Oleh itu, pada peringkat pertama kajian, POFA menjalani pra-rawatan termal pada suhu 500–1,000˚C, dan pengaruh suhu pra-rawatan terhadap sifat kimia dan fizikal POFA telah dihubung kait. Keputusan menunjukkan kandungan karbon POFA telah dinyahkan setelah rawatan pada suhu ≥600˚C, manakala kandungan silika ditingkatkan menjadi >70 wt%. Selain itu, sifat fizikal POFA juga berubah dengan peningkatan suhu pra-rawatan. Pada peringkat kedua kajian, CHFM yang dihasilkan dari POFA yang mempunyai kekuatan tinggi dihasilkan melalui teknik gabungan penyongsangan fasa dan pensinteran. Hasil kajian menunjukkan suhu pra-rawatan POFA, kandungan POFA, parameter penyongsangan fasa (jarak sela udara, kadar aliran cecair penebuk), dan suhu pensinteran membran mempunyai pengaruh yang besar terhadap morfologi dan sifat mekanik CHFM. CHFM mempunyai kekuatan tinggi (98.1 MPa) diperoleh pada keadaan berikut: Suhu pra-rawatan POFA: 700˚C; kandungan POFA: 55 % jisim; jarak sela udara: 5 cm; kadar aliran cecair penebuk: 9 mL/min; suhu pensinteran: 1,050˚C. Untuk mencapai sifat hidrofobik, permukaan CHFM telah diubahsuai melalui celupan salutan hibrid polimetilhidrililoksana/tetraetilorthosilikat (PMHS/ TEOS) pada peringkat ketiga kajian. Teknik pemintalan pasca salutan baharu telah dibangunkan untuk memudahkan pembentukan liang pada lapisan salutan. Pengaruh bilangan lapisan salutan terhadap morfologi CHFM telah dikaji. Kepekatan etanol dan PMHS juga didapati mempengaruhi morfologi permukaan dan sifat hidrofobik CHFM. Sudut sentuhan air (WCA) (108.2˚) dan tekanan masuk cecair dengan air (LEP_w) (1.0 bar) yang tinggi telah dicapai oleh CHFM yang diubah suai pada keadaan berikut: nisbah molar TEOS/etanol: 1:45; nisbah jisim PMHS/TEOS: 1:10; bilangan lapisan: 2; dengan pintalan salutan pasca. Prestasi penyahgaraman DCMD hebat telah dicapai dengan penyahgaraman >99.98% dan fluks 4.8 L/m^{2}h pada tahap kemasinan 35,000 ppm. Hasil kajian ini menunjukkan bahawa CHFM hidrofobik yang berasal dari POFA dapat menjadi alternatif kos rendah yang hebat untuk aplikasi penyahgaraman MD.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
AGMD	-	Air gap membrane distillation
ATR	-	Attenuated total reflection
BET	-	Brunauer, Emmet and Teller
BJH	-	Barrett, Joyner and Halenda
BTME	-	Bis(trimethoxysilyl)ethane
CHFM	-	Ceramic hollow fiber membrane
CHNS	-	Carbon, hydrogen, nitrogen and sulfur
CNT	-	Carbon nanotube
CVD	-	Chemical vapor deposition
DCMD	-	Direct contact membrane distillation
DCDMS	-	Dichlorodimethylsilane
DCMS	-	Dichloromethylsilane
DGM	-	Dusty gas model
DTG	-	Derivative thermogravimetry
EDX	-	Energy dispersive X-ray
FAS	-	Fluoroalkylsilanes
FTIR	-	Fourier transform infrared
GOR	-	Gained output ratio
HDFTHDTES	-	1H,1H,2H,2H-heptadecafluorotetrahydrodecyltriethoxysilane
HDTMS	-	Hexadecyltrimethoxysilane
ICP-OES	-	Inductively coupled plasma optical emission spectroscopy
IR	-	Infrared
IUPAC	-	International Union of Pure and Applied Chemistry
LEP	-	Liquid entry pressure
LEP$_w$	-	Liquid entry pressure with water
LOI	-	Loss on ignition
MD	-	Membrane distillation
MED	-	Multi-effect distillation

MF - Microfiltration
MIP - Mercury intrusion porosimetry
MSF - Multistage flash
NFHTES - 1H,1H,2H,2H-nonafluorohexyltriethoxysilane
NMP - N-methyl-2-pyrrolidone
NiO/YSZ - Nickel oxide/yttria-stabilized zirconia
OTCS - n-octyltrichlorosilane
OTES - n-octyltriethoxysilane
PDMS - Polydimethylsiloxane
PEG - Polyethylene glycol
PES - Polyethersulfone
PFDA - 1H,1H,2H,2H-perfluorodecylacrylate
PFDTES - 1H,1H,2H,2H-perfluorodecyltriethoxysilane
PFOTES - 1H,1H,2H,2H-perfluorooctyltriethoxysilane
PFTDTES - 1H,1H,2H,2H-perfluorotetradecyltriethoxysilane
PMHOS - Polymethylhydroxysiloxane
PMHS - Polymethylhydrosiloxane
PMHS/TEOS - Polymethylhydrosiloxane/tetraethylorthosilicate
PMSQ - Polymethylsilsesquioxane
POFA - Palm oil fuel ash
PP - Polypropylene
PSD - Pore size distribution
PTFE - Polytetrafluoroethylene
PVDF - Polyvinylidenefluoride
PWF - Pure water flux
RMK11 - 11th Malaysia Plan
RO - Reverse osmosis
RTI - Rayleigh-Taylor instability
SAED - Selected-area electron diffraction
SEM - Scanning electron microscopy
SGMD - Sweeping gas membrane distillation
SWRO - Seawater reverse osmosis
TDS - Total dissolved solids

TCODS	-	Trichloro(octadecyl)silane
TCS	-	Trichloromethylsilane
TEM	-	Transmission electron microscopy
TEOS	-	Tetraethylorthosilicate
TG	-	Thermogravimetry
TGA	-	Thermogravimetric analysis
UF	-	Ultrafiltration
VC	-	Vapor-compression evaporation
VMD	-	Vacuum membrane distillation
WCA	-	Water contact angle
XRD	-	X-ray diffraction
XRF	-	X-ray fluorescence
YSZ	-	Yttria-stabilized zirconia

LIST OF SYMBOLS

A - Total permeation area of the membrane

A_i - Surface area of the insulation material

Al - Aluminium atom

Al_2O_3 - Alumina

As(III) - Arsenite

As(V) - Arsenate

B - Geometric pore coefficient

C - Carbon element

C_{fs} - Conductivity of feed solution

CO_2 - Carbon dioxide

C_p - Specific heat capacity

C_{ps} - Conductivity of permeate solution

D_i - Inner diameter of the membrane

D_o - Outer diameter of the membrane

F - Force at which CHFM fractured

H_2 - Hydrogen

H_2O - Water

J_w - Permeate flux

K - Potassium element

KBr - Potassium bromide

K_2O - Potassium oxide

L - Length of the membrane

$\dot{m}$ - Airflow rate

N - Nitrogen atom

N_2 - Nitrogen

NaCl - Sodium chloride

NaOH - Sodium hydroxide

O - Oxygen element

$Q_{dwelling}$ - Energy usage at the dwelling stage

$Q_{heating}$ - Energy usage at the heating stage

Q_{total} - Total energy usage

R - Salt rejection

$\mathrm{R_a}$ - Mean surface roughness

r_{max} - Maximum membrane pore size

Si - Silicon element

SiO_2 - Silica

Si_3N_4 - Silicon nitride

$\mathrm{T_0}$ - Temperature at the hot feed

$\mathrm{T_1}$ - Temperature at the cold permeate

TiO_2 - Titania

t - Time required to achieve the ΔW

t_g - Time required to collect 20 mL of gas

t_{hd} - Duration of the heating or dwelling process

t_i - Thickness of the insulation material

t_w - Time required to collect V_w

V_g - Volume of gas

V_w - Volume of the permeate collected

ZrO_2 - Zirconia

ΔW - Weight change of the permeate tank

$\Delta\theta$ - Temperature change

γ_l - Surface tension of the liquid

θ - Contact angle

λ - Thermal conductivity of the insulation material

ρ_c - Density of the coagulant

ρ_s - Density of the suspension

σ_F - Bending strength of the membrane

$-CH_3$ - Methyl

$-OH$ - Hydroxyl

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

Freshwater scarcity has been one of the major challenges in this modern era. Global climate change, flourishing agricultural and industrial development, rapid global population expansion, as well as aggravated water pollution have put great pressure on the world's freshwater resources. It has been estimated that two-thirds of the global population (4.0 billion people) currently live under the conditions of severe water scarcity for at least one month yearly (UN Water, 2019). Desalination is deemed as one of the most promising methods to augment the freshwater capacity to cater to the immense freshwater needs. Through desalination, seawater and brackish water can be converted into freshwater by removing the dissolved solutes.

Currently, reverse osmosis (RO) accounts for 84% of the total number of desalination plants in the world because of its capabilities to produce high purity water (Jones *et al.*, 2019). RO is a membrane desalination process, which can produce clean water with salt rejections greater than 99% (Lee *et al.*, 2011; Greenlee *et al.*, 2009). However, this technology requires high operating pressures (17–82 bar) that make it susceptible to membrane fouling, consequently compromising freshwater productivity and quality (Jiang *et al.*, 2017; Greenlee *et al.*, 2009). Thermal desalination is another key desalination technology that is widely used in many of the desalination plants in the Middle East (Greenlee *et al.*, 2009; Fritzmann *et al.*, 2007). This energy-intensive technology is commonly fueled by fossil fuels, which are non-environmentally friendly and not sustainable due to the high carbon emission and depleting fossil fuel reserves (Gude *et al.*, 2011; Kalogirou, 2005). Therefore, the development of desalination technology with consistent freshwater productivity and quality, as well as the feasibility to integrate with sustainable energy is greatly needed.

Membrane distillation (MD) is a burgeoning desalination technology and can be a replacement for the conventional desalination processes. It is a hybrid technology bringing together thermal and membrane processes that separate the vaporized solutes from the feed solution through a microporous hydrophobic membrane at fairly high temperatures. MD has several promising characteristics, such as (i) lower operating temperatures than the thermal desalination technologies as the feed solution is not required to be heated to its boiling point for the distillation to occur, (ii) much lower operating pressure compared to RO, (iii) theoretically 100% non-volatile solute rejection, and (iv) the performance is not affected by the high salinity of the feed solution (Ashoor *et al.*, 2016; Alkhudhiri *et al.*, 2012; Al-Obaidani *et al.*, 2008; Banat and Al-Shannag, 2000). In addition, the feasibility of integrating MD with renewable energies, such as the solar and geothermal energies, as well as the low-temperature industrial waste stream also makes it particularly attractive in reducing the operating cost (Lokare *et al.*, 2017; Sarbatly and Chiam, 2013; Blanco Gálvez *et al.*, 2009). Moreover, the lower operating pressure condition allows the use of membranes with larger pore size and lower mechanical properties requirements as compared to RO, thus making MD cost-effective (Tijing *et al.*, 2015; Alkhudhiri *et al.*, 2012). The membranes with hydrophobic properties and larger pore sizes also reduce the susceptibility of MD to fouling (Ashoor *et al.*, 2016; Alkhudhiri *et al.*, 2012).

Since its discovery in the early 1960s, the development and commercial implementation of MD has been relatively sluggish as compared to RO. The slow progress in the commercialization of MD technology has largely been associated with the lack of membrane materials with appropriate characteristics for the MD applications (Drioli *et al.*, 2015; Alkhudhiri *et al.*, 2012; El-Bourawi *et al.*, 2006). The research on the development of the MD membranes has been vibrantly growing since the last two decades (González *et al.*, 2017). In general, polymeric membranes have been extensively studied for MD applications due to their intrinsic hydrophobicity and low surface energy properties, ease of fabrication, low cost, and high availability (Xu *et al.*, 2019; Wang *et al.*, 2016; El-Bourawi *et al.*, 2006). Polytetrafluoroethylene (PTFE), polyvinylidene fluoride (PVDF), and polypropylene (PP) are popularly studied polymers for MD applications (González *et al.*, 2017; Alkhudhiri *et al.*, 2012). However, polymeric membranes possess low thermal stability and chemical resistivity (Wang *et al.*, 2016; Hendren *et al.*, 2009). These will affect the performances of the membranes in MD desalination, especially for long-term operations.

Ceramic membranes can be a viable option for MD applications due to outstanding mechanical, thermal, and chemical stability (Li *et al.*, 2016; Li, 2007). These remarkable properties allow ceramic membranes to operate at higher temperatures and in the presence of chemicals without concern for membrane deterioration (Li, 2007). As a result, ceramic membranes exhibit long membrane life span, which cannot be achieved by polymeric membranes. However, due to the high cost, the deployment of ceramic membranes for MD applications is still lacking (Hubadillah *et al.*, 2019b). Most of the ceramic membranes used in MD studies are made from expensive ceramic materials, such as alumina (Al_2O_3) and titania (TiO_2), which contribute immensely to the high cost of ceramic membranes (Fan *et al.*, 2017; García-Fernández *et al.*, 2017; Kujawa *et al.*, 2014a; Fang *et al.*, 2012). Hence, the development of low-cost ceramic membranes from alternative materials is pivotal to make the membranes more commercially attractive for MD applications.

Apart from developing ceramic membranes from low-cost starting materials, the membrane fabrication cost can also be minimized by lowering the sintering temperature to reduce energy consumption and shorten fabrication duration. The fabrication of ceramic membranes usually involves high sintering temperatures, which is one of the main reasons for the high fabrication cost. For instance, conventional Al_2O_3 membranes require an extremely high sintering temperature (usually >1,500˚C) to reach a trade-off between mechanical strength and porosity, which consequently results in high fabrication cost (Li *et al.*, 2016). Although a large number of studies have been reported on the development of low-cost ceramic membranes, the fabrication of these membranes still involves high sintering temperatures (Hubadillah *et al.*, 2020; Hubadillah *et al.*, 2018a; Jamalludin *et al.*, 2018; Li *et al.*, 2016). Therefore, a contemporary strategy to reduce the fabrication cost of ceramic membranes is by deploying a low-cost ceramic material with inherent sintering aid properties.

In this study, we developed low-cost ceramic hollow fiber membranes (CHFMs) using palm oil fuel ash (POFA) via the phase inversion/sintering technique. POFA is an industrial waste from the thriving palm oil industry. This material is colossal in amount and usually being disposed to open field that poses threats to the surrounding environment and local communities (Hamada *et al.*, 2020; Thomas *et al.*, 2017). POFA is mainly made up of silica (SiO_2) which could provide essential mechanical strength to ceramic membranes (Othman *et al.*, 2017; Thomas *et al.*, 2017). The SiO_2-rich composition could also bestow the ceramic membrane with a lower sintering temperature as compared to Al_2O_3 membranes (Othman *et al.*, 2017). Based on the literature, the development of low-cost ceramic membrane from SiO_2-based alternative materials such as rice husk ash, waste fly ash, ball clay and kaolin have been increasingly embraced in recent years. However, the fabrication of membranes from these materials still involves high sintering temperatures (>1,200˚C) to acquire high mechanical properties, which could be energy- and time-consuming (Hubadillah *et al.*, 2020; Abd Aziz *et al.*, 2019b; Zulkifli *et al.*, 2019; Hubadillah *et al.*, 2018a). The incorporation of liquid phase sintering aid has been known to stimulate diffusion mechanisms of ceramics and reduce sintering temperatures (Fang *et al.*, 2014; Vu *et al.*, 2013). However, the selection and process control of suitable sintering aid is rather complicated. Correspondingly, POFA contains a notable amount of potassium oxide (K_2O). K_2O has a relatively low melting point of ~700˚C, thus could act as a liquid phase sintering aid during membrane sintering. The intrinsic sintering aid properties of POFA could resolve problems related to the addition of sintering aid to the ceramic system, as well as reducing sintering temperature.

Ceramic membranes are inherently hydrophilic due to abundant hydroxyl (–OH) groups on the membrane surface (Fang *et al.*, 2012; Krajewski *et al.*, 2006). On the contrary, the membranes used for MD applications must be hydrophobic. The surface chemistry of ceramic membranes can be reversed from hydrophilic to hydrophobic through surface modification. Fluoroalkylsilane (FAS) is the most commonly used surface modifier to produce hydrophobic ceramic membranes due to the reduction of surface energy caused by the presence of abundant fluorine atoms (Hubadillah *et al.*, 2019b; Krajewski *et al.*, 2006). However, FAS is costly and can lead to high production costs of the hydrophobic ceramic membranes (Hubadillah *et al.*, 2019b; Ahmad *et al.*, 2015). Also, the instability of the FAS coating could cause the leaching of FAS into the water streams during separation processes, leading to detrimental environmental effects (Kujawa *et al.*, 2017a; Kujawa and Kujawski, 2016). The acquisition of hydrophobic properties via membrane surface modification with cheaper and non-fluorinated materials can be the key to overcome these constraints. Nonetheless, the establishment of hydrophobic ceramic membranes using alternative non-fluorinated low surface energy materials is still lacking, thus offering enormous opportunities for exploration.

In this regard, the non-fluorinated polymethylhydrosiloxane/ tetraethylorthosilicate (PMHS/TEOS) with low surface energy has been adopted for ceramic membrane surface modification in this study. PMHS/TEOS is an organic/inorganic hybrid material and possesses the advantageous characteristics of both organic and inorganic compounds (Wang *et al.*, 2017b; Katagiri *et al.*, 2001). Polymethylhydrosiloxane (PMHS) is a polymeric byproduct of the silicon industry, which is inexpensive, hydrophobic, non-toxic, and stable to air and moisture (Yadav *et al.*, 2019; Yang *et al.*, 2006). Having advantages of fascinating reaction character and flexible linear chain, PMHS can effectively take part in the sol-gel process together with tetraethylorthosilicate (TEOS). Besides, it can also tailor the surface chemistry of materials desirably and act as the structure-directing agent to produce a porous coating layer without the employment of a template (Guo *et al.*, 2015; Yang *et al.*, 2008). The incorporation of organic PMHS functional groups into the inorganic TEOS matrix creates a cross-linking organic-inorganic network, which changes the surface chemistry of the material and endows the hybrid material with hydrophobic properties. Moreover, PMHS/TEOS hybrid also inherits the excellent stability properties of inorganic compounds (Wang *et al.*, 2017b). PMHS/TEOS can be synthesized via the sol-gel method that is simple and well suited for large scale production (Sanchez *et al.*, 2011; Sanchez *et al.*, 2005). However, up until today, the use of PMHS/TEOS coating in the fabrication of hydrophobic ceramic membranes has not been explored. Therefore, it was of our interest to study the feasibility of PMHS/TEOS hybrid for the development of hydrophobic ceramic membrane for MD desalination.

1.2 Problem Statements

To circumvent the high fabrication cost of ceramic membranes for MD applications, a novel low-cost hydrophobic CHFM derived from POFA had been proposed in this work. Like other ash-typed materials, POFA comprises of a notable amount of carbon and organic impurities due to incomplete burning (Thomas *et al.*, 2017). The presence of carbon and organic impurities could change the pore size distribution of the membrane and impede the bonding between particles during sintering as well as, most importantly, adversely affecting the SiO_2 composition of the membrane (Thomas *et al.*, 2017; Chandara *et al.*, 2010; Jo *et al.*, 1996). High SiO_2 content of POFA is crucial as it could provide strong mechanical properties to CHFMs (Othman *et al.*, 2017). Carbon and organic impurities can be removed via thermal pre-treatment in the atmosphere (Alsubari *et al.*, 2018; Ali *et al.*, 2017; Chandara *et al.*, 2010). Therefore, in this study, POFA was subjected to thermal pre-treatment at different temperatures before the membrane fabrication process to overcome this challenge. Previous studies have shown that the chemical and physical properties of different ashes (i.e.: rice hull ash, sugarcane bagasse ash) would be changed at different calcination temperatures (Sánchez-Flores *et al.*, 2016; Ribeiro and Morelli, 2014; Yang *et al.*, 2008). Meanwhile, to date, the effect of pre-treatment temperature on the properties of POFA has yet been explored. Hence, the fundamental understanding of the effect of pre-treatment temperature on the chemical and physical properties of POFA was important to be explored in this study.

The pre-treated POFA was used for the fabrication of CHFM. It is widely known that CHFM is brittle due to small diameters which could lead to membrane failure during handling or operations (Wang *et al.*, 2016). Hence, high mechanical strength is particularly crucial for CHFM to ensure long-term performance stability of membranes (Fung and Wang, 2014; Xu *et al.*, 2014). Literature has shown that CHFMs with the mechanical strength of about 100 MPa are capable to produce stable and efficient separation performances (Abdulhameed *et al.*, 2017; Qasim Hussein *et al.*, 2015). Today, the study on the fabrication of the CHFM derived from POFA was still lacking and there has yet any report on the improvement of the mechanical strength of the POFA-derived CHFM. It is anticipated that the change of chemical and physical properties of POFA after pre-treatment at different temperatures could affect the properties of the derived CHFM. The chemical and physical properties of starting material could influence the phase inversion and sintering processes during the membrane fabrication process (Hubadillah *et al.*, 2018b; Hubadillah *et al.*, 2016b). Thus, it is pivotal to investigate how pre-treatment temperatures of POFA could affect the morphology and mechanical strength of the derived CHFM. Moreover, past studies have also witnessed the manipulation of ceramic loading, phase inversion parameters, and sintering temperature to augment the mechanical strength of CHFMs (Hubadillah *et al.*, 2018a; García-Fernández *et al.*, 2017). The variation of these parameters would change the morphological structure of membranes, and as a result, affecting their mechanical strength. Therefore, it was of great interest in this study to investigate the impacts of ceramic loading, phase inversion parameters, and sintering temperature on the mechanical strength of CHFM.

To reverse the surface chemistry of the POFA-derived CHFM from hydrophilic to hydrophobic for MD desalination, the surface of the membrane was modified via dip-coating with PMHS/TEOS hybrid material. Some studies have revealed that PMHS/TEOS sol-gel compositions could affect sol-gel behaviors and change the surface chemistry of the hybrid coating (Yang *et al.*, 2008; Yang *et al.*, 2006). Moreover, the change of sol-gel compositions and the number of coating layer would also affect the morphology of the coating layer (Anderson and Binions, 2014; Yang *et al.*, 2008). Thus, in this study, ethanol and PMHS concentrations of the PMHS/TEOS hybrid solution, and the number of coating layer were manipulated to enhance the hydrophobicity and morphological structure of the membrane coating. Additionally, a highly porous coating layer is also essential to ameliorate the flux across the membrane. Generally, the organic-inorganic hybrid coating layers prepared via the sol-gel method are dense with extremely small pore sizes (Dong *et al.*, 2020; Xiangli *et al.*, 2007). The use of the template-based sol-gel method has been widely adopted to produce porous coating layer, but the process of removing template can be energy consuming and less environmentally-friendly (Chua *et al.*, 2015; Chen *et al.*, 2011). Meanwhile, a study by Yang *et al.* (2008) has shown the successful development of macro-porous SiO_2 films from PMHS/TEOS via the spin coating method without the employment of a template. The fast-moving coating process facilitated the reaction between the unreacted PMHS and ethanol in the sol-gel system which released hydrogen (H_2) gas, producing a highly porous structure. Intrigued by Yang's success, this work adapted his fabrication approach to coat our hollow fiber substrate. Since the geometry of hollow fiber and flat substrate are different, here we developed a facile method to spin the hollow fiber immediately after the dip-coating process to produce a porous hydrophobic CHFM. The proposed approach could offer a simple, effective, and environmentally friendly pathway to produce hydrophobic ceramic membranes for MD desalination.

1.3 Research Objectives

The ultimate objective of this study was to develop a novel hydrophobic POFA-derived CHFM for MD desalination via the phase inversion/sintering technique followed by surface modification with PMHS/TEOS hybrid material. To achieve the final objective, this study was set out with the following specific objectives:

1. To correlate the effect of pre-treatment temperature on the chemical and physical properties of POFA
2. To examine the effects of pre-treatment temperature of POFA, POFA loading, phase inversion parameters, and sintering temperature on the development of high-strength POFA-derived CHFM
3. To investigate the influences of PMHS/TEOS sol-gel compositions, the number of coating layer, and coating procedure on the morphological structure and hydrophobicity, as well as the DCMD desalination performance of the POFA-derived CHFM

1.4 Research Scope

The scopes of the study have been identified and are listed as follows:

For objective 1:

(a) Pre-treating the POFA obtained from a crude palm oil mill in Chaah, Johor via the thermal process at different temperatures (500–1,000˚C).
(b) Studying the morphological changes of POFA via scanning electron microscopy (SEM) and transmission electron microscopy (TEM).
(c) Characterizing the changes of chemical properties of POFA through X-ray fluorescence (XRF) analysis, carbon elemental analysis, and Fourier-transform infrared (FTIR) spectroscopy.
(d) Identifying the changes of crystallinity, surface and pore properties, as well as the thermal stability of POFA using X-ray diffraction (XRD) spectroscopy, Brunauer, Emmet and Teller (BET) analysis, and thermogravimetric analysis (TGA), respectively.

For objective 2:

(a) Determining the rheological behaviors of the ceramic suspensions prepared from the untreated POFA and POFA pre-treated at different temperatures (500–1,000˚C), as well as with different POFA loadings (40–60 wt%) through viscosity tests.
(b) Fabricating CHFMs from the untreated POFA and POFA pre-treated at different temperatures (500–1,000˚C) via the phase inversion/sintering technique.
(c) Manipulating the POFA loading (40–55 wt%), air gap distance (5–15 cm), bore fluid flow rate (6–20 mL/min), and sintering temperature (1,025–1,100˚C) during the membrane fabrication process.

(d) Investigating the morphology, bending strength, surface roughness, as well as the pore size distribution (PSD) and porosity of the POFA-derived CHFM through SEM, three-point bending test, atomic force microscopy (AFM), and mercury intrusion porosimetry (MIP), respectively.

(e) Evaluating the water permeability of the POFA-derived CHFM using a crossflow filtration system at a pressure of 2 bar.

For objective 3:

(a) Preparing PMHS/TEOS hybrid solutions containing PMHS, TEOS, water (H_2O), and ethanol.

(b) Synthesizing SiO_2 powder from the sol-gel solution containing TEOS, H_2O, and ethanol

(c) Characterizing the chemical properties of SiO_2 and PMHS/TEOS hybrid powders via FTIR spectroscopy.

(d) Identifying the thermal stability of PMHS/TEOS hybrid powder via TGA under nitrogen (N_2) and air atmospheres

(e) Modifying the surface of the POFA-derived CHFM by dip-coating with PMHS/TEOS hybrid solution

(f) Evaluating the effects of PMHS/TEOS sol-gel compositions, such as TEOS/ethanol molar ratio (1:40–1:55) and PMHS/TEOS mass ratio (1:2–1:10) on the properties of the surface-modified POFA-derived CHFM

(g) Studying the effects of coating procedures (with and without post-coating spinning) and the number of coating layer (1–4) on the structure of the coating layer of the POFA-derived CHFM

(h) Characterizing the morphology, element distribution, surface topography, and apparent surface PSD, as well as the hydrophobicity of the surface-modified CHFM via SEM, energy-dispersive X-ray (EDX), AFM, and ImageJ analysis, as well as the WCA and LEP_w tests, respectively

(i) Determining the mechanical stability of the coating layer of the hydrophobic CHFM via ultrasonication followed by the WCA test and inductively coupled plasma optical emission spectroscopy (ICP-OES)

(j) Evaluating the gas permeability of the coated membranes using N_2 gas at predetermined pressures (0–1 bar)

(k) Assessing the desalination performance of the POFA-derived CHFMs coated with PMHS/TEOS solutions prepared with different PMHS/TEOS mass ratios (1:2–1:10) using 2,000-ppm synthetic saline solution (Operating conditions: feed temperature: 80˚C; coolant temperature: 10˚C; feed flow rate: 0.25 L/min; coolant flow rate: 0.2 L/min)

(l) Evaluating the desalination performance of the surface-modified CHFMs using synthetic saline solutions of different salt concentrations (2,000, 10,000, and 35,000 ppm) (Operating conditions: feed temperature: 80˚C; coolant temperature: 10˚C; feed flow rate: 0.25 L/min; coolant flow rate: 0.2 L/min)

(m) Analyzing and comparing the costs of the POFA-derived CHFM and the PMHS/TEOS hybrid with other materials reported in the literature

1.5 Research Significance

This research contributes to the development of low-cost hydrophobic ceramic membranes from alternative green ceramic materials. This would make ceramic membranes more economically sensible for MD applications. In addition, this research also embraces the waste-to-wealth concept through the utilization of industrial waste, POFA, for the fabrication of ceramic membranes. This initiative is in accordance with the 11th Malaysia Plan (RMK11) which steps up the focus of our country towards green growth. Moreover, the research on the surface modification of ceramic membrane using PMHS/TEOS hybrid material also helps to expand the frontier of knowledge in the development of hydrophobic ceramic surface with cheap and environmentally friendly non-fluorinated materials. As mentioned earlier, the development of MD desalination is relatively slow as compared to that of RO technology. Regarding this concern, we believe that the outcomes of this research can give significant impacts on the development of MD desalination in the efforts to realize the commercialization of MD technology in the near future. The implementation of MD in desalination will also contribute to achieving the United Nations' Sustainable Development Goal 6 which focuses on the sustainable management of clean water and sanitation.

1.6 Thesis Organization

This thesis reports a novel approach for the development of hydrophobic POFA-derived CHFMs via the phase inversion/sintering technique and surface modification with PMHS/TEOS hybrid for MD desalination. This thesis is organized into seven chapters. Chapter 1 provides a brief introduction concerning conventional desalination technologies, MD desalination, challenges with ceramic MD membranes, and the new approaches of this research for tackling these challenges. Besides, the problem statements, objectives, scopes, and significance of this research have also been addressed in the chapter. Chapter 2 presents a review of the important literature related to the topic of this research. This chapter contains the discussions on conventional desalination technologies, MD as emerging desalination technology, development of ceramic membranes, and surface modification of ceramic membranes for MD applications. Meanwhile, Chapter 3 gives a comprehensive description of the materials, experimental procedures, and characterization techniques used in this research.

The results and discussion of the research are addressed in Chapters 4 to 6. Chapter 4 describes the effect of pre-treatment temperature on the chemical and physical properties of POFA. In the chapter, the chemical composition, morphology, surface and pore properties, thermal behaviors, and crystallinity of the untreated and pre-treated POFA are discussed in detail. Meanwhile, Chapter 5 provides an in-depth discussion on the effects of several parameters, including the pre-treatment temperature of POFA, POFA loading, phase inversion parameters, and sintering temperature towards the fabrication of the POFA-derived CHFMs. In Chapter 6, a thorough discussion of the effects of PMHS/TEOS sol-gel compositions, the number of coating layer, and coating procedures on the morphological structure and hydrophobicity of the POFA-derived CHFM is provided. The desalination performance of the hydrophobic POFA-derived CHFM in DCMD and cost analysis of the developed hydrophobic CHFM are also covered in the chapter. Finally, Chapter 7 provides conclusions based on the findings obtained in this research. Some recommendations are also addressed for subsequent studies to explore the gaps in this research.

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1. **Tai, Z. S.**, Hubadillah, S. K., Othman, M. H. D., Mohamed Dzahir, M. I. H., Koo, K. N., Izadin Tendot, N. I. S. T., Ismail, A. F., Rahman, M. A., Jaafar, J., & Aziz, M. H. A. (2019). Influence of pre-treatment temperature of palm oil fuel ash on the properties and performance of green ceramic hollow fiber membranes towards oil/water separation application. *Separation & Purification Technology*, 222, 264–277. https://doi.org/10.1016/j.seppur.2019.04.046. **(Q1, IF: 5.107)**

2. **Tai, Z. S.**, Aziz, M. H. A., Othman, M. H. D., Mohamed Dzahir, M. I. H., Hashim, N. A., Koo, K. N., Hubadillah, S. K., Ismail, A. F., Rahman, M. A., & Jaafar, J. (2020). Ceramic membrane distillation for desalination. *Separation & Purification Reviews*, 49(4), 317–356. https://doi.org/10.1080/15422119.2019.1610975. **(Q1, IF: 4.940)**

3. **Tai, Z. S.**, Othman, M. H. D., Mohamed Dzahir, M. I. H., Hubadillah, S. K., Koo, K. N., Azali, M. A., Alias, N. H., Mustafa, A., Ooi, B. S., Kurniawan, T. A., & Ismail, A. F. (2020). Design and fabrication of ceramic hollow fiber membrane derived from waste ash using phase inversion-based extrusion/sintering technique for water filtration. *Journal of Asian Ceramic Societies*, Accepted. **(Q1, IF: 2.653)**

4. Hubadillah, S. K., **Tai, Z. S.**, Othman, M. H. D., Harun, Z., Jamalludin, M. R., Rahman, M. A., Jaafar, J., & Ismail, A. F. (2019). Hydrophobic ceramic membrane for membrane distillation: A mini review on preparation, characterization, and applications. *Separation & Purification Technology*, 217, 71–84. https://doi.org/10.1016/j.seppur.2019.02.014. **(Q1, IF: 5.107)**

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1. **Tai, Z. S.**, Othman, M. H. D., Hubadillah, S. K., Ismail, A. F., Rahman, M. A., Jaafar, J., Koo, K. N., & Aziz, M. H. A. (2018). Low cost palm oil fuel ash based ceramic membranes for oily water separation. *Malaysian Journal of Fundamental and Applied Sciences*, 14(4), 419–424. https://doi.org/10.11113/mjfas.v14n4.1218. **(Indexed by SCOPUS)**

Book Chapter

1. **Tai, Z. S.**, Aziz, M. H. A., Othman, M. H. D., Ismail, A. F., Rahman, M. A., & Jaafar, J. An overview of membrane distillation. In: Ismail, A. F., Rahman, M. A., Othman, M. H. D., & Matsuura, T. *Membrane Separation Principles and Applications: From Material Selection to Mechanisms and Industrial Uses*. Amsterdam, Netherlands: Elsevier. 251–281; 2019. https://doi.org/10.1016 /B978-0-12-812815-2.00008-9.