MEMBRANE DISTILLATION OMNIPHOBIC MULLITE HOLLOW FIBRE MEMBRANE WITH FLUOROALKYLSILANE-FUNCTIONALISED TITANIA DEPOSITION

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DEDICATION

This thesis is specially dedicated to my father, Mr Abd Aziz Samsuddin and my mother, Ms Norezzan Ghani,

without whom none of my success would be possible.

For my family and friends

Thank you for the encouragement and love

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ABSTRACT

Membrane distillation (MD), which is capable of achieving high solute rejection, has recently attracted significant attention for desalination. However, membrane fouling and wetting are still the major challenges in membrane distillation systems. Hence, it is essential to prepare an omniphobic membrane for anti-fouling and anti-wetting performance to overcome the issue. This work suggested a facile fabrication method of omniphobic mullite hollow fibre membrane via a one-step synthesis of growing hierarchical titania (TiO_2) particles on the membrane surface through hydrothermal method and followed by surface fluorination. Mullite hollow fibre membrane (HFM) was prepared as a substrate from ball clay using phase inversion and sintering technique. The composition of raw ball clay consisted of 85.9% kaolinite, 9.5% illite, 3.6% quartz and 1% maghemite. The particles of raw ball clay were irregular in shape, and some particles reach dimensions of over 50 μm size. After pre-treatment processes, the particle size of ball clay powder was remarkably reduced to 4.96 μm. The physico-chemical and permeation properties of a membrane were investigated by varying ball clay loading and sintering temperature. After the sintering process, major composition of the hollow fibre membranes was mullite with minor traces of quartz. When the membrane with ball clay loading of 47.5 wt.% was sintered at 1250 °C, its mechanical strength and permeability were comparable to that of membranes fabricated from pure metal oxides. The membrane had an average porosity and pore size of about 50.5 ± 2.1% and 0.61μm, respectively. Subsequently, hydrothermal treatment was carried out at 150°C to acquire re-entrant structures on the hollow fibre membrane's surface followed by the fluorination with 1H, 1H, 2H, 2H-perfluorodecyltriethoxysilane (C8). The formation of rod-like (RL) and flower-like (FL) TiO_2 structures was observed after 5 and 10 hours of hydrothermal process. After surface texturing and fluorination at 48 hours, the hollow fibre membrane, which was initially hydrophilic in nature, exhibited high liquid repellence towards water and low surface tension liquids such as ethylene glycol and olive oil. The order of the membranes in terms of wetting resistance for low surface tension liquids is as follows: C8-FL/TiO_2-HFM > C8-RL/TiO_2-HFM> C8-HFM. The value of contact angle for water on C8-FL/TiO_2-HFM was around 162^o, which is among the highest of previously reported contact angle of ceramic membranes in MD system. Also, the membrane exhibited nearly superomniphobic properties towards olive oil, ~140^o. In addition, the formation of air layers was observed on submerged C8-FL/TiO_2-HFM and C8- RL/TiO_2-HFM, which significantly reduced the deposition of organic substances after 500 minutes of MD with an aqueous NaCl (3.5 wt.%) containing humic acid (10 mg/L). A rise in the permeate salt concentration was observed for C8-HFM but not for C8-FL/TiO_2-HFM and C8-RL/TiO_2-HFM. Moreover, no significant fouling was observed for C8-FL/TiO2-HFM and the membrane exhibited the most stable flux and the highest salt rejection compared to other membranes. These results suggest that the fabricated membrane with micro/nano-roughness from flower-like structures is potential for a robust MD process as compared to other membranes.

ABSTRAK

Penyulingan membran (MD), yang mampu mencapai penyahan zat terlarut yang tinggi, telah menarik banyak perhatian baru-baru ini untuk proses penyahgaraman. Walau bagaimanapun, kekotoran dan pembasahan membran masih menjadi cabaran utama. Oleh yang demikian, penyediaan membran omnifobi yang anti kepada kotoran dan pembasahan adalah penting untuk mengatasi masalah itu. Kajian ini mencadangkan kaedah pembikinan mudah membran omnifobik mulit gentian geronggang melalui satu langkah sintesis pertumbuhan zarah titanium dioksida (TiO_2) yang berhierarki pada permukaan membran melalui kaedah hidrotermal dan diikuti dengan pemfluorinan. Membran mulit gentian geronggang (HFM) yang dijadikan sebagai substrat dari lempung bebola dihasilkan menggunakan teknik penyongsangan fasa dan pensinteran. Komposisi lempung bebola mentah terdiri daripada 85.9% kaolinit, 9.5% illit, 3.6% kuarza dan 1% magemit. Zarah-zarah dalam lempung bebola mentah tidak mempunyai saiz yang sekata dan sesetengah zarah mencapai ukuran lebih dari 50 µm. Selepas proses pra-rawatan, serbuk lempung bebola mengecil kepada 4.96 µm. Sifat fiziko-kimia dan sifat telapan air membran telah diselidiki dengan pelbagai kandungan lempung bebola dan suhu pensinteran. Selepas proses pensinteran, komposisi utama membran seramik gentian geronggang adalah mulit dan sedikit kuarza. Apabila membran dengan kandungan 47.5 wt.% lempung bebola di sinter pada suhu 1250^{o}C, kekuatan mekanik dan kebolehtelapan air adalah setanding dengan membran diperbuat daripada logam oksida tulen. Saiz liang dan keporosan membran tersebut adalah sekitar 0.61 µm dan 50.5 ± 2.1%. Selepas itu, kaedah hidroterma dilaksanakan pada suhu 150^{o}C untuk memperoleh struktur ceruk pada permukaan membran diikuti dengan pemfluorinan dengan 1H, 1H, 2H, 2H-peifluorodekiltrietoxisilana (C8). Penghasilan TiO_2 yang berbentuk seperti batang (RL) dan seperti bunga (FL) dapat dilihat selepas 5 dan 10 jam proses hidroterma. Setelah tekstur permukaan dan fluorinasi pada 48 jam, membran gentian geronggang yang pada awalnya bersifat hidrofilik, menunjukkan penolakan cecair yang tinggi terhadap air dan cecair yang mempunyai tegangan permukaan yang rendah seperti etilena glikol dan minyak zaitun. Urutan membran dari segi anti pembasahan adalah seperti berikut: C8-FL/TiO_2-HFM>C8-RL/TiO_2-HFM>C8-HFM. Nilai sudut sentuh air untuk C8-FL/TiO_2-HFM adalah lebih kurang 162^{o} dan merupakan antara nilai yang tertinggi dikalangan membran seramik yang telah dilaporkan sebelum ini untuk penggunaan penyulingan membran. Membran itu juga menunjukkan sifat hampir superomnifobik terhadap minyak zaitun, ~140^{o}. Selain itu, pembentukan lapisan udara juga dapat dilihat pada C8-FL/TiO_2-HFM dan C8-RL/TiO_2-HFM bila direndamkan dalam air, yang secara signifikan terbukti dapat mengurangkan pemendapan bahan organik pada permukaan membran tersebut setelah 500 minit proses MD dilakukan dengan air larutan NaCl (3.5 wt.%) yang mengandungi asid humik (10 mg/L). Kenaikan kepekatan garam dapat dilihat jika C8-HFM digunakan tetapi tidak untuk C8-FL/TiO_2-HFM dan C8-RL/TiO_2-HFM. Selain itu, tiada kotoran yang ketara dapat dilihat pada C8-FL/TiO_2-HFM dan membran itu juga menunjukkan aliran wap air yang paling stabil dan penyahgaraman yang paling tinggi berbanding dengan membran yang lain. Hasil dapatan ini menunjukkan bahawa membran yang mana permukaannya diperbuat daripada kekasaran mikro/nano dari struktur bunga, menunjukkan potensi untuk penggunaan MD yang lebih mantap berbanding dengan membran yang lain.

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LIST OF ABBREVIATIONS

AGMD	-	Air gap membrane distillation
Al_2O_3	-	Alumina
APTES	-	3-aminopropyl-triethoxysilane
ATR	-	Attenuated total reflection
C6	-	1H,1H,2H,2H-perfluorooctyltriethoxysilane
C8	-	1H,1H,2H,2H-perfluorodecyltriethoxysilane
C10	-	1H,1H,2H,2Hperfluorododecyltriethoxysilane
C12	-	1H,1H,2H,2H-perfluorotetradecyltriethoxysilane
$CaSO_4$	-	Calcium Sulfate
DCM	-	Dichloromethylsilane
DCMD	-	Direct contact membrane distillation
DMC	-	Dimethyldichlorosilane
DTA	-	Differential thermal analysis
EDX	-	Energy dispersive X-ray analyzer
FAS	-	Fluoroalkylsilane
FESEM	-	Field emission scanning electron microscope
Fe_2O_3	-	Iron oxide
FL	-	Flower-like
FTIR	-	Fourier-transform infrared spectroscopy
HCL	-	Hydrochloric acid
HFM	-	Hollow fibre membrane
ICP-OES	-	Inductively coupled plasma - optical emission spectrometry
LEP_w	-	Liquid Entry Pressure of water
MED	-	Multi-effect distillation
MF	-	Microfiltration
MIP	-	Mercury intrusion porosimetry
MSF	-	Multi-stage flash distillation
NaCl	-	Sodium chloride
NF	-	Nanofiltration
NMP	-	N-methyl-2-pyrrolidone

NOM	-	Natural organic matter
OH	-	Hydroxyl group
PA174	-	3-methacryloxypropyltrimethoxysilane
PES	-	Poly-ethersulfone
PEG-30	-	Polyethyleneglycol 30 Dipolyhydroxystrearate
PP	-	Polypropylene
PPFDA	-	1H,1H,2H,2H-perfluorodecyl acrylate
PS	-	Polystyrene
PTFE	-	Polytetrafluoroethylene
PVDF	-	Polyvinylidene fluoride
PVDF-HFP	-	Poly(vinylidene fluoride-co-hexafluoropropylen
RIR	-	Reference intensity ratio
RL	-	Rod-like
RO	-	Reverse osmosis
SDS	-	Sodium dodecyl sulfate
SEM	-	Scanning electron microscope
SGMD	-	Sweeping gas membrane distillation
SiN	-	Silicon nitride
SiC	-	Silicon carbide
SiO_2	-	Silicon dioxide
Si_2N_2O	-	Silicon oxynitride
TCS	-	Trichloromethylsilane
TG	-	Thermogravimetry
TGA	-	Thermogravimetric analysis
TiO_2	-	Titania
TMS	-	Trimethyl- chlorosilane
UV	-	Ultraviolet
VMD	-	Vacuum membrane distillation
WHO	-	World Health Organization
XRD	-	X-ray diffraction
ZnO	-	Zinc oxide

LIST OF SYMBOLS

A	-	Effective area of a membrane
C_f	-	Feed salt concentration
C_p	-	Permeate salt concentration
D	-	Spacing between the solid textures
D_i	-	Inner diameters
D_o	-	Outer diameter
D_{50}	-	Medium value
F	-	Force at which a specimen fails or fractures
H^*	-	Robustness height
J_o	-	Initial flux
J_w	-	Permeate flux across the membrane
L	-	The span
L_{cap}	-	Capillary length of the fluid
M_m	-	Maximum loss of mass
M_t	-	Loss of mass at a certain temperature in the TG curve
P_H	-	Pressure required to force the sagging of the liquid-vapour interface due to height different
P_{re}	-	Pressure reference
P_θ	-	Pressure required to force the sagging into a solid texture
R	-	Salt rejection
Ra	-	Surface roughness
T^*	-	Robustness angle
W	-	Weight of permeate collected
V	-	The collected amount of permeate
f_{lv}	-	Area fraction of the liquid-air interface
f_{sl}	-	Area fraction of the solid-liquid interface
h	-	Time taken by the water passage across the membranes' wall
m_1	-	Weight of the dry membrane
m_2	-	Weight of the wet membrane
r	-	Ratio of the actual surface area to the projected surface area

γ_{sl}	-	Interfacial energies between the solid-liquid
γ_{sv}	-	Interfacial energies between the solid-vapour
γ_{lv}	-	Interfacial energies between the liquid-vapour
$\delta\theta$	-	Sagging angle
$\rho 1$	-	Theoretical density of mullite
$\rho 1$	-	Theoretical density of water
ε	-	Membrane's porosity
ψ	-	Local geometric angle
ϕ	-	Opening angle of a spine
α	-	Titling angle of a spine
θ_e	-	Equilibrium contact angle on a smooth surface
θ	-	Apparent contact angle
Å	-	Angstrom
cm	-	Centimetre
cm^2	-	Cubic centimetre
g	-	Gram
h	-	Hour
Kg	-	Kilogram
L	-	Litre
MPa	-	Mega Pascal
m	-	Metre
m^2	-	Cubic metre
mg	-	Milligram
min	-	Minute
mm	-	Millimetre
mL	-	Millilitre
N	-	Newton
nm	-	Nanometre
ppm	-	Parts per million
^oC	-	Degree Celsius
%	-	Percentage
μm	-	Micrometre
μS	-	Microsiemens

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CHAPTER 1

INTRODUCTION

1.1 Research Background

It is worth quoting that, “water is a driving force of all nature”. Water is a basic need and a foundation of human life. Lack of access to fresh water may lead to competition and struggle in getting new water resources that will possibly trigger crises in the future. Water shortage around the world is no fiction but a real global phenomenon as fresh water systems are not enough for all uses, whether domestic, agricultural, and industrial. A billion people have no access to drinkable water and many fresh water systems from lakes and rivers are too polluted to be used [1] and are drying up day by day at alarming rates. In addition, the world population is growing gradually from 7.6 billion as of mid-2017 to 8.5 billion and 9.7 billion of people to populate the whole world by 2030 and 2050, respectively [2]. The growing human population as well as climate change and industrialization are likely to result in water stress for the many fresh water systems around the world [3]. The rising demand of fresh water in many countries has made seawater desalination an attractive option to supply drinking water [4].

To date, membrane-based technologies have become a centre of attention as an effective method for seawater desalination. Among many desalination technologies, reverse osmosis (RO) is the most commonly used and is the most efficient pressure-driven technology for the production of clean water [5,6]. However, there are some disadvantages to this technology. In most cases, it requires high osmotic pressure, is cost-inefficient for small-scale applications and cannot be used in harsh conditions (e.g., high salinity, high temperature or corrosive) [5]. In this regard, membrane distillation (MD) seems to be a promising alternative for the production of sustainable freshwater due to its numerous distinct advantages over the pressure-driven desalination technology. Owing to its separation mechanism, MD has rejection rates of up to 100% for non-volatile compounds and produces extremely pure water. Besides, MD can operate at relatively low operating pressure and temperature [7]. In fact, the heat demand required by MD could be provided by waste heat and renewable solar energy [8]. Its ability to be potentially compact and more adaptable than conventional thermal desalination processes further raises MD to renown as a cost-effective candidate for portable and off-grid applications [9].

MD is a non-isothermal process and this emerging membrane technology requires a micro-porous hydrophobic membrane to separate vapour phase from liquid phase within the membrane pores. Water molecules evapourate and diffuse through the membrane and then condense at the permeate side, giving high rejection (nearly 100%) of dissolved and non-volatile species. The permeate vapour flux is driven by vapour pressure difference, which is induced by the temperature gradient across the hydrophobic membrane. Generally, there are four basic MD configurations which are direct contact membrane distillation (DCMD), air gap membrane distillation (AGMD), vacuum membrane distillation (VMD), and sweep-gas membrane distillation (SGMD). Among the four different configurations of MD, direct contact membrane distillation (DCMD) is the most attractive route because it is simple and less complex for operations [10].

Hydrophobic microfiltration (MF) membranes are widely used in the MD field [11] and hydrophobic polymeric membranes such as polyvinylidene fluoride (PVDF), polytetrafluoroethylene (PTFE), and polypropylene (PP) are often considered for MD membranes [12]. However, these membranes are well known for their susceptibility to harsh environments - high temperature and high pressure, which consequently results in a short life span of the membranes. The poor stability of polymeric membranes has raised concern for long-term operations of MD systems [13]. Despite the broad potential offered by the MD technology, the unviability of having the appropriate membrane for MD systems results in limited commercial uptake. In contrast to the polymeric membrane, the ceramic membrane is an ideal candidate when long-term stability and porous membrane structure is required [14]. This membrane is robust under harsh environments owing to its superior chemical, mechanical, and thermal stabilities [15,16]. To date, ceramic materials such as alumina (Al_2O_3), titania

(TiO_2), and zirconia (ZnO) are the common materials in fabricating ceramic membranes. Non-oxide ceramic materials like silicon nitride (SiN) and silicon carbide (SiC) have also been documented by a number of studies [16,17]. However, most of the materials are expensive in bulk and the application cost of these kinds of membranes for industrial purpose is one- and three-orders of magnitude more per unit of membrane in comparison to the polymeric membrane [18]. Due to that, some efforts have been devoted to achieving low-cost ceramic membranes from natural-occurring materials such as bauxite [19], kaolin clay [20], rice husk [21], and even inorganic industrial waste like fly ash [22].

Among the many major classifications of clay such as pure kaolinite and montmorillonite, ball clay is an attractive candidate to substitute the expensive commercial ceramic materials in tackling the issue of large-scale uptakes. This clay is one of the cheapest, abundant, and environmentally friendly minerals [23]. Ball clay is aluminosilicate and is rich in kaolinite, which makes up over 70% of the ball clay [23,24]. Thermal treatment of the aluminosilicate clay involves the formation of mullite, which is another type of ceramic that is hard, resistant to chemicals, and provides great strength due to its elongated crystal structure [25]. In Malaysia, ball clay can be found in abundance in the state of Perak and is rich in kaolinite (>75%), with low content of impurities [24]. Besides, because ball clay is an aluminosilicate system and presumably contains trace impurities such as iron oxide (Fe_2O_3), lower temperature is expected for sintering process [19]. The use of ball clay to fabricate ceramic planar membranes for water treatment has been reported in these existing literatures [26,27]. Meanwhile, to the best of my knowledge, no works have been carried out to use ball clay for the fabrication of ceramic membrane in a hollow fibre configuration. Since MD requires microfiltration membranes, enabling ball clay as a material for the fabrication of ceramic hollow fibre membranes is highly advantageous.

Despite being highly resistant to extreme conditions, ceramic membranes are inherently hydrophilic in nature, meaning that they cannot be directly applied for MD technology. Hence, surface modification is vital to acquire a hydrophobic surface prior to MD application. So far, the grafting of ceramic membrane surfaces with the low surface energy material of Fluoroalkylsilane (FAS) has been the most frequently used technique to obtain hydrophobic properties [14]. The FAS includes 1H,1H,2H,2H-perfluorooctyltriethoxysilane (C6), 1H,1H,2H, 2H-perfluorodecyltriethoxysilane (C8), 1H,1H,2H,2Hperfluorododecyltriethoxysilane (C10) and 1H,1H,2H,2H-perfluorotetradecyltriethoxysilane (C12). Generally, the grafting of ceramic membranes from previous studies was mainly applied with C6 and C8 [28] since the hindrance steric effect was observed for more hydrophobic molecules like C10 and C12 [29].

Though successful hydrophobic modification of ceramic membrane was achieved through FAS functionalization, hydrophobic membranes still fail to perform under the harsh conditions due to fouling and wetting [12]. By shifting towards superhydrophobicity, surface modification of membranes can be an effective method to alleviate pore fouling and wetting [30]. Meanwhile, omniphobic membranes are more effective in combatting incoming feed liquids because they exhibit super-repellency towards both high and low surface tension materials [31–35]. Omniphobic surfaces can be fabricated with the combination of appropriate surface roughness and low surface energy functional groups [36–38]. At present, numerous studies on the omniphobic membrane surface have used nanoparticles to construct nanoroughness. For instance, an omniphobic membrane was successfully fabricated after depositing silicon dioxide (SiO_2) [32,34,39] and ZnO nanoparticles [12]. Wetting and fouling experiments on the omniphobic membranes indicated higher resistance not only towards low surface tension feed liquid but also foulants as compared to hydrophobic membranes.

Designing an omniphobic surface that remains unwetted to any liquid requires re-entrant structures and a layer of low surface energy at the composite interface [12,31,38]. When it is immersed in liquids, a protective air layer called plastron will be formed [31], thus making it capable of being an antifouling and antiwetting agent. Deposition of sphere-like nanoparticles including TiO_2, SiO_2, and ZnO has been intensively reported to compose re-entrant structures on membrane surfaces for MD to mimic the “lotus effect” [12,40–42]. Meanwhile, to prepare a more robust omniphobic surface, a hierarchical re-entrant structure should be created in addition to having specific chemical composition. Typically, micro/nano-structured surface is fabricated to create hierarchical re-entrant surface through a multiple step, where a finer length scale texture is deposited on an underlying coarser length scale texture. This hierarchical texture exhibited better omniphobicity as compared to surfaces with a single scale texture [43]. The hydrothermal synthesis of TiO_2 can be an effective method for the impartment of hierarchical re-entrant structures due to its capability of constructing particles with various morphologies (flower-like, rod-like, etc.) [44] at fast reaction velocities and low-costs [45]. Different morphologies of rod-like and flower-like can be obtained by controlling reaction times [46]. This modification has been applied in various fields such as solar cells, electrical systems, and photocatalytic systems [47]. Through this simple hydrothermal process, TiO_2 particles can be grown directly on the substrate surface, thus simplifying the complex process of creating micro/nano-roughness besides, the adhesion of particles can be improved [48]. Hierarchical structures such as the flower-like morphology have been used to develop re-entrant structures for the preparation of omniphobic surfaces on other various substrates [44,49,50]. The hierarchical particles have the ability to introduce more air pockets around them, consequently imparting a stable Cassie-Bexter regime with a high water contact angle (>150^o) [51]

Meanwhile, for the ceramic membrane support, it is worth noting that its properties (pore size, porosity, mechanical strength, etc.) are crucial for MD [52] and it can change significantly under the process conditions of sintering, the composition of ceramic suspension, the distribution of particle size as well as the parameters of extrusion [53–55]. For instance, the particle size distribution of ceramic powder can affect the pore size and selectivity of membrane [56]. Other factors include the ceramic composition in ceramic-solvent-polymer suspension solution that can affect the mechanical strength and quality of sintered membrane [57]. Besides, during the process of sintering, a compromise always has to be made between the pore size and porosity, and the mechanical strength of membrane [56]. As a reference, it has been suggested that the accepted values of membrane porosity and pore size for MD vary from 30% to 85% [58] and from 0.2μm to 1μm [16,59], respectively. Meanwhile, the mechanical strength of ceramic membrane has to be more than 50 MPa in order to be on par with the strength of some high purity metal oxide ceramic membranes [60].

1.2 Problem Statement

Ceramic membranes offer long-term stability as their lifespan was reported to be 20 years, which is generally fourfold longer than that of polymeric membranes [61,62]. Because of that, hydrophobic ceramic membranes have been developed and applied for MD applications [12,21,32,63,64]. Among the many ceramic materials, ball clay serves as a promising aluminosilicate material for ceramic membrane fabrication. Mullite can be produced through thermal decomposition of ball clay, which is one of the most attractive ways of preparing mullite because of the abundance of clay and the relatively low sintering temperature for mullitisation [23]. Because of its potential, ball clay can be highly advantageous for MD membranes. To make hydrophobic ceramic membranes, fluorination with FAS has been a common method [16,64–68]. However, the membranes' surface hydrophobicity does not seem to reach extreme water repellency as most of the water contact angle results were below 150°. Therefore, membrane fouling and wetting are still major hitches for hydrophobic ceramic membranes. FAS-functionalized ceramic membranes can be easily wetted with low surface tension materials (e.g. dissolved organic matter, surfactants and hydrophobic species) that exist in wastewater or seawater [12,37,69,70]. The high affinity of these contaminants towards hydrophobic membrane surfaces contributes to membrane fouling and wetting and consequently severe declines in permeate flux, salt rejection, and energy efficiency [31,71,72].

To date, applications of micro/nanomaterials have been widely explored on polymeric membranes to enhance the membranes' omniphobicity, and these omniphobic surfaces display contact angles greater than 150° with a wide range of surface tensions [37,73,74]. Meanwhile, the application of omniphobic surfaces with hierarchical textures for ceramic membranes is still scarce since many of the previous works focused on polymeric membranes as substrates and the methods were complex as they involved multiple steps in developing the hierarchical textures. Recently a one-step synthesis of hierarchical texture on the surface of ceramic membranes was developed by growing flower-like silicon oxynitride (Si_2N_2O) nanowires via a vapour–solid process [63]. The membrane had a water contact angle of 160°. However, the method required a high operating temperature that was around 1400°C to grow the particles. To the best of my knowledge, studies have yet to report the use of a one-step synthesis of the hydrothermal method to prepare omniphobic ceramic membranes with hierarchical re-entrant structure at the interface, particularly for MD applications. In comparison with various other ceramic membrane configurations, ceramic hollow fibre membranes could enhance performance in terms of flux as they have higher packing densities and are less resistant to water vapour [12]. These advantages show that this type of membranes provides promising membrane substrates to extend the membranes' application for desalination via MD. Therefore, this study aims to develop a novel omniphobic ceramic hollow fibre membrane with hierarchical re-entrant structure by growing hierarchical TiO_2 particles via a one-step synthesis of the hydrothermal method that is followed by fluorination. The morphology of the TiO_2, which has rod-like or flower-like structure, is controlled through hydrothermal reaction time. Moreover, the ceramic hollow fibre membrane, which is mullite, is synthesised from aluminosilicate ball clay. Its properties are controlled via the composition of ball clay suspension as well as the temperature of sintering.

1.3 Objective of Research

The main objective of this present study is to develop mullite hollow fibre membranes with omniphobic properties, where the substrate is made from naturally-occurring ball clay and the omniphobic surface is made from fluorinated (FAS-modified) TiO_2 particles, namely nanorods and hierarchical microflowers. To achieve this goal, the following objectives need to be accomplished.

i. To investigate the properties of mullite hollow fibre membrane by fabricating at different ball clay powder loading and sintering temperature in order to produce microfiltration membrane for MD application.

ii. To examine the omniphobicity of the mullite hollow fibre membranes through pre-roughening at different hydrothermal reaction time and post-fluorination at different grafting time.

iii. To evaluate the desalination performance, and antifouling and antiwetting properties of the omniphobic mullite hollow fibre membranes via DCMD process and subsequent surface characterization with a feed of saline solution that contains humic acid as a fouling agent.

1.4 Scopes of Study

The present study is conducted for facial fabrication of omniphobic mullite ceramic hollow fibre membranes as potential robust membranes with antiwetting and antifouling properties for MD. In order to achieve the objectives of this research, the following scopes are outlined

i. Ball clay powder was prepared via the sequence of pretreatment processes such as purifying, milling, drying, and calcination to reduce its particle size, narrowing its distribution size and forming anhydrous ball clay, respectively.

ii. Fabricating mullite hollow fibre membranes as substrates from ball clay through phase inversion-based extrusion followed by sintering process. Ceramic suspension containing ball clay powder in the range of 37.5 to 50 wt.% and sintering temperatures in the range of 1150˚C to 1300˚C were performed.

iii. Characterizing the physicochemical properties of the ball clay and the as-prepared mullite hollow fibre membranes such as particle size distribution, morphological, mineralogical, thermal and mechanical properties, water permeability as well as pore size and porosity. The physical properties of the membrane were evaluated to ensure that the membrane is suitable for MD application.

iv. Preparing omniphobic surfaces with re-entrant structures on the mullite hollow fibre membranes via pre-roughening by TiO_2 particles via hydrothermal method at different reaction times (0 to 10 hours) and post-flourinating with 1H,1H,2H,2H-Perfluorodecyltriethoxysilane (C8) at different grafting times (24 to 74 hours).

v. Characterizing the physicochemical properties of the omniphobic membranes in terms of morphologies, surface composition, surface roughness and pore size and porosity.

vi. The assessment of surface wettability with water and low surface tension liquids (humic acid, glycol, olive oil, etc.) via contact angle test.

vii. Performing integrity studies in terms of thermal, mechanical, and acid and base stability of the omniphobic mullite hollow fibre membranes prior to MD application.

viii. Evaluating the performances of the omniphobic ceramic hollow fibre membranes in terms of permeate flux, flux reduction factor, and salt rejection in DCMD process with feed of water and saline solution (3.5 wt%), respectively.

ix. Evaluating the antifouling and antiwetting properties through normalized flux, salt rejection, and humic acid rejection of the omniphobic membranes by adding humic acid (10 mg/L) as a fouling agent in saline solution (3.5 wt%).

x. Post-fouling analysis was carried out on fouled membranes to assess the surface morphology, chemistry, and omniphobicity.

1.5 Research Contribution

This study contributes a facile approach in terms of surface development of ceramic membranes towards omniphobicity that will benefit their antifouling and antiwetting properties during membrane distillation operations. Besides, this work aims to contribute to the growing area of MD research by exploring surface with re-entrant structures using particles with different geometries, particularly rod-like and hierarchical flower-like structures, which were grown in one-step through hydrothermal method, in which has not been reported so far in literatures. Moreover, this study would provide opportunities for mullite hollow fibre membranes fabricated from the abundantly available ball clay for membrane technology applications.

1.6 Thesis Organization

This thesis shows a facile approach to fabricating omniphobic ceramic hollow fibre membranes by constructing re-entrant structures (TiO_2 nanorods and microflowers) with a low surface energy material (C8) on the surface. The organization of this thesis is as follows.

Chapter One describes issues and gaps of current membrane distillation-related work. Three objectives are stated along with the project scopes that serve to accomplish the objectives. The benefits of this work are also presented. Chapter Two reviews the current literature related to water scarcity, advantages of MD, fouling and wetting in MD, as well as the fundamental design of an omniphobic surface. The review also includes recent progress in the fabrication technique of omniphobic membranes for MD. The benefits of using ceramic membranes are also reviewed in this chapter. Chapter Three describes the framework of the research methodology, which consists of step-by-step approaches that lead towards the goals of this project. The complete framework includes all materials, experimental setups, working procedures, and analytical methods required to fabricate ceramic hollow fibre membranes and omniphobic coatings. The characterization technique and membrane performance test of the DCMD system are also described in detail.

Results and discussion are elaborated in Chapter Four, Five, and Six. Chapter Four explains the fabrication, characterization, and permeation property of ceramic hollow fibre membranes fabricated from naturally-occurring ball clay. Firstly, the properties of ball clay powder in terms of thermal, morphological, mineralogical, and chemical properties before and after a sequence of pre-treatment processes were evaluated. The prepared ball clay powder was then subjected to membrane fabrication at different ball clay powder loading and sintering temperature. The physicochemical properties of the fabricated hollow fibre membrane are also discussed. In Chapter 5, the fabrication steps of omniphobic ceramic hollow fibre membrane are described. The morphologies and the distribution of TiO_2 particles on the surface of hollow fibres were investigated in detail by controlling the hydrothermal reaction times and grafting times. The surface chemistry, surface wettability, coating integrity, pore size, and porosity of the omniphobic ceramic hollow fibre membranes were also evaluated. In Chapter 6, membrane distillation studies and subsequent membrane surface characterizations were carried out. Organic fouling on the omniphobic membranes was evaluated using DCMD test with feed of saline solution, containing humic acid as a fouling agent. Post-fouling analysis on the fouled membranes, in terms of morphology, chemistry, and omniphobicity, is discussed in detail. Finally, Chapter 7 provides general conclusions of the study as well as some recommendations for future work to fill the knowledge gaps.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Mohd Haiqal Abd Aziz**, Mohd Hafiz Dzarfan Othman, Nur Hashimah Alias, Tomonobu Nakayama, Yoshitaka Shingaya, N. Awanis Hashim, Tonni Agustion, Kurniawan, Takeshi Matsuura, Mukhlis A.Rahman, Juhana Jaafar. (2020). Enhanced omniphobicity of mullite hollow fibre membrane with organosilane-functionalized TiO_2 microflowers and nanorods layer deposition for desalination using direct contact membrane distillation. Journal of Membrane Science, Vol 607, 118137. (**Q1, IF: 7.015**)
2. **Mohd Haiqal Abd Aziz**, Mohd Hafiz Dzarfan Othman, Nur Awanis Hashim, Mohd Ridhwan Adam, Azeman Mustafa. (2019). Fabrication and characterization of mullite ceramic hollow fibre membrane from natural occurring ball clay. Applied Clay Science, Vol 177, 51-62. (**Q1, IF: 4.605**)