POLYVINYLIDENE FLUORIDE-BASED DUAL-LAYER HOLLOW FIBRE NANOCOMPOSITE MEMBRANES FOR MEMBRANE DISTILLATION

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DEDICATION

This thesis is dedicated to my parents, who taught me that education and soft skills are the most powerful tools in this world and have always motivated me to be who I am today.

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ABSTRACT

Membrane distillation (MD) is a technology that makes use of the temperature difference created across the membrane. One of the main challenges in MD research is to fabricate membranes with high wetting resistance and high permeate flux. To date, very limited work has reported on the use of both hydrophobised carbon-based nanoparticles and hydrophobicity gradient in achieving those qualities. Thus, this work was focused on the fabrication of dual-layer hollow fibre polyvinylidene fluoride-based nanaocomposite membranes with hydrophobic gradient. The nanocomposite membranes were incorporated with hydrophobised nanoparticles, i.e. multi-walled carbon nanotube (MWCNT) and graphene nanoplatelet (GNP). The effect of different concentrations of nanoparticles (1 and 2 wt%) on the performance of the membranes was investigated as well. The nanoparticles were oxidised with concentrated nitric acid and sulphuric acid, hydrophobised with 1H,1H,2H,2H-perfluorodecyltriethoxysilane and characterised to confirm the hydrophobisation and determine their specific surface area. The hydrophobicity gradient was created by introducing the hydrophobised nanoparticles in the outer layer dope solution and hydrophilic polyethylene glycol in the inner layer dope solution. The dual-layer membranes were then fabricated and characterised to determine their wetting resistance, chemical composition, surface and cross-sectional morphology, pore size, porosity and contact angle before their performances were tested in a direct contact membrane distillation (DCMD) set-up. MWCNT and GNP were successfully oxidised and hydrophobised as confirmed by energy-dispersive X-ray spectroscopy (EDX) and Fourier transform infrared spectroscopy (FTIR) results. Both the hydrophobised nanoparticles had smaller Brunauer-Emmett-Teller (BET) specific surface area as compared to the pristine nanoparticles. All the fabricated membranes had an asymmetrical structure with finger-like and sponge-like pores in the inner and outer layers respectively. The outer layers of the nanocomposites membranes were hydrophobic and the membrane with 2 wt% of hydrophobised GNP had the highest contact angle of 111.1°. All the inner layers of the membranes were found to be hydrophilic, thus proving that hydrophobicity gradients were achieved in the membranes. The surface roughness, contact angle and wetting resistance of all the nanocomposite membranes were higher than the neat membrane and the values increased with increasing concentration of nanoparticles. Membranes incorporated with GNP showed better performance in terms of contact angle and wetting resistance. All the nanocomposite membranes showed better DCMD performance than the neat membrane. The membrane incorporated with 2 wt% of hyrophobised GNP achieved the highest flux of 8.27 kg/(m^2h) at feed temperature of 80°C. All the membranes achieved a salt rejection of more than 99%. Nonetheless, the flux of the membranes were quite low compared to the flux of membranes reported in the literature, likely due to small pore sizes and dense interfaces.

ABSTRAK

Penyulingan membran (MD) adalah teknologi yang menggunakan perbezaan suhu yang dihasilkan merentasi membran. Salah satu cabaran utama dalam penyelidikan MD adalah untuk mendapatkan membran dengan rintangan pembasahan yang tinggi dan fluks penelapan yang tinggi. Sehingga kini, dilaporkan kajian terhadap penggunaan nanopartikel hidrofobik berasaskan karbon dan kecerunan hidrofobik untuk mendapatkan kualiti-kualiti tersebut adalah sangat terhad. Oleh itu, penyelidikan ini telah ditumpukan terhadap penghasilan membran nanokomposit dwi-lapisan geronggang gentian polivinilidena florida yang mempunyai kecerunan hidrofobik. Membran nanokomposit digabungkan dengan nanopartikel hidrofobik, iaitu tiub nano karbon berbilang dinding (MWCNT) dan nanoplatelet grafena (GNP). Kesan kepekatan nanopartikel yang berbeza (1 dan 2% berat) terhadap prestasi membran juga dikaji. Nanopartikel dioksidakan dengan asid nitrik pekat dan asid sulfurik, dihidrofobikkan dengan 1H, 1H, 2H, 2H-perfluorodekiltrietoksisilana dan dicirikan untuk mengesahkan hidrofobisasi dan menentukan luas permukaan spesifiknya. Kecerunan hidrofobik telah diwujudkan dengan memperkenalkan nanopartikel hidrofobik dalam larutan dop lapisan luar dan polietilena glikol hidrofilik dalam larutan dop lapisan dalam. Membran-membran dua lapisan kemudian telah dihasilkan dan dicirikan untuk menentukan rintangan pembasahannya, komposisi kimia, morfologi permukaan dan keratan rentas, saiz liang, keliangan dan sudut sentuhan sebelum prestasinya diuji dalam susunan penyulingan membran sentuhan langsung (DCMD). MWCNT dan GNP berjaya dioksidasi dan dihidrofobikkan seperti yang disahkan oleh keputusan analisis spektroskopi penyebaran tenaga sinar X-ray (EDX) dan spektroskopi inframerah jelmaan Fourier (FTIR). Kedua-dua nanopartikel hidrofobik mempunyai luas permukaan Brunauer-Emmett-Teller (BET) yang lebih kecil berbanding dengan nanopartikel asli. Semua membran mempunyai struktur tidak bersimetri dengan liang seperti jari dan span di lapisan dalam dan luar. Lapisan luar membran nanokomposit adalah hidrofobik dan membran dengan 2% berat GNP hidrofobik mempunyai sudut sentuhan tertinggi iaitu 111.1°. Semua lapisan dalam membran didapati hidrofilik, maka membuktikan bahawa kecerunan hidrofobik dicapai dalam membran. Kekasaran permukaan, sudut sentuhan dan rintangan pembasahan semua membran nanokomposit lebih tinggi daripada membran asli dan nilainya meningkat apabila kepekatan nanopartikel meningkat. Membran yang digabungkan dengan GNP menunjukkan prestasi yang lebih baik dari sudut sentuhan dan rintangan pembasahan. Semua membran nanokomposit menunjukkan prestasi DCMD yang lebih baik daripada membran asli. Membran yang digabungkan dengan 2% berat GNP yang dihidrofobikkan mencapai fluks tertinggi 8.27 kg /(m^2j) pada suhu 80°C. Semua membran mencapai penyingkiran garam lebih daripada 99.9%. Walaupun begitu, fluks membran agak rendah berbanding dengan fluks membran yang pernah dilaporkan dalam literatur, mungkin disebabkan oleh saiz liang kecil dan antara muka yang padat.

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LIST OF ABBREVIATIONS

AC - Activated carbon
AGMD - Air gap membrane distillation
ATM - Atomic force microscopy
BET - Brunauer-Emmett-Teller
BP - Bucky paper
CNIM - Carbon nanotube immobilized membrane
CNT - Carbon nanotube
DCMD - Direct contact membrane distillation
DMAc - Dimethylacetamide
DMF - N,N-dimethylformamide
EIPS - Evaporation-induced phase separation
FAS - Fluoroalkylsilane
FTES - 1H, 1H, 2H, 2H-perfluorooctltriethoxysilane
FTIR-ATR - Fourier transform infrared spectroscopy-attenuated total reflectance
F-POSS - Fluorinated-decyl polyhedraloligomeric silsesquioxane
FTCS - 1H, 1H, 2H, 2H-perfluorododecyltrichlorosilane
IPA - Isopropyl alcohol
GNP - Graphene nanoplatelet
GO - Graphene oxide
LEP - Liquid Entry Pressure
LGMD - Liquid gap membrane distillation
MD - Membrane distillation
MED - Multiple effect distillation
MEMD - Multi-effect membrane distillation
MGMD - Material gap membrane distillation
MIP - Mercury intrusion porosimetry
MMM - Mixed-matrix-membrane
MSF - Multi-stage flash distillation
MWCNT - Multi-walled carbon nanotube

NIPS	-	Non-solvent induced phase separation
NF	-	Nanofiltration
OMD	-	Osmotic membrane distillation
PAN	-	Polyacrylonitrile
PDA	-	Poly-dopamine
PDMS	-	Polydimethylsiloxane
PDTS	-	1H,1H,H,2H-perfluorodecyltriethoxysilane
PE	-	Polyethylene
PES	-	Polyethersulfone
PET	-	Polyethylene terephthalate
PFDTES	-	1H,1H,2H,2H-Perfluorodecyltriethoxysilane
PFTS	-	1H, 1H, 2H, 2H-perfluorooctyltriethoxysilane
PGMD	-	Permeate gap membrane distillation
PH	-	Poly(vinylidene fluoride-*co*-hexafluoropropylene)
PP	-	Polypropylene
PSF	-	Polysulfone
PTFE	-	Polytetrafluoroethylene
PVC	-	Polyvinyl chloride
PVDF	-	Polyvinylidene fluoride
RO	-	Reverse osmosis
SEM	-	Scanning electron microscopy
SGMD	-	Sweeping gas membrane distillation
TEP	-	Triethyl phosphate
TIPS	-	Thermally-induced phase separation
TGA	-	Thermogravimetric analysis
THF	-	Tetrahydrofuran
VIPS	-	Vapour-induced phase separation
VMD	-	Vacuum membrane distillation
V-MEMD	-	Vacuum-multi-effect membrane distillation
XRF	-	X-ray fluorescence
ZLD	-	Zero liquid discharge

LIST OF SYMBOLS

A - Surface area of membrane
B - Dimensionless geometric factor
C_f - Concentration of salt in the feed
C_p - Concentration of salt in the permeate
J - Permeate flux
m - Mass of water collected
N - Molar flux
r - Surface roughness factor
r_{max} - Maximum pore radius of membrane
$<r^{\alpha}>$ - Average pore radius for Knudsen diffusion
R - Surface roughness factor
t - Operation time
T_f - Feed temperature
T_p - Permeate temperature
T_1 - Feed temperature on themembrane surface
T_2 - Permeate temperature on the membrane surface
γ_{lv} - Liquid/vapour interfacial tension
γ_{sl} - Solid/liquid interfacial tension
γ_{sv} - Solid/vapour interfacial tension
γ_L - Liquid surface tension
δ - Membrane thickness
ε - Membrane porosity
τ - Membrane tortuosity

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CHAPTER 1

INTRODUCTION

1.1 Background

Water scarcity is a pressing predicament that is afflicting the humankind worldwide as shown in Figure 1.1. Around 6 billion people are expected to face the consequences of water shortage by 2050 (Boretti and Rosa, 2019). Fresh water makes up 3% of the hydrosphere and only 30% of it is available for drinking. The remaining 70% remains inaccessible in the forms of ice caps, glaciers and deep underground water (Sarah *et al.*, 2019; Khawaji, Kutubkhanah and Wie, 2008).

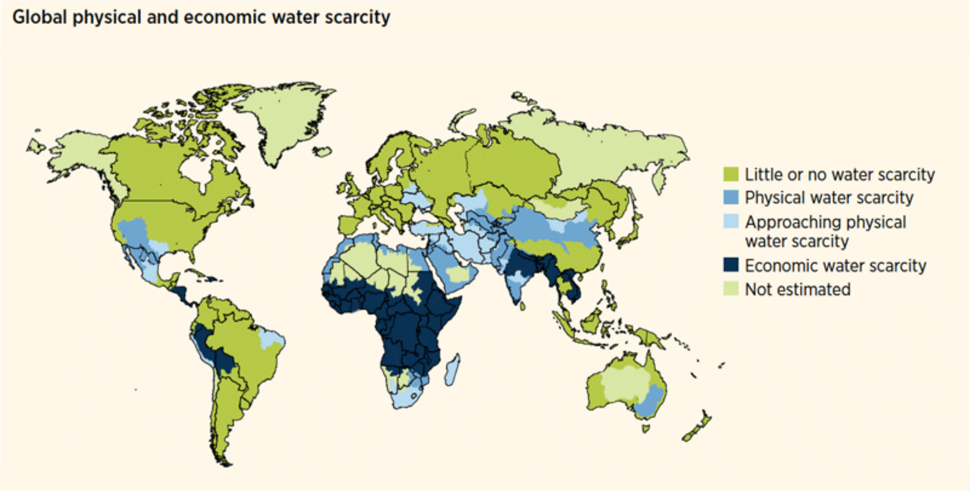

Figure 1.1 Water scarcity around the world ("International Decade for Action", 2014)

Rapid industrialisation and urbanisation in addition to the climate change and burgeoning world's population have irrefutably diminished the fresh water availability and escalated demand for fresh water around the world (Teoh and Mohammad, 2019). Unfortunately, the problem is exacerbated also by the acts of unscrupulous people who pollute water sources through illegal activities such as discharge of chemical waste into the rivers. In order to tackle this challenge, better management of water demand alone is not sufficient; a larger supply of water is pertinent too (Gude *et al.*, 2017). Scientists and engineers have been looking into unconventional water resources such as seawater, industrial waste water and municipal brine (Chen *et al.*, 2017). In that regards, seawater desalination is seen as a very feasible option due to the ubiquity of seawater around us. Desalination refers to the removal of salt from seawater to produce fresh water suitable for our daily use. As of now, about 15906 desalination plants operate worldwide with almost half of the desalination capacity concentrated in the Middle-East and North Africa regions (Jones *et al.*, 2019).

Over the years, various technologies have been employed for desalination use including multi-stage flash distillation (MSF), multiple-effect distillation (MED), reverse osmosis (RO), nanofiltration (NF) and membrane distillation (MD) (Pinto and Marques, 2017). In the course of time, the dominance of thermal-based technologies (MSF and MED) has been overtaken by membrane-based technologies (RO and NF), fueled by the increasing awareness of saving energy. As of now, RO accounts for about 69% of the technology used in desalination plants worldwide (Ahmed, Hashaikeh and Hilal, 2019; Jones *et al.*, 2019). Despite the success of the aforementioned desalination technologies for decades, they come with drawbacks such as high energy requirement which have limited their worldwide use (Pinto and Marques, 2017). Scientists are now focusing efforts on making these technologies more efficient and inexpensive.

One of the relatively newer technologies, still in its research and development stage, is membrane distillation (MD) patented by Bodell in 1963 (Bodell, 1963). MD utilises a microporous membrane which acts as a physical barrier through which water vapour passes through, driven by vapour pressure gradient. The water vapour then condenses on the cold permeate side forming the permeate flux (Deshmukh *et al.*, 2018). This technology has garnered much interest recently because its plant is operable using renewable and low-grade energies such as solar energy and waste heat from factories respectively, occupies a small space and its operation is independent of the feed solution concentration (Gullinkala *et al.*, 2010; Voutchkov, 2018). Many MD configurations have been invented in the course of time, however there are a few common configurations of MD, namely direct contact, vacuum, sweeping membrane and air gap MD. The world's inaugural seawater MD plant was opened in Maldives by a Netherlands-based company called Aquaver in 2014 (Drioli, Ali and Macedonio, 2015).

An ideal membrane for MD is expected to have features such as high wetting resistance, high permeability, low fouling rate, low thermal conductivity and excellent chemical stability (Alkhudhiri, Darwish and Hilal, 2012). Membrane scientists have been trying to introduce the desired features to the membrane by modifying the type of material, structure, morphology and composition of the membrane, among others. Polymeric and ceramic membranes have attracted a lot of attention as the materials with high suitability for MD applications (Wang and Chung, 2015). Although ceramic membrane has been appreciated for its high tolerance to harsh conditions, its high cost has rendered it less favourable commercially. Polymeric membrane is seen as a plausible cheaper alternative, but it comes with some drawbacks such as low chemical and thermal stability which need to be addressed (Hubadillah *et al.*, 2018).

Some of the polymeric membranes that have been studied for MD include polyvinylidene fluoride (PVDF), polytetrafluoroethylene (PTFE), polypropylene (PP) and polydimethylsiloxane (PDMS) (Thomas *et al.*, 2017). Membranes made from these polymers have been fabricated in either flat, hollow fibre or electrospun forms (Eykens *et al.*, 2017). The recent trend in the field of MD is the fabrication of polymeric composite and nanocomposite membranes having superior properties to the unmodified counterparts. Dual-layer composite membranes have been highly favoured as the two layers consisting of different materials or structures have different properties and serve different functions. The dual-layer membranes can be produced in a single step either by co-casting or co-extrusion (Xia *et al.*, 2018).

The addition of inorganic nanoparticles such as hydrophobised silicon dioxide (SiO_2) and titanium dioxide (TiO_2) and carbon-based nanoparticles such as multi-walled carbon nanotube (MWCNT), activated carbon (AC), graphene and its derivatives such as graphene nanoplatelet (GNP) and graphene oxide (GO) are proven to enhance the performance of polymeric membranes in MD (Drioli, Ali and Macedonio, 2015). Like inorganic nanoparticles, carbon-based nanoparticles are sometimes hydrophobised too to enhance their hydrophobicity and thus enhance the wetting resistance of MD membranes. Functional groups such as hydroxyl groups are usually introduced to them by oxidation before reacting them with fluoroalkylsilane agents (An *et al*., 2017b). Generally, these nanoparticles, in its raw or modified forms, can augment the flux, hydrophobicity and mechanical strength of polymeric membranes (Goh, Ismail and Ng, 2013).

Tremendous research efforts that have been devoted to MD in the past five decades have definitely accelerated the progress of this desalination technology. The large-scale commercialisation of MD is, however, a milestone yet to be achieved mainly due to the lack of high-performance membranes. Hence, membranes of very high quality, nearing the properties of an ideal membrane, will very much fuel the growth of this technology towards that milestone.

1.2 Problem Statements

In pursuit of a high wetting resistance, high hydrophobicity has been a highly sought-after quality in membranes especially in commercially-attractive hollow fiber membranes (Teoh, Chung and Yeo, 2011; Tang *et al.*, 2012). Coating hollow fibre membranes with low surface energy material such as flouroalkylsilanes may not be the best way to achieve high hydrophobicity as poor adhesion between coating solution and the polymer may give rise to delamination (Wirasate and Boerio, 2005). Alternatively, grafting or introducing grafted nanoparticles into the membrane saves the membrane from delamination issues. Very limited work has been done on grafting carbon-based nanoparticles for MD purpose. However, none of these works has been focused on the hydrophobisation of graphene or its derivatives.

Besides that, a major hitch in the commercialisation of MD is the low permeate flux associated with the membranes (Alkhudhiri, Darwish and Hilal, 2012). An ingenious approach to increase the flux is to introduce a hydrophobicity gradient into the membranes. A hydrophilic or less hydrophobic inner layer in a dual-layer membrane will allow the inner layer to be partially filled with permeate water which reduces the path length of water vapour. A reduction in the length that the water vapour has to travel through translates to a lower mass transfer resistance (Khayet *et al.*, 2005). The coupling of the aforementioned high hydrophobicity with this hydrophobicity gradient will be a stepping stone to achieving both high wetting resistance and high permeability. Nevertheless, the challenge lies in introducing high hydrophobicity only to the outer layer while maintaining the hydrophilicity or low hydrophobicity in the inner layer. In light of the thin outer layer of the dual-layer membranes, subjecting a dual-layer membrane to grafting solutions can let the solution penetrate into the inner layer which can enhance the hydrophobicity of the inner layer too. A solution to this problem is to incorporate grafted hydrophobic nanoparticles only in the outer layer of the membrane. To date, no work has been done on incorporating hydrophobised nanoparticles into dual-layer hollow fibre membranes fabricated via phase-inversion-based co-extrusion process.

Many cutting-edge studies on MD have employed carbon-based nanoparticles in order to achieve desired qualities. The capabilities of MWCNT and graphene-based materials to enhance water flux in MD via ways that inorganic nanoparticles are incapable of such as absorption-desorption have been reported (Woo *et al.*, 2016; Bhadra, Roy and Mitra, 2016b). Many of these works are focused on a single nanoparticle. The performances of two carbon-based nanoparticles in a dual-layer membrane have never been compared on the same grounds. Research on comparing the performances of these carbon-based nanoparticles on the same membrane will be able to point out the relative strengths and weaknesses of the nanoparticles and may provide a new research direction to the nano-enabled technology in MD.

Therefore, to address the research gaps aforementioned, this research was focused on the fabrication of dual-layer hollow fibre membranes with hydrophobicity gradients containing hydrophobised MWCNT or hydrophobised graphene nanoplatelet (GNP) in the outer layer as shown in Figure 1.2. Polyethylene glycol (PEG) was introduced in the inner layer to increase hydrophilicity and induce the formation of finger-like pores. Finger-like pores will allow the influx of more water into the inner layer. MWCNT was chosen as it is cheaper than other carbon-based nanoparticles such as AC while GNP was chosen because it can be more easily mass produced than graphene (Cataldi, Athanassiou and Bayer, 2018). Also, GNP is multi-layered like MWCNT and does not contain any other atom except carbon, unlike GO which contains oxygen, enabling fairer comparison with MWCNT.

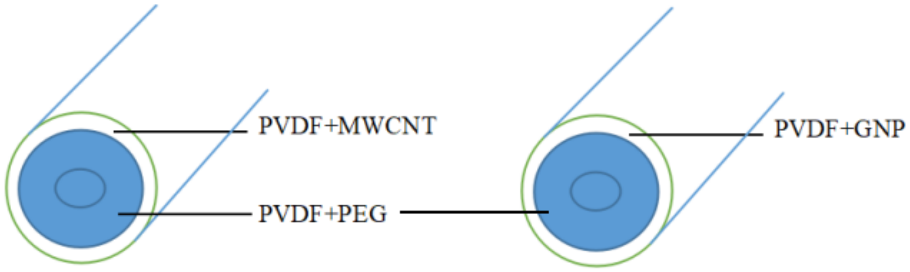

Figure 1.2 Target membranes

1.3 Objectives

The main objective of this research was to fabricate PVDF-based dual-layer hollow fibre nanocomposite membranes with hydrophobicity gradients, incorporating PEG and hydrophobised MWCNT or GNP. The specific research objectives were as follows:

1. To characterise the oxidised and hydrophobised MWCNT and GNP
2. To investigate the effect of different concentrations of MWCNT and GNP on the properties of the dual-layer membrane by characterisations of the membrane
3. To evaluate the direct contact membrane distillation (DCMD) performances of the dual-layer membranes incorporated with different concentrations of MWCNT and GNP

1.4 Scope of Study

The following scopes of study were identified to achieve the outlined objectives:

1) To characterise the oxidised and hydrophobised MWCNT and GNP

a) Oxidation of MWCNT and GNP

i- Oxidising using concentrated H_2SO_4 (95-97%) and HNO_3 (65%) mixture
ii- Confirming oxidation using Energy-dispersive X-ray spectroscopy (EDX) and Fourier Transform Infrared Spectroscopy (FTIR)

b) Hydrophobisation of MWCNT and GNP

i- Hydrophobising with 3 wt% of 1H,1H,2H,2H-perfluorodecyltriethoxysilane (PFDTES)
ii- Confirming hydrophobisation using EDX and FTIR and determining specific surface area using Brunauer-Emmett-Teller (BET) method

2) To investigate the effect of different concentrations of MWCNT and GNP on the properties of the dual-layer membrane by characterisations of the membrane

c) Fabrication of dual-layer hollow fiber membranes

i- Preparing two dope solutions for each spin-
    i) inner layer dope composition: PVDF/NMP/PEG
    ii) outer layer dope composition: PVDF/NMP/MWCNT or GNP
ii- Varying loading concentration of both MWCNT and GNP at 0, 1 and 2 wt%

d) Characterisation of membranes

i- Studying membrane's surface and cross-sectional morphology by scanning electron microscopy (SEM) and atomic force microscopy (AFM)
ii- Measuring hydrophobicity and hydrophilicity by water contact angle analysis

iii- Measuring pore size, porosity and tortuosity by mercury intrusion porosimetry (MIP)
iv- Measuring wetting resistance by measuring liquid entry pressure (LEP)
v- Studying chemical composition by EDX analysis

3) To evaluate the direct contact membrane distillation (DCMD) performances of the dual-layer membranes incorporated with different concentrations of MWCNT and GNP

e) DCMD performance

i- Preparing modules of 17.5 cm length containing 4 membranes
ii- Testing with synthetic seawater: 3.5 wt.% NaCl for 1.5 hours
iii- Setting temperature of the feed and permeate solutions at 80 and 10 °C respectively and feed and permeate flow rates at 50 L/hr

1.5 Significance of Research

Although the ideal properties of a membrane suitable for MD have been thoroughly understood, the membranes fabricated thus far are still quite far from the status of high-performing membranes. Numerous methods are being used and improvised by scientists to attain the desired qualities. Thus, this study has contributed to the deeper understanding of some of those methods besides exploring areas which have the potential to create breakthroughs in MD. Also, the number of water-stressed regions in Malaysia has been augmenting at an alarming rate especially near densely populated areas. As water is so important for numerous reasons including human consumption, industrial use and agriculture, Malaysia will benefit greatly from a steady supply of fresh water all year round. This research could contribute to the advancement of membrane distillation technology which has the potential to translate into Malaysia's very first cost-effective large-scale desalination plant.

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LIST OF PUBLICATIONS

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1) **Ravi, J.**, Othman, M. H. D., Matsuura, T., Ro'il Bilad, M., El-badawy, T. H., Aziz, F., Ismail, A. F., Rahman, M. A., & Jaafar, J. (2020). Polymeric membranes for desalination using membrane distillation: A review. In *Desalination* (Vol. 490). Elsevier B.V. **(Q1, IF= 7.098)**