POLYETHYLENEIMINE INTERLAYERED THIN FILM NANOCOMPOSITE REVERSE OSMOSIS MEMBRANE FOR IMPROVED WATER DESALINATION

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ABSTRACT

Water desalination is the most effective strategy in dealing with global water crisis. However, the current thin film composite (TFC) reverse osmosis (RO) membranes which dominate the desalination process are still susceptible to permeability and selectivity trade-off, fouling and chlorine attack. These issues were resolved in this work by optimizing the synthesis conditions of interfacial polymerization (IP) technique for TFC membrane fabrication followed by adopting interlayer-assisted IP technique and graphene oxide (GO) incorporation for the fabrication of thin film nanocomposite (TFN) membrane. Polyethyleneimine (PEI) was used as interlayer for TFN membrane fabrication to avoid defects caused by GO incorporation in the polyamide (PA) layer. The effects of post IP rinsing on the TFC membrane were first investigated prior to the TFN membrane fabrication. It was found that the rinsing solution properties such as boiling point, surface tension and miscibility could affect the efficiency of unreacted monomers removal, altering the physicochemical properties of PA layer and yielding reproducible TFC membrane with higher water flux and least deteriorated salt rejection. Aqueous solution rinsing was found to be able to enhance membrane pure water flux (PWF) from 17.53 to 22.56 $L/m^2 \cdot h$ at 15 bar without significantly trading off its promising sodium chloride (NaCl) rejection (97.70%) when compared to the control membrane and organic solvent-rinsed membrane. For the TFN membrane, the presence of PEI interlayer was found to improve the distribution and orientation of GO in the PA layer which minimized the defects formed. Compared to the typical TFN membrane fabricated using conventional IP, the PEI-interlayered TFN membranes containing the same amount of GO (0.015 wt/v%) were found to exhibit a relatively thinner but rougher PA. As a result, almost all PEI-interlayered TFN membrane exhibited better desalination performances than the typical TFN membrane. It was also discovered that the substrate of membrane coated with a single layer of 0.05 wt/v% PEI followed by 60-min drying produced promising TFN membrane (i.e., iTFN-C0.05-T60-L1), achieving 96.66% NaCl rejection and 2.24 $L/m^2 \cdot h \cdot bar$ PWF. The experimental results also revealed that the use of optimum GO loading (0.01 wt/v% GO) in the PA layer fabricated via interlayer-assisted IP could further improve TFN membrane performance, leading to the highest PWF (2.66 $L/m^2 \cdot h \cdot bar$) achieved without compromising NaCl rejection (~97.5%). This was caused by the improved membrane surface hydrophilicity and roughness paired with the nanochannels created by GO. The optimized TFN membrane also showed improved resistivity against alginate and least deteriorated desalination property after chlorination. Although the antibacterial property of GO was hindered by the PA layer, the membrane still exhibited better antibacterial property than that of commercial RO membrane. The outcomes of this study suggested that properly arranged GO in PA layer is necessary to minimize the formation of defects that could be detrimental for membrane separation. The position of GO in PA layer is particularly important to optimize its functionality. As a conclusion, the PEI-interlayered TFN membrane fabricated in this study portrayed a great potential in addressing the drawbacks of commercial TFC membrane for seawater or brackish water desalination.

ABSTRAK

Penyahgaraman air adalah strategi yang paling efektif dalam menangani krisis air global. Namun, membran osmosis berbalik (RO) komposit filem nipis (TFC) yang mendominasi proses penyahgaraman kini masih terdedah kepada keseimbangan antara kebolehtelapan dan kememilihan, pengotoran dan serangan klorin. Masalah-masalah ini diselesaikan dalam kajian ini dengan mengoptimumkan keadaan teknik sintesis pempolimeran antaramuka (IP) untuk pembuatan membran TFC diikuti dengan menggunakan teknik antara lapisan berbantu IP dan penggabungan grafin oksida (GO) untuk fabrikasi membran komposit nano filem nipis (TFN). Polietilenaimina (PEI) digunakan sebagai lapisan antara untuk fabrikasi membran TFN bagi mengelakkan ketidaksempurnaan yang disebabkan oleh penggabungan GO dengan lapisan poliamida (PA). Kesan pembilasan selepas IP pada membran TFC diselidiki terlebih dahulu sebelum fabrikasi membran TFN. Didapati bahawa sifat larutan bilas seperti suhu didih, tegangan permukaan dan kebolehcampuran boleh menpengaruhi kecekapan penyingkiran monomer yang tidak bertindak balas, mengubah sifat fizikokimia lapisan PA dan menghasilkan membran TFC yang boleh dihasilkan semula dengan fluks air yang lebih tinggi dan penolakan garam yang paling sedikit merosot. Pembilas larutan akueus didapati mampu meningkatkan PWF membran dari 17.53 hingga 22.56 $L/m^2 \cdot h$ pada 15 bar tanpa mengubah penolakan natrium klorida (NaCl) dengan ketara (97.70%) jika dibandingkan dengan membran kawalan dan membran yang dibilas oleh pelarut organik. Untuk membran TFN, kehadiran lapisan antara PEI didapati meningkatkan penyebaran dan orientasi GO dalam lapisan PA dan meminimumkan pembentukan ketidaksempurnaan. Dibandingkan dengan membran TFN biasa yang dibuat dengan menggunakan IP konvensional, membran TFN dengan lapisan antara PEI dengan kuantiti GO yang sama (0.015 wt/v%) mempunyai PA yang lebih nipis tetapi kasar. Hasilnya, hampir semua membran TFN dengan lapisan antara PEI menunjukkan prestasi penyahgaraman yang lebih baik daripada membran TFN biasa. Didapati bahawa substratum membran yang dimodifikasi dengan lapisan tunggal PEI sebanyak 0.05 wt/v% diikuti pengeringan 60-minit menghasilkan membran TFN (iaitu iTFN-C0.05-T60-L1) dengan prestasi penyahgaraman terbaik (96.66% penolakan NaCl dan 2.24 $L/m^2 \cdot h \cdot bar$). Hasil eksperimen juga menunjukkan bahawa penambahan GO yang optimum (0.01 wt/v% GO) dalam lapisan PA yang dibuat dengan teknik antara lapisan berbantu IP dapat menambahbaik prestasi membran TFN, memberi peningkatan dalam PWF (2.66 $L/m^2 \cdot h \cdot bar$), tanpa menjejaskan penolakan NaCl (~97.5%). Ini disebabkan oleh peningkatan kehidrofilikan dan kekasaran permukaan membran disamping saluran nano yang dibuat oleh GO. Membran TFN yang dioptimumkan ini juga menunjukkan ketahanan yang lebih baik terhadap alginat dan kemerosotan sifat penyahgaraman yang sedikit selepas pengklorinan. Walaupun sifat antibakteria GO dihalang oleh lapisan PA, membran ini masih menunjukkan sifat antibakteria yang lebih baik apabila dibandingkan dengan membran RO kelas komersial. Kajian ini menunjukkan bahawa GO yang disusun dengan betul dalam lapisan PA diperlukan untuk meminimumkan pembentukan ketidaksempurnaan yang boleh memudaratkan prestasi penyahgaraman membran. Kedudukan GO dalam lapisan PA penting untuk mengoptimumkan fungsinya. Sebagai kesimpulan, membran TFN dengan lapisan antara PEI yang dibuat dalam kajian ini mempunyai potensi besar dalam mengatasi kekurangan membran TFC komersial untuk penyahgaraman air laut atau air payau.

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LIST OF ABBREVIATIONS

2D	-	two dimensional
3D	-	three dimensional
AAPTS	-	1-(2-amino-ethyl)-3-aminopropyl] trimethoxysilane
AFM	-	atomic force microscopy
Ag	-	silver
ALD	-	atomic layer deposition
ATR	-	attenuated total reflectance
$BaCl_2.2H_2O$	-	barium chloride 2-hydrate
$BaSO_4$	-	barium sulfate
BN	-	boron nitride
BSA	-	bovine serum albumin
B. subtilis	-	*Bacillus subtilis*
BTCE	-	biphenyl acid chloride
CA	-	cellulose acetate
CD	-	carbon dot
CDA	-	cellulose diacetate
CeO_2	-	cerium oxide
CFIC	-	5-chloroformyloxy-isophthaloyl chloride
ClO_2	-	chlorine dioxide
CN	-	carbon nitride
CNC	-	cellulose nanocrystal
CNT	-	carbon nanotube
COF	-	carbon organic framework
CP	-	concentration polarization
CTA	-	cellulose triacetate
Cu	-	copper
CuO	-	copper oxide
DABA	-	triamine 3,5-diamino-N-(4-aminophenyl) benzamide
DI	-	deionized
DNA	-	deoxyribonucleic acid

E. coli	-	*Escherichia Coli*
ED	-	Electrodialysis
EDA	-	ethylenediamine
EDX	-	elemental dispersive X-ray
EG	-	ethylene glycol
FAPA	-	fully aromatic polyamide
FESEM	-	field emission scanning electron microscopy
FO	-	forward osmosis
FTIR	-	Fourier transform infrared spectroscopy
GDP	-	gross domestic product
GO	-	graphene oxide
GQD	-	graphene quantum dot
HCl	-	hydrochloric acid
HDH	-	Humidification-dehumidification
HEMA	-	2-hydroxyethyl methacrylate
HFBA	-	2,2,3,4,4,4-hexafluorobutylacrylate
HNO_3	-	nitric acid
HNT	-	halloysite nanotube
H_2O_2	-	hydrogen peroxide
hPAN	-	hydrolyzed polyacrylonitrile
H_2SO_4	-	sulfuric acid
HSO_4^-	-	hydrogen sulfate
ICIC	-	5-isocyanato-isophthaloyl chloride
IP	-	interfacial polymerization
IPC	-	isophthaloyl chloride
KBr	-	potassium bromide
KCl	-	potassium chloride
$KClO_3$	-	potassium chlorate
KH_2PO_4	-	potassium dihydrogen phosphate
$KMnO_4$	-	potassium permanganate
LBL	-	layer-by-layer
LDH	-	layered double hydroxide
MED	-	multi-effect distillation

MF	-	microfiltration
$MgCl_2$	-	magnesium chloride
MMT	-	montmorillonite
Mn_2O_7	-	dimanganese heptoxide
mmBTEC	-	3,3',5,5'-biphenyl tetraacyl chloride
mNT	-	modified nanoporous titanate
MOF	-	metal organic framework
MoS_2	-	molybdenum disulfide
MPD	-	m-phenylenediamine
MPIA	-	polymetaphenylene isophthalamide
MPTA	-	polymetaphenylene trimesamide
MSF	-	multi-stage flash distillation
MW	-	molecular weight
MWCNT	-	multiwalled carbon nanotube
MWCO	-	molecular weight cut-off
NaAlg	-	sodium alginate
NaCl	-	sodium chloride
Na_2CO_3	-	sodium carbonate
Na_2HPO_4	-	disodium hydrogen phosphate
$NaNO_3$	-	sodium nitrate
NaOCl	-	sodium hypochlorite
NaOH	-	sodium hydroxide
NF	-	nanofiltration
NO_2	-	nitrogen dioxide
NO_3^-	-	nitrate
N_2O_4	-	dinitrogen tetroxide
OPD	-	o-phenylenediamine
OSN	-	organic solvent nanofiltration
PA	-	polyamide
PAA	-	polyacrylic acid
P.aeruginosa	-	*Pseudomonas aeruginosa*
Pal	-	palygorskite
PALS	-	Positron Annihilation Spectroscopy

PBS	-	phosphate-buffered saline
PDA	-	polydopamine
PE	-	polyelectrolyte
PEG	-	polyethylene glycol
PEI	-	polyethyleneimine
PES	-	polyethersulfone
PET	-	polyester
PFDTES	-	1H,1H,2H,2H-perfluorodecyltriethoxysilane
PFM	-	Pore Flow model
PI	-	polyimide
PIP	-	piperazine
PPD	-	p-phenylenediamine
P. putida	-	*Pseudomonas putida*
PSf	-	polysulfone
PV	-	pervaporation
PVA	-	polyvinyl alcohol
PVDF	-	polyvinylidene fluoride
PWF	-	pure water flux
PWP	-	pure water permeability
RNA	-	ribonucleic acid
RO	-	reverse osmosis
ROS	-	reactive oxygen species
SANS	-	Small-Angle Neutron Scattering
S. aureus	-	*Staphylococcus aureus*
SD	-	solar distillation
SDM	-	Solution-Diffusion model
SEM	-	scanning electron microscopy
SKM	-	Spiegler-Kedem model
TDS	-	total dissolved solid
TEM	-	transmission electron spectroscopy
TiO_2	-	Titanium dioxide
TFC	-	thin film composite
TFN	-	thin film nanocomposite

TMC	-	trimesoyl chloride
TNS	-	titania nanosheet
TNT	-	titania nanotube
TOC	-	total organic carbon
UF	-	ultrafiltration
UV-Vis	-	ultraviolet-visible
VC	-	vapor compression
WHO	-	World Health Organization
XPS	-	X-ray photoelectron spectroscopy
XRD	-	X-ray diffraction
ZnO	-	zinc oxide

LIST OF SYMBOLS

A_s	-	solvent transport parameter
A	-	permeability
A_m	-	effective area of membrane
A_{w1}	-	initial membrane water permeability
A_{w2}	-	membrane water permeability after washing
B	-	solute transport parameter
C	-	mean concentration over the thickness of the membrane
C_I	-	ion concentration
C_f	-	solute/salt concentration in the feed solution
C_p	-	solute/salt concentration in the permeate solution
C_w	-	concentration of dissolved water in membrane
C_m	-	concentration of solute in the fluid at the feed/membrane interface
D_v	-	solvent diffusivity
D_s	-	solute diffusivity
d-spacing	-	interlayer spacing
FRR	-	flux recovery rate
I	-	bacterial inhibition rate
J	-	flux
J_v	-	solvent flux
J_s	-	solute flux
K_s	-	solute partition coefficient between the solution and membrane phases
K_S	-	absorption of salt
L	-	solvent permeability coefficient
N_m	-	bacterial concentration of the membrane sample on the agar plate
N_o	-	bacterial concentration of the blank sample on the agar plate
P'	-	local solute permeability
P	-	solute permeability
p_f	-	feed pressure

p_p	-	permeate pressure
R	-	rejection
r	-	ratio of actual area to the projected area of the surface
R_a	-	average roughness
R_i	-	ideal gas constant
R_r	-	real retention
R_D	-	dilution ratio
R_s	-	solute rejection
R_v	-	recovery
RMS	-	root mean square roughness
S_v	-	solvent solubility
T	-	temperature
TOC_f	-	TOC of solute in feed
TOC_p	-	TOC of solute in permeate
V_v	-	solvent partial molar volume
Z	-	number of visible bacteria colonies on the agar plate
σ	-	reflection coefficient
δ	-	pore length
π	-	osmotic pressure
Δp	-	transmembrane pressure difference
Δt	-	collection time of permeate
ΔV	-	volume of permeate
Δx	-	membrane thickness
Δπ	-	osmotic pressure difference between the feed and permeate
γ	-	liquid/gas surface tension
γ_{sl}	-	solid/liquid interfacial energy
γ_{so}	-	solid/gas surface energy
$\gamma^P{}_S$	-	polar force of surface tension
Υ	-	surface tension
μ	-	viscosity

θ	-	diffraction angle of nanofiller peak
θ^*	-	apparent contact angle
θ_Y	-	equilibrium contact angle
λ	-	irradiation wavelength in X-ray diffraction spectrometer
ρ	-	density

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

Water crisis has become a threatening issue in many parts of the world despite the Earth is covered by 75% of water (Ali *et al.*, 2018). 97.5% of the 75% is made up of seawater and saline aquifers (Zaidi *et al.*, 2015) while fresh water only comprises of 2.5%. Out of the 2.5% of fresh water, only 0.3% is usable by human (Youssef, Al-Dadah and Mahmoud, 2014). The situation worsens when the available conventional freshwater resources are polluted by human activities and overexploited. In the 20th century, human population increases by four times but the demand for water has increased by nine times (Shenvi, Isloor and Ismail, 2015).

Figures 1.1A and 1.1B show the water withdrawal, gross domestic product (GDP) pro-capita and world population and the breakdown of human water consumption from various sectors while Figure 1.1C demonstrates the graphical concept of water crisis when demand is continuously increased while clean water availability is decreasing. The linear relationship of water withdrawal and world population, as observed in Figure 1.1A, further confirms that human population is the primary factor for the increase in global water demand. The global water withdrawal, which is in line with the sectoral water consumption, is predicted to rise continuously, with no sign of slowing down, until ~2099 (see Figure 1.1B). The drastic rise in irrigation and livestock water consumption is attributable to the increasing food demand, while the surge in domestic and industrial water use is ascribed to the increase in electricity and energy usage. Climate change is another factor that contributes to the remarkably high irrigation water consumption (Wada and Bierkens, 2014). It is intuitive that the growing demand and shrinking water availability will ultimately cross each other (see Figure 1.1C) when water withdrawal rate overcomes the nature's self-replenishing rate. At that moment, water crisis happens.

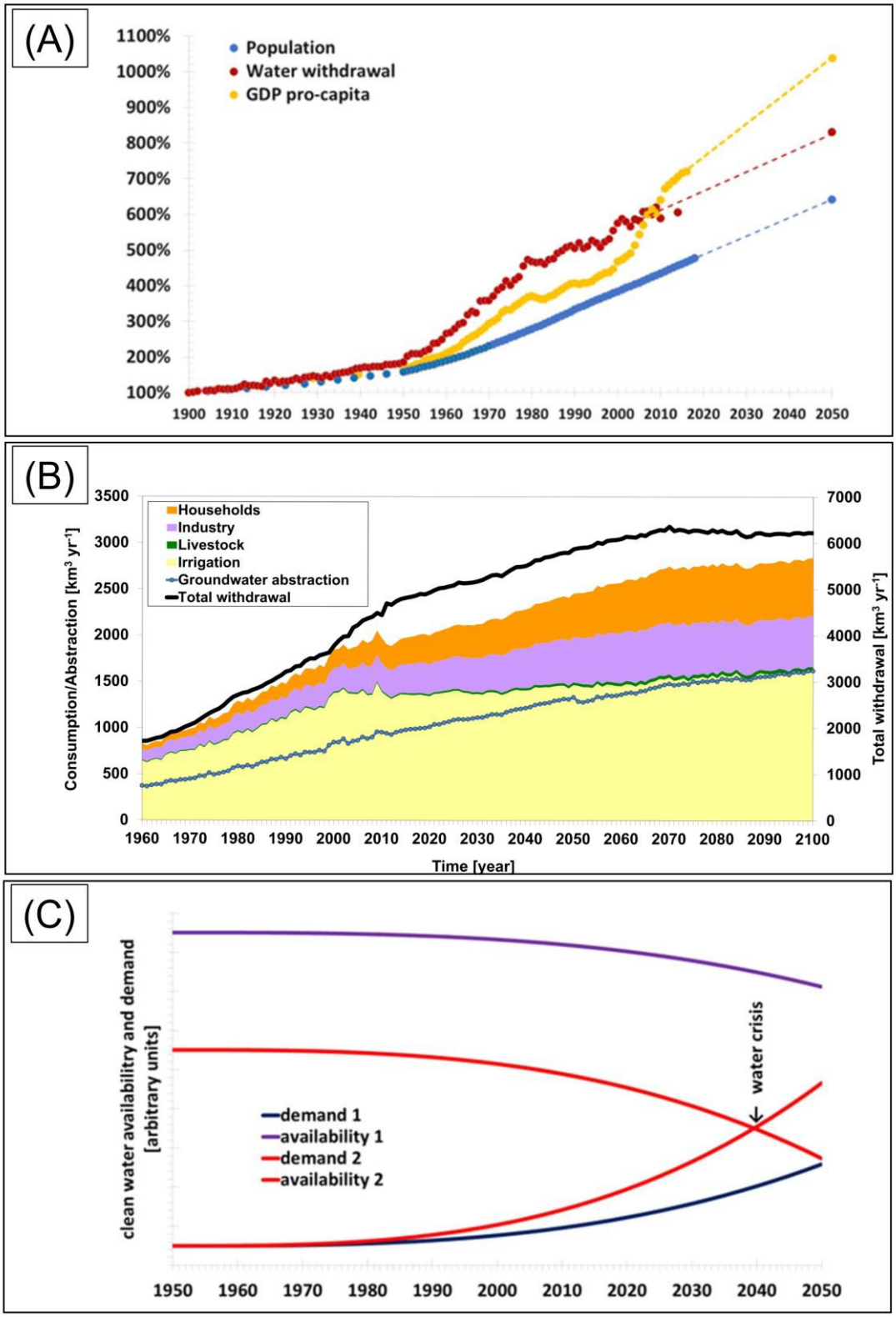

Figure 1.1 (A) Water withdrawal, GDP pro-capita and world population (Boretti and Rosa, 2019), (B) Estimated and projected trends of total blue water withdrawal, sectoral blue water consumption and ground water extraction over the period of 1960 to 2099 (Wada and Bierkens, 2014) and (C) graphical concept of water scarcity, resulting from a more than linear growing demand and a similarly more than a linear reduction of clean water availability (Boretti and Rosa, 2019)

It was previously mentioned by Service (2006) in his article "Desalination Freshens Up" that over 1 billion of people did not have access to clean drinking water and approximately 2.3 billion of people, which were about 41% of world population, were living in water stress region. It is also revealed in the United Nations World Water Development Report Edition 2018 that approximately 6 billion of people will suffer from clean water scarcity in 2050. Figure 1.2 predicts expansion of water stress region from 2019 to 2050. Based on the Global Risk report, water crisis has been listed and remained as the top 5 risk by severity of impact since 2014 (Brende, 2020).

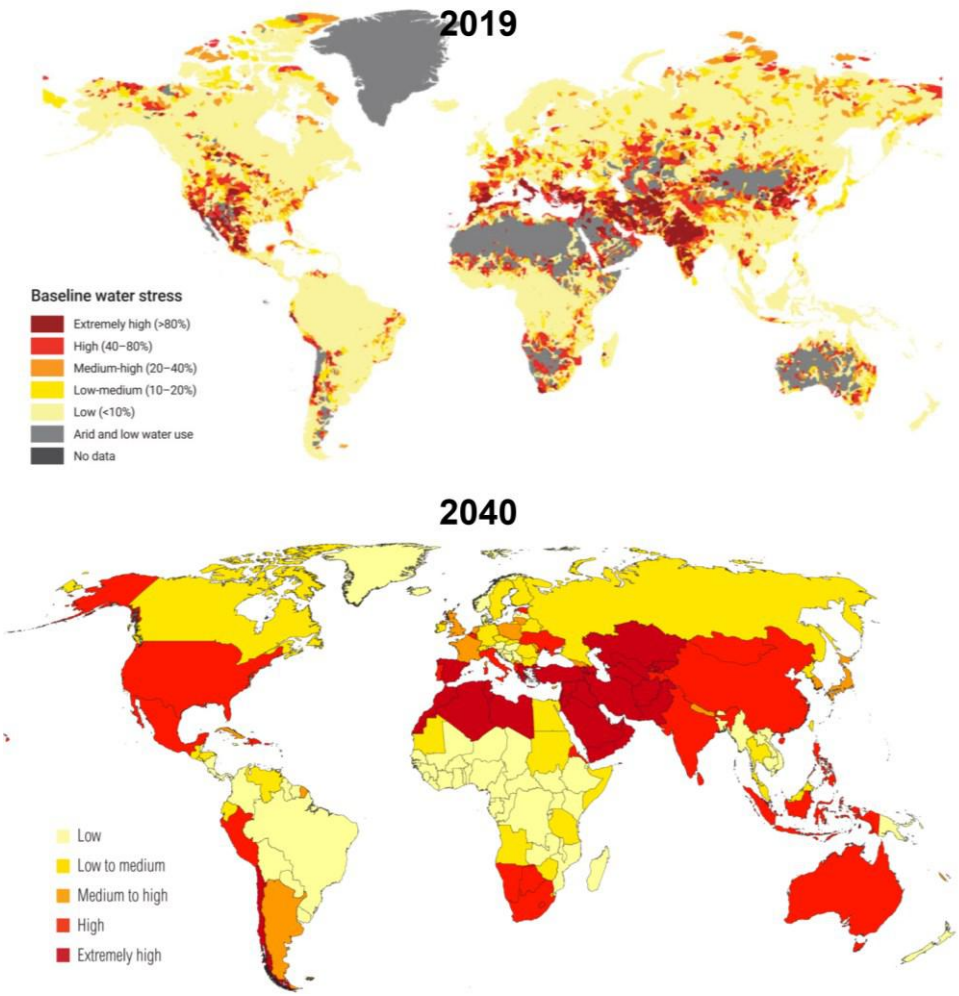

Figure 1.2 Expansion of water-stressed region from year 2019 (Uhlenbrook *et al.*, 2020) to year 2040 (Luo, Young and Reig., 2015)

The water crisis issue has urged mankind to search for a solution in resolving the Earth's meagered fresh water supplies, leading to the development of water reuse and water desalination (Jiang, Li and Ladewig, 2017). Water reuse has been widely

applied to produce freshwater for irrigation, industrial activities, recharging of ground water and as a method for indirect drinking water production. Meanwhile, water desalination remains as the most preferred choice for drinking water production due to its ability to produce freshwater that fulfils the drinking water standard of the World Health Organization (WHO) (Greenlee *et al.*, 2009).

The two commonly applied desalination technologies are membrane-based technology and thermal-based technology. However, due to high energy consumption of thermal-based technology, membrane-based technology is more preferred. Membrane-based technology which is pressure-activated utilizes high pressure to force water across semipermeable membrane and leave salts behind (Youssef, Al-Dadah and Mahmoud, 2014). Reverse osmosis (RO) is currently the most promising membrane-based desalination technology. Besides being energy saving, RO also exhibits excellent separation performance with high water permeability fulfilling the demand of human population (Jiang, Li and Ladewig, 2017). Figure 1.3 shows the number and capacity of operational desalination plants by technology. According to Jones *et al.* (2019), RO is the most dominating process that accounts for 84% of the total number of operational desalination plants and produces 69% of global desalinated water.

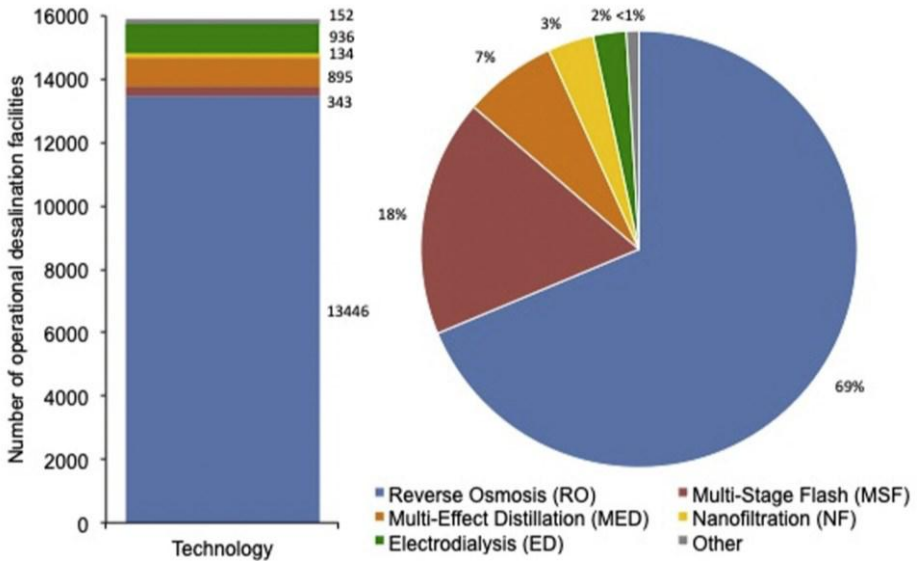

Figure 1.3 Number and capacity of operational desalination facilities by technology (Jones *et al.*, 2019)

To date, desalination technology is dominated by thin film composite (TFC) polyamide (PA) RO membranes (Lee, Arnot and Mattia, 2011), which was introduced by Cadotte and his co-workers in the 1970s (Cadotte *et al.*, 1980). This type of membrane consists of three layers, ultrathin PA layer, porous substrate and non-woven polyester (PET) fabric that is prepared via interfacial polymerization (IP) method. It exhibits excellent salt rejection of >99% and relatively higher water permeability, ~0.74 $L/m^2 \cdot h \cdot bar$ as compared to asymmetric RO membranes. TFC membrane also exhibits better tolerance to temperature, chemical, wider range of pH and compaction. Nevertheless, the TFC membrane is subject to ubiquitous trade-off relationship between permeability and selectivity which hinders the simultaneous improvement of membrane water permeability and solute rejection. Apart from that, the TFC membrane is also susceptible to fouling and chlorine attack (Li, Yan and Wang, 2016). Following this, numerous studies were carried out by membrane scientists to address its drawbacks by modifying the structural properties and surface chemistry of TFC membrane, aiming to increase its resistibility against potential foulants and free active chlorine as well as improving its water permeability. Interlayer regulated IP process is one of the emerging strategies to improve the performances of TFC membrane (Dai, Li and Wang, 2020; Ng *et al.*, 2021). Compared to the conventional IP, interlayer-assisted IP usually produces PA which is thinner, smoother and more compact. These features grant the resultant TFC membrane its higher water permeability and salt rejection (Dai, Li and Wang, 2020).

Another effective strategy is to integrate inorganic nanofillers into the ultra-thin barrier layer, resulting in the development of new type of composite membrane named as thin film nanocomposite (TFN) membrane. The nanofillers can be added into the selective PA layer via IP method. Addition of nanofillers is not only able to improve membrane water permeability, but also prevent the membrane selectivity from being compromised, as initially reported by Jeong *et al.* (2007). Apart from that, the incorporation of nanofillers could improve membrane antifouling, chlorine resistivity, antibacterial, mechanical property and thermal resistivity. Some of the inorganic nanofillers investigated include carbon-based (e.g., carbon nanotube (CNT), graphene oxide (GO)), metal and metal oxides-based (e.g., silver (Ag), copper (Cu), titanium dioxide (TiO_2), zinc oxide (ZnO) and metal organic framework (MOF)), silica, zeolite and halloysite nanotube (HNT) (Saleem and Zaidi, 2020).

Lately, GO has become one of the promising nanofillers for the development of TFN RO membrane (Liu and Xu, 2016). GO is attractive due to two main reasons, i.e., unique structure and superior hydrophilicity (Liu and Xu, 2016; Akther *et al.*, 2020). Its two dimensional (2D), single atom-thick sheet-like structure is capable of creating additional channels for water to pass through when they are stacked (Choi *et al.*, 2013). Meanwhile, the existence of abundant oxygen functional entities such as hydroxyl, epoxy and carboxyl groups locating at the surfaces and edges of GO could make it highly hydrophilic (Dreyer *et al.*, 2010; Shamaila, Sajjad and Iqbal, 2016; Inurria *et al.*, 2019). These properties provide the resultant TFN membrane with enhanced surface hydrophilicity, leading to improved permeability and antifouling property, when the GO is incorporated into the PA layer. Besides, the ability of GO to form intermolecular hydrogen bonding with PA layer could prevent the amide bond from being attacked by free chlorine (Ali *et al.*, 2016). GO also possesses antimicrobial property which is useful in mitigating biofouling of the resultant TFN membrane (Inurria *et al.*, 2019). Direct contact of bacteria with GO nanosheets results in the disruption of the cell membrane integrity and the oxidation of cellular components, which can induce loss of cell viability (Li *et al.*, 2011; Sanchez *et al.*, 2012; Mangadlao *et al.*, 2015). Albeit all the advantages of GO incorporated TFN membranes, there remains some limitations in the membrane properties that needed to be addressed such as incomplete accommodation, uneven distribution and random arrangement within the PA layer (Lai *et al.*, 2019; Lim *et al.*, 2020; Rodríguez *et al.*, 2020). Therefore, this study aims to investigate the combine effects of intermediate layer and GO incorporation on the physicochemical properties and performances of the resultant TFN membranes for desalination process. The effects of intermediate layer on the deposition of GO in the PA layer are explored.

1.2 Problem Statements

IP technique is widely employed for TFC and TFN membranes fabrication. Generally, it involves the reaction of two monomers (i.e., amine in aqueous medium and acyl chloride in organic medium) at the interface of two immiscible solutions to produce a thin and dense PA layer followed by heat treatment to improve the cross-

linking degree and remove excess solvents (Ghosh *et al.*, 2008). Nevertheless, the current TFC membrane still suffers from permeability/selectivity trade-off, chlorine attack and fouling. To deal with these problems, studies were conducted to alter the physicochemical properties of TFC membrane, among which varying PA synthesis conditions (i.e., IP parameters) and nanofillers incorporation (Xu, Wang and Li, 2013; Otitoju, Saari and Ahmad, 2018) are some of the effective strategies. Lately, the potential of post IP rinsing in improving water flux at the expense of solute rejection of TFC membrane was reported by Chong *et al.* (2018). Post IP rinsing is often required to remove unreacted monomers from the TFC membrane surface prior to heat treatment process. However, its effects on membrane physicochemical properties remain largely unclear. Thus, the first part of this research intends to study in depth the effects of post IP rinsing on the physicochemical properties and performances of TFC membranes by using various organic solvents (i.e., hexane, cyclohexane and Isoparrafin-G) to compare with water at different rinsing conditions (i.e., with rinsing (meaning the membrane is immediately rinsed after IP) and with post air-drying and rinsing (meaning the membrane is subjected to 1 min air-drying after IP before subjecting to rinsing). It is anticipated that highly reproducible TFC membranes with insignificant permeability/selectivity trade-off could be fabricated and serve as a stable baseline for the development of PEI-interlayered TFN membranes.

In this study, GO nanosheets are proposed as the nanofillers for the fabrication of TFN membrane owing to its unique structure and superior hydrophilicity (containing an abundance of oxygen rich functional groups). Nevertheless, there remain several problems that need to be addressed when adding GO into the PA layer via IP method. In terms of method, IP process which involves the use of rubber roller to remove excess amine monomers prior to introducing acyl chloride monomers often leads to uneven distribution and/or significant loss of GO from the substrate surface (Lai *et al.*, 2019), especially when GO is weakly adhered to the substrate (Lim *et al.*, 2020). In terms of nanofillers, large lateral size of GO could affect PA integrity by obstructing the diffusion of amine to the organic phase and its incomplete accommodation within the PA layer. As reported by Akther *et al.* (2020), GO with average area of 1.06 μm^2 or lateral size <5 μm could lead to defective PA layer. Despite being atom-level thick, the large lateral size of GO makes it difficult to be completely accommodated within the several hundreds of nanometer thick PA (Yin, Zhu and

Deng, 2016; Rodríguez *et al.*, 2020), particularly when these nanosheets are not properly arranged (Lim *et al.*, 2020). Therefore, in the second part of this study, a positively charged polyethyleneimine (PEI) is used as interlayer while the rubber rolling method is replaced with oven-drying method to remove the excess amine monomers. The purpose of using PEI interlayer is to improve the arrangement/orientation and adherence of negatively charged GO via electrostatic interaction while the adoption of oven-drying method is to minimize uneven distribution of GO on substrate surface. Interlayer-assisted IP is an emerging technique that could precisely control the polymerization process to produce thin and defect-free PA layer. This technique has been demonstrated for the fabrication of nanofiltration (NF) and forward osmosis (FO) membranes, but its potential use for RO membrane fabrication is yet to be documented. It is anticipated that TFN membranes with enhanced PA integrity, by minimizing defects formation, could be generated via the PEI-interlayered assisted IP.

Loading is another important factor when adding GO into the PA layer. Even though GO exhibits good dispersity in aqueous or polar solvents, excessive amount of GO can still lead to agglomeration. GO agglomeration or aggregation is undesired because it could lead to the formation of non-selective interfacial voids that adversely affects the separation performances of membrane. To date, numerous studies were conducted to investigate the effects of GO loading on the properties and performances of TFN membrane (He *et al.*, 2015; Yin, Zhu and Deng, 2016; Ali *et al.*, 2016) but not for interlayered TFN membrane. Alignment of GO induced by PEI could possibly affect the maximum amount of GO that could be accommodated within the PA layer. Thus, the third part of this study will investigate the effects of GO loading on the properties and performances of PEI-interlayered TFN membrane. In addition, the change in GO position due to the presence of PEI interlayer could possibly affect its positive features. Thus, the performances of PEI-interlayered TFN membrane with respect to chlorine resistivity, antifouling and antibacterial property will be evaluated at the last part of this study.

1.3 Research Objectives

Based on the research problems highlighted in the previous sub-section, the following objectives are set out:

1. To investigate the effects of post IP rinsing on the physicochemical properties and filtration performances of TFC RO membrane by varying the types of rinsing solution and rinsing conditions.

2. To investigate the impacts of PEI interlayer on the physicochemical properties and filtration performances of TFN RO membrane by depositing the PEI on the surface of polysulfone (PSf) substrate at different coating parameters.

3. To investigate the effects of GO on the physicochemical properties and filtration performances of PEI-interlayered TFN RO membrane by incorporating different GO loadings into the PA layer.

4. To evaluate the performances of selected PEI-interlayered TFN RO membrane with respect to chlorine resistivity, antifouling and antibacterial properties.

1.4 Research Scopes

In order to achieve the objectives of this research, the following scopes are planned:

For Objective 1:

(a) Fabricating TFC RO membranes via IP using rubber rolling technique (aqueous phase: 2 wt/v% m-phenylenediamine (MPD); organic phase: 0.1 wt/v% trimesoyl chloride (TMC)). The resultant membranes are subjected to post IP rinsing using water and various organic solvents (i.e., hexane, cyclohexane and Isoparaffin-G) at different rinsing conditions (i.e., with rinsing and with post air-drying and rinsing) prior to heat treatment.

(b) Characterizing surface hydrophilicity, morphology, roughness and functional groups of TFC RO membranes using contact angle goniometer, field emission scanning electron microscopy (FESEM), atomic force microscopy (AFM) and attenuated total reflectance Fourier-transform infrared spectroscopy (ATR-FTIR).

(c) Estimating membranes pore radii based on irreversible thermodynamic model and Steric Hindrance Pore Model by filtrating 40 mg/L glucose, glycerol and ethylene glycol (EG) at different pressures (i.e., 11, 13, 15 and 17 bar) using dead-end RO filtration system.

(d) Evaluating the pure water flux (PWF) and sodium chloride (NaCl) rejection of TFC RO membranes fabricated via IP using RO water and 2000 mg/L NaCl, respectively, as feed solution in dead-end RO filtration system.

For Objective 2:

(a) Synthesizing GO from graphite powder using modified Hummers' method.

(b) Characterizing the physicochemical properties of self-synthesized GO using transmission electron microscopy (TEM), AFM, FTIR, Ultraviolet-visible (UV-vis) spectrophotometry and X-ray diffraction (XRD) spectroscopy.

(c) Modifying PSf substrate using PEI solution by varying several coating parameters (i.e., concentration (0.005–0.2 wt/v%), drying time (0–120 min) and coating layer number (1–3 layers)).

(d) Characterizing surface hydrophilicity, morphology, roughness, charge and functional groups of PEI-coated PSf substrates using contact angle goniometer, FESEM, AFM, zeta potential analyzer and ATR-FTIR.

(e) Determining PEI-coated PSf substrates molecular weight cut-off (MWCO) by filtrating 100 mg/L polyethylene glycol (PEG) with different molecular weight (MW) (i.e., 600, 3400, 8000, 10,000 and 12,000 g/mol) using dead-end RO filtration system.

(f) Fabricating TFN RO membranes via IP and interlayer-assisted IP using oven-drying technique (aqueous phase: 2 wt/v% MPD and 0.015wt/v% GO; organic phase: 0.1 wt/v% TMC). The resultant membrane is subjected to post IP rinsing

using the best rinsing solution and condition discovered from Objective 1 prior to heat treatment.

(g) Characterizing surface hydrophilicity, morphology, roughness, and functional groups of TFN RO membranes using contact angle goniometer, TEM, FESEM, AFM and ATR-FTIR.

(h) Evaluating the pure water permeability (PWP) and NaCl rejection of TFN RO membranes fabricated via IP and interlayer-assisted IP using RO water and 2000 mg/L NaCl, respectively, as feed solution in dead-end RO filtration system.

For Objective 3:

(a) Fabricating TFC RO membrane via IP using oven-drying technique (aqueous phase: 2 wt/v% MPD; organic phase: 0.1 wt/v% TMC). The resultant membrane is subjected to post IP rinsing using the best rinsing solution and condition discovered from Objective 1 prior to heat treatment.

(b) Fabricating TFC and TFN RO membranes via interlayer-assisted IP using oven-drying technique (aqueous phase: 2 wt/v% MPD and 0–0.02wt/v% GO; organic phase: 0.1 wt/v% TMC). The resultant membrane is subjected to post IP rinsing using the best rinsing solution and condition discovered from Objective 1 prior to heat treatment.

(c) Characterizing surface hydrophilicity, morphology, roughness, charge and functional groups of TFN RO membranes using contact angle goniometer, FESEM, AFM, zeta potential analyzer, ATR-FTIR and X-ray photoelectron spectroscopy (XPS).

(d) Evaluating the PWP and NaCl rejection of TFC and TFN RO membranes fabricated via IP and interlayer-assisted IP using RO water and 2000 mg/L NaCl, respectively, as feed solution in dead-end RO filtration system.

For Objective 4:

(a) Comparing chlorine resistivity of TFC with optimized TFN RO membrane fabricated via interlayer-assisted IP by exposing the membranes to different concentrations of sodium hypochlorite (NaOCl) (i.e., 500 and 1000 mg/L).
(b) Comparing antifouling property of TFC with optimized TFN RO membrane fabricated via interlayer-assisted IP using a mixture of 1000 mg/L sodium alginate (NaAlg) with 2000 mg/L NaCl as feed solution.
(c) Comparing antibacterial property of TFC with optimized TFN RO membrane fabricated via interlayer-assisted IP using two different types of bacteria (i.e., gram-negative Escherichia Coli (*E. coli*) and gram-positive Staphylococcus aureus (*S. aureus*)).

1.5 Significance of Study

Over the past decade, TFN membrane acted as one of the most promising strategies to tackle all the limitations of PA TFC membranes. Previous studies showed that the addition of nanofillers, particularly GO, into the PA layer mostly focused on overcoming the membrane permeability and selectivity trade-off, chlorine sensitivity and fouling propensity (Chae *et al.*, 2015; He *et al.*, 2015; Yin, Zhu and Deng, 2016; Ali *et al.*, 2016; Inurria *et al.*, 2019). Little attention was paid on the effects of GO deposition on PA integrity and thus the membrane performances. Referring to some of the previous studies, random arrangement of large GO within the PA layer could affect membrane integrity and jeopardize its selectivity (Lim *et al.*, 2020; Rodríguez *et al.*, 2020). Significant loss and/or poor distribution of GO within the PA layer could reduce the positive features of GO in the synthesized TFN membrane (Lai *et al.*, 2016b, 2019).

This study aims to pioneer the production of GO incorporated TFN membrane with improved arrangement/orientation and optimized loading that paves ways to the better PA integrity via interlayer-assisted IP technique. The use of interlayer for the fabrication of NF and FO membranes are common but not for RO membrane.

Introducing interlayer as demonstrated in this work is able to improve the deposition of nanofillers in the RO membrane, improving the membrane water permeability without significantly compromising its selectivity. The findings of this work also provides an in-depth insight to the effects of GO position on membrane physicochemical properties and their ultimate performances on membrane chlorine resistivity, antifouling and antibacterial property. This could prevent membrane researchers from stumble upon the same issue and speed up the development of membrane for saline water desalination in the future. The outcomes of the study clearly indicates the potential of interlayer-assisted IP in producing GO-containing TFN membrane with better PA integrity and properties for water desalination.

1.6 Assumptions and Limitations of Study

Due to the flexibility of IP parameters (i.e., monomer types and concentrations, immersion and reaction time, heat treatment conditions and types of organic solvent used), the parameters used for the fabrication of TFC and TFN membranes as reported by Wan Azelee *et al.* (2017) was adopted to narrow down the scope. The incomplete removal of preservatives coated on commercial substrate could possibly affect the formation of PA or the performance of resultant TFC membrane and to narrow down the scope again, the exact preservatives removal procedure that was reported by Chong *et al.* (2018) was followed. The amount of nanofillers deposited within the selective layer was not quantified as it was nearly impossible to isolate the thin PA from the nanofillers. Moreover, it was difficult to distinguish whether the elemental composition of carbon and oxygen in the selective layer and the chemical bonding of O=C=O and C-C analyzed from the high-resolution spectra was contributed by GO or PA since both of them contain these elements and functional groups. The exact chemical interaction between MPD and PEI was unable to be accurately determined since it is not a strong chemical bond like ionic or covalent bond.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Ng, Z.C.**, Lau, W.J., Wong, K.C., Al-Ghouti, M.A. & Ismail, A.F. (2021). Improving Properties of Thin Film Nanocomposite Membrane through Polyethyleneimine Intermediate Layer: A Parametric Study. *Separation and Purification Technology, 274,* 119035. https://doi.org/10.1016/j.seppur.2021.119035. **(Q1, IF: 5.774)**

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