



University of Technology Sydney
FACULTY OF ENGINEERING

**DESIGN AND FABRICATION OF NOVEL
NANOFIBER MEMBRANES VIA
ELECTROSPINNING TECHNIQUE FOR
MEMBRANE DISTILLATION**

by

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A Thesis submitted in fulfilment for the degree of
Doctor of Philosophy

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CERTIFICATE OF ORIGINAL AUTHORSHIP

I certify that the work in this thesis has not previously been submitted for a degree nor has it been submitted as part of requirements for a degree except as part of the collaborative doctoral degree and/or fully acknowledged within the text.

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LIST OF ABBREVIATIONS

2D	Two-dimensional
AD	Adsorption desalination
AED	Adsorption energy distribution
AFM	Atomic force microscopy
AGMD	Air gap membrane distillation
APS	Accelerated precipitation softening
ATR-FTIR	Attenuated total reflectance – Fourier transform infrared spectroscopy
BaSO ₄	Barium sulfate
BET	The Brunauer-Emmett-Teller
BJH	The Barrett-Joyner-Halenda
BNNPs	Boron nitride nanoparticles
BTEAC	Benzyltriethylammonium chloride
CA	Contact angle
Ca(OH) ₂	Calcium hydroxide
CaCO ₃	Calcium carbonate
CaSO ₄	Calcium sulfate
CBD	Coal bed methane
CDI	Capacitive deionization
CFP	Capillary flow porometry
CNTs	Carbon nanotubes
COD	Chemical oxygen demand
C-PVDF	Commercial PVDF membrane
CSG	Coal seam gas

CuO	Copper oxide
DC	Direct current
DCMD	Direct contact membrane distillation
DI	De-ionized
DMF	N, N-dimethylformamide
DS	Draw solution
EDX	Energy dispersive x-ray spectroscopy
ENM	Electrospun nanofiber membrane
ERD	Energy recovery device
FeCl ₃	Ferric chloride
FO	Forward osmosis
FOHC	The FO-RO hybrid Desalination Research Center
FS	Feed solution
GMVP	The Global MVP project
G/PH	Graphene/Polyvinylidene fluoride-co-hexafluoropropylene
GO	Graphene oxide
GOR	Gain output ratio
HA	Humic acid
HCl	Hydrochloric acid
h-BN	The hexagonal boron nitride
iCVD	Initiated chemical vapor deposition
IP	Interfacial polymerization
IPA	Isopropanol
KORAE	The Korean Optimized RO desalination for Advanced Energy saving
LEP	Liquid entry pressure

LiCl	Lithium chloride
MBR	Membrane bioreactor
MCDI	Membrane capacitive deionization
MCr	Membrane crystallization
MDBR	Membrane distillation-membrane bioreactor
MED	Multi-effect distillation
MEMD	Multi-effect membrane distillation
MD	Membrane distillation
MF	Microfiltration
MGMD	Material gap membrane distillation
MSF	Multi-stage flash
MWNTs	Multi-walled nanotubes
N6	Nylon-6
NaCl	Sodium chloride
NaOH	Sodium hydroxide
NCC	Nanocrystalline cellulose
NIPS	Non-solvent induced phase separation
NOMs	Natural organic matters
NTIPS	Non-solvent with thermally induced phase separation
OCM	Orthogonal collocation method
ODEs	Ordinary differential equations
OMW	Olive mill wastewater
PA	Polyamide
PACl	Polyaluminum chloride
PAM	Polypropylene acid ammonium

PAN	Polyacrylonitrile
PDEs	Partial differential equations
PDMS	Polydimethylsiloxane
PEI	Polyetherimide
PES	Polyethersulfone
PET	Polyethylene terephthalate
PGMD	Permeate gap membrane distillation
PH	Polyvinylidene fluoride-co-hexafluoropropylene (PVDF-co-HFP)
PP	Polypropylene
PRO	Pressure retarded osmosis
PS	Polystyrene
PSD	Pore size distribution
PSf	Polysulfone
PTFE	Polytetrafluoroethylene
PVA	Polyvinyl alcohol
PVAc	Polyvinyl acetate
PVC	Polyvinyl chloride
PVDF	Polyvinylidene fluoride
PVDF-CTFE	Poly(vinylidene fluoride-co-chlorotrifluoroethylene)
RCW	Recirculating cooling water
RED	Reverse electrodialysis
RF	Radio frequency
RO	Reverse osmosis
SA	Sliding angle
SAED	Selected area electron diffraction

SCCM	Standard cubic centimetre per minute
SDS	Sodium dodecyl sulfate
SEC	Specific energy consumption
SEM	Scanning electron microscopy
SFE	Surface free energy
SGMD	Sweeping gas membrane distillation
SiO ₂	Silicon dioxide
SMM	Surface modifying macromolecules
SrSO ₄	Strontium sulfate
SUS	Stainless steel
SWRO	Seawater reverse osmosis
TB	Tri-bore
TCD	Tip-to-collector distance
TCM	Traditional Chinese medicine
TEM	Transmission electron microscopy
TIPS	Thermally induced phase separation
TiO ₂	Titanium oxide
TFC	Thin film composite
TGA	Thermogravimetric analysis
TOC	Total organic carbon
TPC	Temperature polarization coefficient
TSS	Turbidity and fine particulates
UF	Ultrafiltration
UTM	Universal testing machine
VIPS	Vapor induced phase separation

VMD	Vacuum membrane distillation
VMEMD	Vacuum multi effect membrane distillation
VODE	Variable coefficient ordinary differential equation
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction

LIST OF SYMBOLS

A	Effective area of the membrane
C	Cold fluid
C_{AGMD}	Mass transfer coefficient
C_{AGMD-G}	Mass transfer coefficient related with the graphene effect
C_p	Permeate concentration
C_f	Feed concentration
C_m	Membrane mass transfer coefficient
C_p	Specific heat capacity
D_{AB}	Water vapor diffusion coefficient
d_h	Equivalent hydraulic diameter
$Flux_E$	Flux of each point
$Flux_I$	Flux of the initial point
g	Gravity
h	Heat transfer coefficient
H	Hot fluid
I_D	D band
I_G	G band
J	Water vapour flux
J_{AGMD}	Water vapour flux by AGMD
J_{AGMD-G}	Water vapour flux by AGMD with graphene effect
J/J_0	Normalized flux
k_x	Transversal thermal conductivity
k_z	Axial thermal conductivity

L	Channel length
LMH	L/m ² h
M_w	Molecular mass of water
P_{avgm}	Log mean air pressure based from both sides of the membrane
P_{avgma}	Log mean air pressure within the air gap
P_C	Water vapor pressure on the air gap layer
P_H	Water vapor pressure on the membrane surface
Pr	Prandtl number
P_T	Total pressure of water vapor and air
P_w	Vapor pressure of pure water
r	Membrane pore radius
R	Universal gas constant
R_a	Mean roughness
R_{AGMD}	Total mass transfer resistance in the AGMD
γ_m	Surface tension of the membrane in contact with air
γ_{ml}	Surface tension of the membrane in contact with liquid
γ_l	Surface tension of liquid in contact with air
γ_m^T	Total surface free energy
γ_m^{LW}	Lifshitz van der Walls interaction of the membrane
γ_m^{AB}	Lewis acid-base interaction of the membrane
γ_m^+	Electron acceptor parameter
γ_m^-	Electron donor parameter
Re	Reynolds number
R_G	Molecular diffusion resistance related with the graphene effect

R_K	Knudsen diffusion resistance
R_M	Molecular diffusion resistance
R_{M-air}	Molecular diffusion resistance in the air gap
RT	Room temperature
SR	Salt rejection ratio
$T_{avg,a}$	Average temperature based from both sides of the membrane
$T_{avg,m}$	Average temperature at the air gap
u	Flow velocity
W_1	Weight of the saturated membrane
W_2	Weight of the dry membrane
x_{NaCl}	Mole fraction of NaCl
x_w	Mole fraction of water
t	Operating duration
γ_w	Activity coefficient of water
δ	Thickness
δ_m	Membrane thickness
δ_a	Air gap thickness
Δg	Mass of permeate
ε	Membrane porosity
k	Thermal conductivity
λ	Latent heat of water
μ	Viscosity
ρ	Liquid density
ρ_e	Density of ethanol

ρ_d	Density of PVDF material
τ_m	Membrane pore tortuosity
Γ	Total flow rate of condensate at the bottom of the condensing surface
<i>air</i>	Air
<i>cf</i>	Condensate film
<i>cp</i>	Cooling plate
<i>f</i>	Feed
<i>fl</i>	Fluid
<i>gap</i>	Air gap
<i>m</i>	Porous membrane
<i>si</i>	Solid membrane

ABSTRACT

In recent decades, many regions of the world suffer from water scarcity, which is one of the most critical issues in the world. The main challenge is to supply fresh water to water shortage regions. In addition, waterborne illness has been caused through the consumption of the contaminated drinking water in these regions. Seawater desalination is one of the alternative ways to produce freshwater. However, current desalination technologies like reverse osmosis (RO), multi-stage flash (MSF), and multi-effect distillation (MED) have several issues such as high energy consumption, a low recovery rate of total water, and large footprint. Among the several techniques to replace conventional desalination techniques, membrane distillation (MD) is one of the promising technologies. Currently, microfiltration (MF) membranes are implemented for MD application due to their suitable pore size distribution. However, some properties of MD are still needed to be enhanced, especially the high hydrophobicity to avoid membrane pore wetting and high porosity to increase permeate flux. With the development of nanotechnology, electrospinning is becoming a promising technology to fabricate hydrophobic and highly porous membranes. Thus, the objectives of this dissertation are to fabricate a suitable membrane for MD technology by electrospinning technique.

Novel nanofiber membranes fabricated by electrospinning technique are herein proposed for MD application to treat seawater and RO brine from coal seam gas (CSG) produced water. The electrospun membrane could be tailored to have superhydrophobicity, high porosity, adequate pore sizes and narrow pore size distribution, and thin thickness, so it could be used in applications of high-performance MD process. To further improve the MD performance of the electrospun membranes,

three different methods were considered: (i) Janus-type hydrophobic/hydrophilic nonwoven membrane to reduce mass transfer resistance, (ii) nano-materials embedded membrane to improve liquid entry pressure (LEP), and (iii) surface modification of electrospun membranes to treat challenging wastewater sources.

Janus-type hydrophobic/hydrophilic dual-layer nanofiber nonwoven membranes were initially fabricated by a facile electrospinning technique and applied for desalination by air gap MD (AGMD). As-spun neat single and dual-layer nanofiber membranes composed of a hydrophobic polyvinylidene fluoride-co-hexafluoropropylene (PH) top layer with different supporting hydrophilic layer made of either polyvinyl alcohol (PVA), nylon-6 (N6), or polyacrylonitrile (PAN) nanofibers were fabricated with and without heat-press post-treatment. Surface characterization showed that the active layer (i.e., PH) of all electrospun nanofiber membranes (ENMs) exhibited a rough, highly porous (>80% porosity), and hydrophobic surface ($CA > 140^\circ$), while the other side was hydrophilic ($CA < 90^\circ$) with varying porosity. Heat-pressing the membrane resulted to thinner thickness (from $>129 \mu\text{m}$ to $<100 \mu\text{m}$) and smaller pore sizes ($<0.27 \mu\text{m}$). The AGMD experiments in a cross-flow set up were carried out with constant inlet temperatures at the feed and permeate streams of 60 ± 1.5 and 20 ± 1.5 °C, respectively. The AGMD module had a membrane area of 21 cm^2 and the thickness of the air gap was 3 mm. The neat single and dual-layer ENMs showed a water permeate flux of about $10.9 \sim 15.5 \text{ L/m}^2 \text{ h}$ (LMH) using 3.5 wt % NaCl solution as feed, which was much higher than that of a commercial PVDF membrane (~ 6 LMH). The provision of a hydrophilic layer at the bottom layer enhanced the AGMD performance depending on the wettability and characteristics of the support layer. The PH/N6 dual-layer nanofiber

membrane prepared under the optimum condition showed flux and salt rejection of 15.5 LMH and 99.2 %, respectively, which has good potential for AGMD application.

Three different nanomaterials were incorporated in polymeric solutions for the improvement of liquid entry pressure (LEP), which were carbon nanotubes (CNTs), graphene, and hexagonal boron nitride (h-BN). Firstly, superhydrophobic, robust, mixed PH nanofiber membranes were fabricated incorporating CNTs as nanofillers to impart additional mechanical and hydrophobic properties. The electrospun membrane has been designed to have two cohesive layers, a thin CNT/PH top layer and a thick neat PH bottom layer. Through different characterization techniques, CNTs were found to be widely distributed on/in the nanofibers, where more beads-on-string were formed at higher CNT content. However, the beads-on-string did not significantly affect the membrane porosity and pore size, as well as did not degrade the MD performance. Highly-porous structure was observed for all membranes and the nanofiber membrane showed comparable pore sizes with a commercial flat-sheet PVDF membrane but at a much higher porosity (>85%). The contact angle increased to higher superhydrophobicity at 158.5° upon the incorporation of 5 wt% CNTs in the nanofiber due to increased roughness and added effect of hydrophobic CNTs. The liquid entry pressure also increased when 5 wt% CNT was added compared to the neat PH nanofiber membrane. The resulting flux of the 5 wt% CNT-incorporated nanofiber membrane (24-29.5 L/m²h) was consistently higher than the commercial PVDF membrane (18-18.5 L/m²h), with an average increase of 33-59% depending on the feed water type (35 or 70 g/L NaCl solution) without compromising the salt rejection (>99.99%). The present nanofiber membranes containing CNTs with one-step electrospinning fabrication show high potential for direct contact MD (DCMD) desalination application.

The following study demonstrated the development of a graphene-loaded electrospun nanofiber membrane and evaluation of their desalination performance in AGMD. Different concentrations of graphene (0-10 wt%) were incorporated in/on electrospun PH membrane to obtain a robust, and superhydrophobic nanocomposite membrane. The results showed that graphene incorporation has significantly enhanced the membrane structure and properties with an optimal concentration of 5 wt% (i.e., G5PH). Characterization of G5PH revealed membrane porosity of >88%, contact angle of >162° (superhydrophobic), and high LEP of >186 kPa. These favorable properties led to a high and stable AGMD flux of 22.9 L/m²h or LMH (compared with ~4.8 LMH for the commercial PVDF flat-sheet membrane) and excellent salt rejection (99.99%) for 60 h of operation using 3.5 wt% NaCl solution as feed (feed and coolant inlet temperatures of 60 and 20°C, respectively). A two-dimensional dynamic model to investigate the flux profile of the graphene/PH membrane is also introduced. The present study suggests that exploiting the interesting properties of nanofibers and graphene nanofillers through a facile electrospinning technique provides high potential towards the fabrication of a robust and high-performance MD membrane.

Another study focused on h-BN embedded nanofiber membrane to maintain flux stability in a long-term AGMD process. The hexagonal lattices of the BN nanoparticles (BNNPs) were modified by hydroxide-assisted ball milling without damage occurred during the exfoliation processes, and they were encapsulated in PH electrospun nanofiber membrane. Characteristics of the BN-PH membrane indicated almost similar regarding membrane thickness, fiber size, porosity and pore size. However, contact angle (153.2°) and LEP (214 kPa) of the BN-PH membrane were higher than that of the neat PH membrane, which showed that the BN-PH membrane could have less wetting

issues compared with the neat PH membrane. Besides, thermal conductivity of the neat PH and BN-PH was 0.025 W/mK and 0.009 W/mK, respectively, as expected that the BN-PH membranes could lead to a high MD water vapor flux performance due to reduced mass transfer resistance and also reduction in conductive heat loss via the membrane. The initial water vapor flux of the neat PH membrane was 11.42 LMH, however, it suffered wetting problem in less than 4 h operation. On the other hand, the BN-PH membrane showed a stable water vapor flux (18 LMH) and salt rejection (99.99%) performances even after 280 h of MD operation. This membrane has a good potential for long-term application of MD for seawater. Future interest in this study may be to find a mechanism for the improved water vapor flux performance of the BNNPs enabled electrospun nanofiber MD membrane.

MD process is also considered to treat wastewater or other challenging wastewater such as the one from textile, dye, and oil industries. However, MD membranes should be improved for preventing membrane wetting issues commonly caused by low surface tension liquids such as surfactants, benzene, methanol, and hexane. Thus, this study described the development and performance of an omniphobic poly(vinylidene fluoride) (PVDF) membrane fabricated by electrospinning and surface-modified by CF₄ plasma, for AGMD. The effect of different duration of plasma treatment on the nanofiber membrane characteristics was investigated. The AGMD performance of the membranes was evaluated using real RO brine produced from CSG produced water that was added with low surface tension liquid (surfactant) as feed solution. Results indicated the formation of new CF₂-CF₂ and CF₃ bonds after plasma treatment, which lowered the surface energy of the membrane, providing omniphobic property, as indicated by its wetting resistance to different low surface tension liquids such as methanol, mineral oil

and ethylene glycol. Though no appreciative changes in morphology of the membrane were observed after plasma treatment, optimal treatment condition of 15 min (i.e., P/CF-15 membrane) exhibited lotus effect membrane surface with increased LEP of 187 kPa compared to 142 kPa for neat membrane. AGMD performance showed stable normalized flux (initial flux of 15.3 L/m²h) and rejection ratio (99.99%) for P/CF-15 even with the addition of up to 0.7 mM sodium dodecyl sulfate surfactant to the RO brine from CSG produced water feed, while commercial PVDF membrane suffered membrane wetting after 0.3 mM of surfactant addition. Based on the results, the present omniphobic membrane has good potential for producing clean water from challenging waters containing high salinity and organic contaminants.

The aim of this study is the development of suitable electrospun nanofiber membranes for MD. This study mainly focuses on the newly-developed one-dimensional and two-dimensional nano-materials embedded nanofiber membranes, which suffer less wetting issue and have improved water vapor flux performance in MD. It also investigates a simple surface modification technique to generate anti-wetting property on the membrane surface. Overall, this author successfully fabricated several electrospun nanofiber membranes with enhanced water vapor flux and stable salt rejection performances for the treatment of seawater, seawater RO brine and CSG RO brine by MD applications. The fabricated electrospun nanofiber membranes exhibited better performances than the commercial PVDF membranes due to their suitable morphologies and characteristics for MD application. Thus, proper membranes were fabricated which led to enhanced membrane properties such as superhydrophobicity and anti-wetting property. And their MD performances have been compared with the ones in the previous reports. This study may therefore contribute to future MD researches regarding

using electrospinning for the developments of a commercial electrospun nanofiber MD membrane.

TABLE OF CONTENTS

ACKNOWLEDGEMENTS.....	ii
LIST OF ABBREVIATIONS	xiv
LIST OF SYMBOLS	xx
ABSTRACT	xxiv
TABLE OF CONTENTS.....	xxxii
LIST OF FIGURES	xxxix
LIST OF TABLES	xlvi
CHAPTER 1	1
INTRODUCTION.....	1
1.1 Introduction	2
1.2 Objectives and scope of the research.....	7
1.3 Structure of the study	8
CHAPTER 2	10
LITERATURE REVIEW.....	10
2.1 Introduction	11
2.2 Global water scenario	11
2.3 History of seawater desalination technologies.....	13
2.4 Improvements of desalination technologies	16
2.4.1 RO technologies	16
<i>2.4.1.1 Development of energy recovery device (ERD)</i>	<i>17</i>
<i>2.4.1.2 Evolution of reverse osmosis membrane</i>	<i>18</i>
2.4.2 Hybrid thermal desalination processes	19

2.5	Alternative membrane based-technologies for desalination	20
2.5.1	Forward osmosis.....	20
2.5.2	Pressure retarded osmosis.....	22
2.5.3	Membrane distillation (MD)	22
2.5.4	Capacitive deionization.....	24
2.5.5	Reverse electrodialysis	25
2.6	Membrane Distillation (MD).....	26
2.6.1	Overview of MD	26
2.6.2	Theoretical background.....	28
2.6.2.1	Mass transfer	28
2.6.2.2	Heat transfer	31
2.6.2.3	Temperature polarization coefficient (TPC)	32
2.6.3	MD membrane characteristics	34
2.6.3.1	Liquid entry pressure (LEP)	34
2.6.3.2	Pore size distribution (PSD)	36
2.6.3.3	Porosity.....	37
2.6.3.4	Thickness.....	37
2.6.3.5	Hydrophobicity of the MD membrane.....	38
2.6.4	Membrane fabrication and modification for MD	39
2.6.4.1	Phase separation techniques.....	39
2.6.4.2	Electrospinning technique.....	45
2.6.4.2.1	Electrospinning parameters	47
2.6.4.2.2	Electrospinning for membrane separation technology.....	48
2.6.4.2.3	Electrospinning for MD membrane.....	50
2.6.4.3	Surface modification	54
2.6.5	MD fouling	57

2.6.6	MD fouling control and cleaning	64
2.6.7	Pre-treatment for MD	64
2.6.8	Membrane flushing	70
2.6.9	Gas bubbling	72
2.6.10	Chemical cleaning	75
2.6.11	Management of seawater brine by hybrid MD process.....	76
	2.6.11.1 Membrane crystallization (MCr)	76
	2.6.11.2 MD/PRO hybrid process	77
2.7	Concluding remarks	78
CHAPTER 3		79
MATERIALS AND METHODS		79
3.1	Introduction	80
3.2	Experimental materials	80
3.3	Fabrication and modification techniques	80
	3.3.1 Electrospinning device	80
	3.3.2 CF ₄ plasma modification	82
3.4	Laboratory MD process.....	82
	3.4.1 DCMD experiments	82
	3.4.2 AGMD experiments	84
3.5	Membrane characterization and measurements.....	85
	3.5.1 Scanning Electron Microscope (SEM) and energy dispersive X-ray spectroscopy (EDX).....	85
	3.5.2 Transmission electron microscopy (TEM).....	86
	3.5.3 Liquid entry pressure (LEP)	86
	3.5.4 Contact angle (CA).....	87
	3.5.5 Sliding angle (SA)	87

3.5.6	Surface free energy (SFE).....	88
3.5.7	Surface roughness by atomic force microscopy (AFM).....	88
3.5.8	Tensile properties	89
3.5.9	Pore size and pore size distribution (PSD).....	89
3.5.10	Porosity	89
3.5.11	X-ray diffraction (XRD).....	90
3.5.12	X-ray photoelectron spectroscopy (XPS).....	90
3.5.13	Attenuated total reflectance – Fourier transform infrared spectroscopy (ATR-FTIR).....	90
3.5.14	Raman spectra.....	91
3.5.15	The Brunauer-Emmett-Teller (BET).....	91
CHAPTER 4		92
Hydrophobic/hydrophilic dual-layer electrospun nanofiber membrane for seawater desalination by membrane distillation application		92
4.1	Introduction.....	93
4.2	Experimental	97
4.2.1	Materials	97
4.2.2	Dope preparation	97
4.2.3	Electrospinning.....	98
4.2.4	AGMD test	99
4.3	Results and discussion	99
4.3.1	Morphology.....	99
4.3.2	Pore size and pore size distribution (PSD).....	104
4.3.3	Contact angle (CA).....	105
4.3.4	Liquid entry pressure (LEP).....	106
4.3.5	Mechanical properties.....	107

4.3.6	AGMD performances	109
4.3.6.1	<i>Effect of different support layer</i>	109
4.3.6.2	<i>Effect of feed temperature</i>	112
4.3.6.3	<i>Comparison with other AGMD flat-sheet membrane</i>	115
4.4	Concluding remarks	116
CHAPTER 5		119
	SUPERHYDROPHOBIC NANOFIBER MEMBRANE CONTAINING CARBON NANOTUBES FOR HIGH-PERFORMANCE MEMBRANE DISTILLATION	119
5.1	Introduction	120
5.2	Experimental methods	122
5.2.1	Materials	122
5.2.2	Dope preparation	122
5.2.3	Electrospinning parameters	123
5.3	Results and discussion	124
5.3.1	Morphology	124
5.3.2	Liquid entry pressure (LEP)	131
5.3.3	Membrane hydrophobicity	132
5.3.4	Membrane topography and roughness	134
5.3.5	Mechanical properties	136
5.3.6	Structural and chemical analysis	138
5.3.7	DCMD performance	140
5.3.7.1	<i>Effect of CNT concentration</i>	140
5.3.7.2	<i>Effect of salt concentration</i>	143
5.3.7.3	<i>Role of CNTs in the DCMD performance</i>	145
5.3.7.4	<i>Comparison with other studies</i>	147

5.4	Concluding remarks	149
CHAPTER 6		151
WATER DESALINATION USING GRAPHENE-ENHANCED ELECTROSPUN NANOFIBER VIA MEMBRANE DISTLLATION		151
6.1	Introduction	152
6.2	Model development	154
6.2.1	Two-dimensional dynamic model for AGMD module.....	154
6.2.2	Heat transfer in the hot and coolant channels	155
6.2.3	Mass transfer	156
6.3	Materials & methods	157
6.3.1	Materials	157
6.3.2	Dope preparation	158
6.3.3	Electrospinning.....	158
6.4	Results and discussion	159
6.4.1	Membrane characteristics and morphology	159
6.4.2	Structural and chemical characterization.....	166
6.4.3	Thermal and mechanical properties of the G/PH nanofiber.....	168
6.4.4	BET of the G/PH membrane	170
6.4.5	AGMD performance of the G/PH membrane	172
6.4.6	AGMD performance of the G/PH membrane	176
6.5	Concluding remarks	178
CHAPTER 7		180
BORON NITRIDE EMBEDDED MEMBRANE BY ELECTROSPINNING FOR SEAWATER DESALINATIO BY MEMBRANE DISTILLATION		180

7.1	Introduction	181
7.2	Materials and methods	182
7.2.1	Materials	182
7.2.2	Fabrication of OH-BNNPs by hydroxide-assisted ball milling	183
7.2.3	Dope preparation	183
7.2.4	Electrospinning of superhydrophobic BN-PH nanofiber	183
7.2.5	Evaluation of the BN-PH nanofiber membrane by AGMD	184
7.2.6	Characterization and measurements.....	184
7.3	Results and discussion	186
7.3.1	Morphology of the BN nanoparticles and BN-PH nanofiber	186
7.3.2	Chemical analysis.....	191
7.3.3	Mechanical properties.....	193
7.3.4	Long-term AGMD performances	194
7.4	Concluding remarks	197
CHAPTER 8		199
CF₄ PLASMA-MODIFIED OMNIPHOBIC ELECTROSPUN NANOFIBER MEMBRANE FOR TREATMENT OF RO BRINE FROM COAL SEAM GAS PRODUCED WATER		199
8.1	Introduction	200
8.2	Materials and methods	203
8.2.1	Dope preparation and electrospinning conditions	203
8.2.2	CF ₄ plasma modification	203
8.2.3	Air gap membrane distillation performance test	204
8.2.4	Contact angle (CA), sliding angle (SA) and surface free energy (SFE).....	205
8.3	Results and discussion	207
8.3.1	Morphologies and characteristics of the fabricated membranes	207

8.3.2	Plasma polymerization and deposition.....	209
8.3.3	Plasma polymerization and deposition.....	211
8.3.4	AGMD performance of omniphobic membrane	217
8.4	Concluding remarks	224
CHAPTER 9		226
CONCLUSIONS AND RECOMMENDATIONS.....		226
9.1	Conclusions	227
9.1.1	Janus-type electrospun nanofiber membrane	227
9.1.2	Nanomaterials-incorporated electrospun nanofiber membranes	229
9.1.3	CF ₄ plasma modified electrospun nanofiber membrane	232
9.2	Recommendations	234
REFERENCES.....		237

LIST OF FIGURES

Figure 1.1. Desalination capacities installed in the World and percentages of feed water type (Shahzad et al. 2017).....	2
Figure 1.2. Different configurations of membrane distillation (MD) application: (a) Direct contact MD (DCMD), (b) Air gap MD (AGMD), (c) Vacuum MD (VMD) and (d) Sweep gas MD (SGMD).	4
Figure 2.1. Timeline of the development of desalination technologies	15
Figure 2.2. (a) Total specific energy consumption (SEC) of each process for seawater desalination. (b, c) Comparison of the operating cost components for MSF and RO technologies (adapted from (Ghaffour, Missimer & Amy 2013; Shahzad et al. 2017)).	17
Figure 2.3. Schematic diagram of the FOHC hybrid desalination demonstration plant (adapted from (Chekli et al. 2016)).....	21
Figure 2.4. Different forms of wettability of a membrane: (A) non-wetted, (B) surface-wetted, (C) partial-wetted, and (D) fully-wetted (adapted from (Gryta 2007)).	35
Figure 2.5. (a) Bottom view of tri-bore spinneret and (b) cross sections of tri-bore hollow fiber membrane (adapted from (Luo et al. 2014)).	45
Figure 2.6. Since 1990, annual publication on the electrospinning as searched via Scopus.	46
Figure 2.7. Schematic illustration of (a) electrospinning device and the Taylor cone formation (adapted from (Ahmed, Lalia & Hashaikeh 2015; Baji et al. 2010))......	47

Figure 2.8. Average pore size of the membranes used in different membrane processes (Lalia, Kochkodan, et al. 2013).....	49
Figure 2.9. (a, b) Surface SEM images of (a) 20 wt% PVDF-co-HFP and (b) 10 wt% PVDF-co-HFP with 10 wt% TiO ₂ electrospun membranes and (c, d) cross-sectional SEM images of (c) PVDF-co-HFP single-layer and (d) PVDF-co-HFP/PAN dual-layer electrospun membranes (adapted from (Lee, An, et al. 2016; Tijing, Woo, et al. 2014)). ..	54
Figure 2.10. (a) The effect of fouling on the temperature distribution of DCMD membrane, and; microscopic images of membranes fouled by (b) CaCO ₃ and (c) protein, and (c) a virgin (un-fouled) membrane (Figures b-d are adapted from (Gryta 2008)). ..	60
Figure 2.11. Factors affecting membrane fouling: (a) foulant characteristics (concentration, molecular size, solubility, diffusivity, hydrophobicity, charge, etc.); (b) membrane properties (hydrophobicity, surface roughness, pore size and PSD, surface charge, surface functional groups); (c) operational conditions (flux, solution temperature, flow velocity), and; (d) feed water characteristics (solution chemistry, pH, ionic strength, presence of organic/inorganic matters). ..	61
Figure 2.12. The fouling sites on a membrane can be divided into surface fouling (external) or pore blocking (internal).....	62
Figure 2.13. Schematic representation of the different fouling mechanisms according to fouling material found in MD. In the real world processes, fouling usually occurs as mixed fouling, i.e., the combination of different of fouling mechanisms happening	

simultaneously. The dotted lines in the diagram with areas a, b, c and M show the different instances of mixed fouling between two or more fouling mechanisms. 63

Figure 2.14. Permeate flux and feed concentration of CaSO₄ versus time during five repetitive DCMD tests with membrane flushing after each test. A fresh 2000 mg/L CaSO₄ was used after each test (adapted from (Nghiem & Cath 2011)). 72

Figure 2.15. Schematic of the (a) air inlet position in the feed side of the MD module and the (b) photographic image of the air nozzle (adapted from (Chen et al. 2013)). 74

Figure 2.16. MD/PRO hybrid desalination demonstration plant (adapted from (Drioli, Ali & Macedonio 2015) and www.glovalmvp.org). 77

Figure 3.1. Schematic diagram of the electrospinning device: (a) syringe pump, (b) polymer solution, (c) tip, (d) nanofiber, (e) collector, (f) plate and (g) high voltage power supply. 81

Figure 3.2. Schematic illustrations of the direct contact membrane distillation (DCMD) system. 83

Figure 3.3. Schematic diagram of air gap membrane distillation (AGMD) process: (a) membrane, (b) air-gap, (c) condenser, (d) flow meter and (e) pump 85

Figure 3.4. Schematic lay-out of the LEP apparatus (Woo et al. 2016). 87

Figure 4.1. High and low magnification surface (a, c) and cross-sectional (b, d) SEM images of (a, b) neat PH (M1) and (c, d) heat-pressed PH (M2) membranes. 101

Figure 4.2. SEM images of the active layer made of PH (a1-c1), cross-section (a2-c2) and hydrophilic support layer (a3-c3) of the dual-layer membranes: (a) M4 (PH/PAN), (b) M6 (PH/N6), and M8 (PH/PVA).	103
Figure 4.3. Pore size distribution of the membranes: (a) M1, M2 and C-PVDF, (b) M3 and M4, (c) M5 and M6, and (d) M7 and M8.....	105
Figure 4.4. Schematic illustrations of water drops on the active-layer of the electrospun nanofiber membranes: (a) without heat-press process, and (b) with heat-press process.	106
Figure 4.5. Stress-strain curves of the electrospun nanofiber membranes and commercial membrane.....	109
Figure 4.6. (a) Flux and (b) salt rejection performance of the commercial PVDF membrane and the heat-pressed electrospun nanofiber membranes.....	112
Figure 4.7. (a) Flux and (b) salt rejection performance of the different temperature using the PVDF-HFP/Nylon-6 membrane.....	113
Figure 4.8. Surface SEM images of the membrane after 20 h of test at feed site temperature of: (a) feed – 70 °C and (b) feed – 80 °C.....	115
Figure 5.1. Morphological images and corresponding fiber size distributions of (a, d) PH and (b, e) 5CNT nanofiber, and (c) commercial PVDF membranes. Also shown is the (f) pore size distribution of PH and C-PVDF as measured by porometry	126
Figure 5.2. Cross-sectional SEM image of the 5CNT nanofiber composed of a thin top 5wt% CNT/PH electrospun layer and a thicker bottom neat PH electrospun layer.....	129

Figure 5.3. (a) Liquid entry pressure and contact angle measurements, and (b) schematic representation of the structure of the neat and CNT-incorporated nanofiber structures, (c) SEM images showing the beads and CNTs on the fiber, and (d) SEM image of the pristine CNTs used in this study	133
Figure 5.4. AFM (2D and 3D) images of the (a) neat, (b) 1CNT and (c) 5CNT nanofibers and (d) the commercial flat sheet membrane	135
Figure 5.5. (a) Typical stress-strain curves of the neat (i) PH, (ii) 5CNT, and (iii) C-PVDF membranes	137
Figure 5.6. (a) Raman, (b) XRD, (c) FTIR, and (d) EDX spectra results of the PH and 5CNT nanofiber membranes	140
Figure 5.7. Flux performance of the commercial PVDF membrane and the electrospun nanofibrous membranes at different CNT concentrations using (a) deionised water and (b) 35 g/L feed.....	143
Figure 5.8. Effect of feed type on the DCMD performance of different membranes tested in the present study: (a) deionised water, (b) 35 g/L NaCl and (c) 70 g/L NaCl	145
Figure 5.9. Schematic depiction of the working mechanism of CNT/PH nanofiber membrane for MD application in the present study.....	147
Figure 6.1. Schematic diagram of the AGMD module structure	154
Figure 6.2. Surface SEM images of the G/PH and neat PH electrospun nanofiber membranes: (a) neat PH, (b) G1PH, (c) G3PH, (d) G5PH, (e) G7PH and (f) G10PH.	160

Figure 6.3. TEM images of the G/PH electrospun nanofiber membranes: (a) G5PH, (b) G7PH and (c) G10PH.....	162
Figure 6.4. Average contact angle of the fabricated G/PH and neat PH electrospun nanofiber membranes.....	163
Figure 6.5. AFM images of (a) the PH18 and (b) the G5PH membranes. The mean roughness (Ra) of the PH18 and G5PH membranes was $0.623 \pm 0.01 \mu\text{m}$ and $0.719 \pm 0.03 \mu\text{m}$, respectively.....	164
Figure 6.6. Surface and cross-section morphologies of the G5PH electrospun nanofiber membrane (a and b) by SEM and (c and d) surface morphology of the G5PH electrospun nanofiber membrane by TEM.....	165
Figure 6.7. (a) EDX and (b) C/F ratio by EDX of the G/PH and neat PH electrospun nanofiber membranes.....	166
Figure 6.8. XRD spectra of (a) neat PH and G5PH membranes, and (b) graphene powder; (c) FTIR peaks of neat PH and G5PH membranes, and; (d) Raman spectra of the G5PH membrane and graphene powder.....	167
Figure 6.9. (a) TGA and (b) stress-strain curves of the G5PH and neat PH electrospun nanofiber membranes.....	169
Figure 6.10. Nitrogen adsorption-desorption isotherms (a) BJH pore size distributions and (b) for G5PH and neat PH membranes and (c) nitrogen adsorption energy distributions on G5PH and neat PH membranes.....	171

Figure 6.11. Flux and salt rejection performances of the G/PH and neat PH membranes for 20h operation (Inlet temp at feed = 60°C; Inlet temperature at coolant = 20°C).... 173

Figure 6.12. (a) Flux and (b) salt rejection of the G5PH electrospun nanofiber membrane and commercial PVDF membrane 174

Figure 6.13. Schematic of the effect of graphene on the membrane for AGMD process 175

Figure 6.14. Experimental and predicted flux for commercial PVDF, (b) Predictions for G5PH flux by the AGMD models with and without the presence of graphene sheet and (c) comparison of membrane distillation coefficients for G5PH..... 176

Figure 7.1. (a) Low-magnification TEM image of OH-BNNPs, (b) layer to layer distance of OH-BNNPs, (c) selected-area diffraction (SAED) patterns, (d, e) surface SEM images of the as-spun neat PH and the BN-PH electrospun nanofiber membranes, respectively and (f) the corresponding EDX elemental distribution of B and K in the BN-PH nanofiber samples. 187

Figure 7.2. SEM images of (a) the neat PH and (b) the BN-PH electrospun nanofiber membranes and corresponding fiber size distributions..... 188

Figure 7.3. Pore size distribution of the neat PH and BN-PH electrospun nanofiber membranes. The mean pore sizes of the neat PH and BN-PH are both 0.72 μm 189

Figure 7.4. Atomic force microscopy (AFM) images of (a) the neat PH and (b) the BN-PH electrospun nanofiber membranes. The mean roughness (R_a) of the neat PH and BN-PH membranes was 353.0 ± 14 nm and 515 ± 21 nm, respectively. The maximum

vertical distance between the highest data points (R_{\max}) of the neat PH and BN-PH membranes was 2233.5 ± 208.5 nm and 4170.5 ± 236.5 nm, respectively. 190

Figure 7.5. (a) Raman spectroscopy, (b) XRD patterns of the OH-BNNPs, neat PH and BN-PH electrospun nanofibers, (c, d) N1s and B1s narrow XPS scan of the OH-BNNPs and BN-PH electrospun nanofiber and (e) FT-IR of the OH-BNNPs, neat PH and BN-PH electrospun nanofibers. 193

Figure 7.6. (a) Stress-strain curves and (b) ultimate tensile strength (UTS) and tensile modulus of the neat PH and BN-PH electrospun nanofiber membranes 194

Figure 7.7. Water vapor flux and conductivity performances of (a) the neat PH electrospun nanofiber, (b) the commercial PVDF and (c) the BN-PH nanofiber membranes with feed solution containing 3.5 wt% NaCl in the AGMD tests. The feed and coolant temperatures were 60°C and 20°C, respectively. The flow rates of all AGMD tests were both maintained at 12 L/h in the feed and coolant stream. The initial water vapor flux of the neat PH, commercial PVDF and BN-PH membranes were 11.42 LMH, 6.13 LMH and 18.76 LMH, respectively. 196

Figure 8.1. Surface SEM images of the neat and CF₄ modified electrospun nanofiber membranes: (a) Neat, (b) P/CF-5, (c) P/CF-10, (d) P/CF-15, (e) P/CF-20, (f) P/CF-30 and (g) P/CF-60 208

Figure 8.2. (a, c, e) XPS survey scans and (b, d, f) carbon spectra of (a, b) the neat, (c, d) P/CF-15 and (e, f) P/CF-60 electrospun nanofiber membranes 211

Figure 8.3. Contact and sliding angle measurements of the neat and CF₄ modified electrospun nanofiber membranes at different treatment times 213

Figure 8.4. Contact angles of the neat and CF ₄ modified electrospun nanofiber membranes by ethylene glycol ($\gamma = 47.7$ mN/m), mineral oil ($\gamma = 30.0$ mN/m) and methanol ($\gamma = 22.7$ mN/m).....	215
Figure 8.5. Schematic illustrations of the neat and CF ₄ plasma modified electrospun nanofiber membranes.....	215
Figure 8.6. LEP of the neat and CF ₄ modified electrospun nanofiber membranes.....	217
Figure 8.7. (a) Flux and (b) rejection performances of the neat and CF ₄ modified electrospun nanofiber membranes by AGMD process using RO brine from CSG produced water as feed for 24 h.....	219
Figure 8.8. (a) Flux and salt rejection performances of the P/CF-15 nanofiber membrane by AGMD process using RO brine from CSG produced water as feed for 72 h and (b) normalized flux and salt rejection performances of the P/CF-15 nanofiber membrane by AGMD process using RO brine from CSG produced water with SDS additives as feed for 72 h.....	220
Figure 8.9. Normalized flux and (b) salt rejection of the C-PVDF and P/CF-15 electrospun nanofiber membranes by RO brine from CSG produced water with SDS as feed. The Porosity, mean pore size, membrane thickness, contact angle and LEP of the C-PVDF were 70.3%, 0.22 μm , 107.4 μm , 131.1° and 213.3 kPa, respectively.....	223
Figure 8.10. Comparison between the behavior of as-spun neat and CF ₄ plasma modified electrospun nanofiber membranes in AGMD process.....	224

LIST OF TABLES

Table 2.1. Classification and overview of desalination technologies	16
Table 2.2. Reports in literature on flat-sheet and hollow fiber membranes using phase separation techniques for MD application (Inlet temperatures: feed = 60°C, permeate = 20°C and feed water: 3.5 wt% NaCl).....	42
Table 2.3. Reports in literature on electrospun membrane for various membrane-based water filtration treatment applications	49
Table 2.4. Reports in literature on electrospun nanofiber membrane for MD application (Inlet temperatures: feed = 60°C, permeate = 20°C).	52
Table 2.5. Reports in literature on modified membranes using various modification techniques for MD application (Inlet temperatures: feed = 60°C, permeate = 20°C). ...	56
Table 2.6. Some pretreatment strategies for MD application reported in literature	70
Table 4.1. Reports in literature on hydrophobic/hydrophilic dual-layer membrane for MD application (Inlet temperatures: feed = 60°C, permeate = 20°C).....	95
Table 4.2. Electrospinning conditions used in the present study	99
Table 4.3. Characteristics of the membranes used in this study	102
Table 4.4. Comparison of results using different membranes in AGMD application ..	116
Table 5.1. Dope compositions used for electrospinning in the present study.....	123
Table 5.2. Properties of the commercial and fabricated nanofiber membranes.....	127

Table 5.3. Mechanical properties of the neat and 5 wt% CNT/PH nanofiber membranes, and the commercial PVDF membrane	138
Table 5.4. MD performance of the present study in comparison with other reports in literature at the following DCMD conditions: Feed/permeate inlet temperatures: 60/20°C; 35 g/L NaCl feed solutions.	148
Table 6.1. Electrospinning conditions of the G/PH and neat PH nanofiber membranes	159
Table 6.2. Characteristics of the neat and G/PH electrospun membranes and commercial membrane.....	161
Table 7.1. Characteristics of the neat PH and BN-PH electrospun nanofiber membranes used in the present study	188
Table 8.1. Characteristics of RO brine from CSG produced water	205
Table 8.2. Membrane codes and characteristics of the neat and CF ₄ modified electrospun nanofiber membrane	209
Table 8.3. Surface compositions of the neat and CF ₄ treated electrospun nanofiber membranes (at. %)	210
Table 8.4. Contact angle of diiodomethane and surface free energy (SFE) of the CF ₄ modified and neat electrospun nanofiber membranes.....	214

Table 8.5. Contact angle and LEP of the electrospun nanofiber membranes before and after operations by AGMD using RO brine from CSG produced water containing SDS as feed223