POLYAMIDE THIN FILM NANOCOMPOSITE MEMBRANE INCORPORATED WITH CARBON NANOTUBES/GRAPHENE OXIDE FOR CARBON DIOXIDE REMOVAL

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DEDICATION

This thesis is dedicated to my beloved mother, who taught me to be firm in my principle but flexible with my perspective by amassing knowledge from diverse sources. I would also like to dedicate my work to my father, who taught me that perseverance and hard work are integral parts of success as even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

Carbon capture and storage (CCS) is a feasible option to reduce the atmospheric carbon dioxide (CO_2) concentration that has is rising in an alarming rate. However, the implementation of CCS is hampered by the high operating cost that is associated with CO_2 removal from the emission sources. Membrane is one of the sustainable technologies that holds much potential to drive down this cost. Thin film nanocomposite (TFN) is a particularly attractive membrane as its nanomaterial-embedded ultrathin selective layer could permit good separation efficiency at high rate of mass transport. Even though carbon nanotube (CNT) is a prized nanomaterial that could greatly elevate the membrane strength and separation performance, its high aggregation tendency limits its usefulness for the development of CNT-based TFN. In this study, the dispersibility of CNT was improved through amino functionalization and addition of graphene oxide (GO). Amino groups on the surface of functionalized CNT (ACNT) sterically hindered the nanotubes from bundling while the amphiphilic GO acted as dispersant that prevented clustering of the nanomaterials. This allowed successful incorporation of the nanofillers into the membrane ultrathin selective layer during interfacial polymerization (IP). The impacts of nanofillers loading, combination and ratio on IP were systematically explored. The nanomaterials' properties such as hydrophilicity and adsorptivity was found to affect the reactivity of IP which in turn altered the characteristics of selective layer. Gas separation results show that incorporation of ACNT improved the membrane selectivity due to its narrow openings that favor transport of the small CO_2. On the other hand, incorporation of GO led to the formation of relatively thin selective layer which improved the membrane gas permeability. Co-incorporation of ACNT with GO boosted the permeability and CO_2/nitrogen selectivity of TFN by 30% and 60%, respectively, compared to the control membrane. The resulted TFN was also more reproducible and stable under elevated temperature and exposure to air. The outcome of this study suggested that synergetic incorporation of ACNT and GO provides an additional degree of freedom to control the formation of selective layer as compared to single-filler incorporation. TFN containing these two geometrically different carbon-based nanomaterials showed fascinating properties and deserves further in-depth development.

ABSTRAK

Pemerangkapan dan penyimpanan karbon (CCS) merupakan kaedah yang mampu dilaksanakan untuk mengurangkan kepekatan karbon dioksida (CO_2) dalam atmosfera yang kian meningkat pada kadar yang membimbangkan. Walau bagaimanapun, pelaksanaan CCS terhalang oleh tingginya kos pengendalian yang dikaitkan dengan proses penyingkiran CO_2 daripada punca pelepasan. Membran merupakan salah satu teknologi lestari yang berpotensi tinggi untuk mengurangkan kos ini. Komposit nano filem tipis (TFN) adalah membran yang sangat menarik kerana lapisan selektifnya amat nipis dan mengandungi bahan nano yang mampu memberikan kecekapan pemisahan yang baik pada kadar pengangkutan jisim yang tinggi. Walaupun tiub nano karbon (CNT) adalah bahan nano berharga yang boleh meningkatkan kekuatan serta prestasi pemisahan membran, kecenderungan penggumpalan yang tinggi menghadkan kegunaannya untuk pembangunan TFN berasaskan CNT. Dalam kajian ini, penyerakan CNT telah ditambah baik melalui pemfungsian amino dan penambahan grafin oksida (GO). Kumpulan-kumpulan amino di permukaan CNT yang telah difungsikan (ACNT) akan menghalang secara sterik tiub-tiub nano tersebut daripada terikat manakala GO yang bersifat amfifilik bertindak sebagai penyerak yang menghalang pengelompokan bahan-bahan nano. Ini menjayakan penggabungan pengisi-pengisi nano ke dalam lapisan selektif membran yang amat nipis semasa pempolimeran antara muka (IP). Kesan-kesan daripada muatan, kombinasi dan nisbah pengisi-pengisi nano kepada IP telah diterokai secara sistematik. Sifat-sifat bahan-bahan nano seperti kehidrofilikan dan keterserapan didapati boleh menjejaskan kereaktifan IP yang kemudian mengubah ciri-ciri lapisan selektif. Keputusan pemisahan gas menunjukkan bahawa penggabungan ACNT telah meningkatkan kememilihan membran kerana bukaannya yang sempit memudahkan pengangkutan CO_2 yang kecil. Sebaliknya, penggabungan GO membawa kepada pembentukan lapisan selektif yang lebih tipis yang mana telah meningkatkan kebolehtelapan membran terhadap gas. Penggabungan ACNT bersama GO telah menggalakkan kebolehtelapan dan kememilihan CO_2/nitrogen TFN, masing-masing sebanyak 30% dan 60%, berbanding membran kawalan. TFN yang dihasilkan juga lebih mampu dihasilkan semula dan stabil apabila terdedah kepada udara serta suhu yang tinggi. Hasil kajian ini mencadangkan bahawa penggabungan ACNT dan GO secara sinergi memberikan darjah kebebasan yang lebih tinggi untuk mengawal pembentukan lapisan selektif berbanding penggabungan pengisi secara tunggal. TFN yang mengandungi kedua-dua bahan nano berasaskan karbon yang berbeza geometri ini menunjukkan ciri-ciri menarik dan patut dibangunkan secara lebih mendalam.

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LIST OF ABBREVIATIONS

2D	-	two dimensional
3D	-	three dimensional
ACNT	-	amino-functionalized carbon nanotube
AFM	-	atomic force microscopy
AGO	-	amino-functionalized graphene oxide
ATR	-	attenuated total reflection
CA	-	cellulose acetate
CCS	-	carbon capture and storage
CE	-	closed environment
CMS	-	carbon molecular sieve
CNT	-	carbon nanotube
CVD	-	chemical vapor deposition
DGBAmE	-	diethylene glycol bis(3-aminopropyl) ether
DNMDAm	-	3,3'-diamino-N-methyldipropylamine
EO	-	ethylene oxide
E-spun	-	electrospun
FESEM	-	field emission scanning electron microscopy
FO	-	forward osmosis
FTIR	-	Fourier transform infrared spectroscopy
GO	-	graphene oxide
GPU	-	gas permeation unit
HCl	-	hydrochloric acid
ID	-	internal diameter
IL	-	ionic liquid
ImGO	-	imidazole modified GO
IP	-	interfacial polymerization
IPC	-	isophthaloyl chloride
l-Phe	-	*l*-phenylalanine
MEDA	-	N-methyldiethanolamine
MMM	-	mixed matrix membrane

MOF	-	metal organic framework
MPD	-	m-phenylenediamine
MWCNT	-	multi-walled carbon nanotube
MWCO	-	molecular weight cut-off
nCNT	-	m-phenylenediamine modified carbon nanotube
NF	-	nanofiltration
nHNT	-	modified halloysite nanotube
NMP	-	N-methyl-2-pyrrolidone
OCNT	-	oxidized multiwalled carbon nanotube
OD	-	outer diameter
OE	-	opened environment
PA	-	polyamide
PC	-	polycarbonate
PDMS	-	polydimethylsiloxane
PEA	-	polyetheramine
PEG	-	polyethylene glycol
PEI	-	polyetherimine
PEO	-	polyethylene oxide
PES	-	polyethersulfone
PI	-	polyimide
PILM	-	poly(ionic liquid) membrane
PIP	-	piperazine
POS	-	polysiloxane
PRG	-	porous reduce graphene oxide
PSF	-	polysulfone
PVA	-	polyvinylalcohol
PVAm	-	polyvinyl amine
PVDF	-	polyvinylidene fluoride
PVP	-	polyvinyl pyrrolidone
r.t.	-	room temperature
RH	-	relative humidity
RO	-	reverse osmosis
RTIL	-	room temperature ionic liquid

SDS	-	sodium dodecyl sulfate
SILM	-	supported ionic liquid membrane
STP	-	standard pressure and temperature
SWCNT	-	single-walled carbon nanotube
Syn. Con.	-	synthesis condition
TEM	-	transition electron microscopy
TETA	-	triethylenetetramine
TFC	-	thin film composite
TFN	-	thin film nanocomposite
TMC	-	trimesoyl chloride
TNT	-	titanium nanotube
UF	-	ultrafiltration
UV-Vis	-	ultraviolet-visible spectrophotometry
WCA	-	water contact angle
XRD	-	X-ray diffraction spectroscopy

LIST OF SYMBOLS

$\ddot{R}_{x:y}$	-	ratio between filler '*x*' and filler '*y*'
$\bar{P}$	-	permeability
θ_{XRD}	-	diffraction angle of sample
θ_{XRD}	-	X-ray diffraction angle
$\ddot{F}$	-	type of filler combination
$\ddot{L}$	-	filler loading
ΔP	-	percentage change in permeance
ΔSP	-	difference in separation performance in term of permeance, permeability or selectivity
$\Delta\alpha$	-	percentage change in selectivity
A	-	effective membrane area
A_f	-	absorbance of UV by feed solution
A_p	-	absorbance o UV by permeate solution
Aq	-	aqueous phase
C	-	packing constant in Washburn's equation
D	-	diffusivity
d_k	-	kinetic diameter
dp	-	pore diameter
d-spacing	-	interlayer distance
I_d	-	intensity of D-band in Raman spectrum
I_g	-	intensity of G-band in Raman spectrum
Kn	-	Knudsen number
L	-	length
l	-	thickness of membrane selective layer
l_F	-	thickness of PA film above support
l_P	-	penetration depth of PA into support
l_{Pavg}	-	average PA penetration at different synthesis condition
l_T	-	total thickness of selective layer of fabricated composite membrane ($l_F + l_{Pavg}$)
m	-	mass

M	-	molar
n	-	number of pores
N_p	-	molar flow rate through single pore
N_t	-	total molar flow rate
Og	-	organic phase
P	-	permeance
Q	-	volumetric flowrate of gas
R	-	universal gas constant
R_a	-	average plane roughness
r_m	-	average pore radii
R_{p-v}	-	peak-to-valley roughness
S	-	solubility
SP	-	separation performance in term of permeance, permeability or selectivity
T	-	temperature
t	-	time
T_g	-	glass transition temperature
Vap_{hex}	-	n-hexane vapor saturated environment
α	-	selectivity
γ	-	surface tension
Δp	-	pressure differential
ζ	-	zeta potential
η	-	viscosity
θ_h	-	hexane contact angle
θ_w	-	water contact angle
λ_{Kn}	-	gas mean free path length
λ_{Raman}	-	irradiation wavelength in Raman spectroscopy
λ_{XRD}	-	irradiation wavelength in X-ray diffraction spectrometer
ρ	-	density

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

In recent years, awareness on the global warming has increased tremendously as people around the world began to feel the effects of dramatically raising earth temperature. Change in the world climate pattern and damage to the natural ecosystem are some of the devastations of global warming that affect our daily life (Powell and Qiao, 2006). The surge in anthropogenic activities including fossil fuel and coal combustion, natural gas exploration, biogas anaerobic digestion, and deforestation over the past century has resulted in the release of large amount of carbon dioxide (CO_2) into the atmosphere. This unregulated atmospheric CO_2 acts as an invisible blanket that retard the dissipation of heat from the earth, leading to accumulation of heat and consequently global warming. The global population is expected to expand close to 10 billion inhabitants by year 2050 (Lalia *et al.*, 2013), the increase in the world resources and energy demand is inevitable and will only bring about greater greenhouse gases emission (Adewole *et al.*, 2013). Hence, removal and mitigation of CO_2 emission are highly desired and thus becoming hot research topics (Lindau, Jönsson and Wimmerstedt, 1995). In addition to adverse environmental impacts, CO_2 also represents serious problem to the natural gas processing industry as it can lower the heating value of product stream and corrode the pipelines of transportation system due to its acidic nature (Zhang *et al.*, 2013).

CO_2 capture and storage (CCS) is well accepted by scientists and policy makers as a viable approach to mitigate this alarming issue (Dixon *et al.*, 2013; Pires *et al.*, 2011). However, separating this potent greenhouse component accounts for nearly 80% of the total cost involved in CO_2 sequestration (Choi, Kim and Lee, 2013; Favre, 2011). Therefore, development of economic means for capturing the CO_2 from the source of emission is crucial for successful implementation of CCS (Czyperek *et al.*,

2010). Membrane technology prevails as a viable option due to its design simplicity and modularity that ease scaling up process. Membrane separation is relatively energy efficient compared to conventional approaches such as absorption and cryogenic separation (Sanders *et al.*, 2013). Hybrid phase composite membrane known as mixed matrix membrane (MMM) is currently the most intensively studied membrane due to its capability to surpass the inherent performance limitations, i.e. Robeson's trade-off of polymeric material (Adewole *et al.*, 2013; Bastani, Esmaeili and Asadollahi, 2013). MMM consists of a dispersed phase of inorganic fillers within a polymeric host matrix. The fillers in used can be categorized as porous and non-porous materials. Porous materials include zeolite, carbon molecular sieve (CMS), activate carbon, metal organic framework (MOF) and carbon nanotube (CNT). Examples of non-porous materials are silica, titanium oxide (TiO_2) and fullerene particles.

In a typical MMM, fillers can occupy up to 30% of the membrane total weight (Bastani *et al.*, 2013; Castro-Muñoz and Fíla, 2019; Kim and Nair, 2013; Shahid *et al.*, 2015). Since the dense skin of membrane is the primary layer responsible for separation function, embedment of nanofillers should be focused within the skin layer in order to fully harness the nanofillers potential and achieve a well-balance productivity-to-cost ratio. Despite the colossal amount of studies (Guo *et al.*, 2015; Kiadehi *et al.*, 2015b; Quan *et al.*, 2017; De Sitter *et al.*, 2014; Zhang *et al.*, 2019) and reviews (Cui, Kundalwal and Kumar, 2016; Swain *et al.*, 2017; Yao and Wang, 2014; Zhang *et al.*, 2013) that showed promise of membrane performance enhancement though the incorporation of nanofillers, controlling the distribution of nanofillers within the MMM has been proven to be extremely difficult (Aroon *et al.*, 2010a). In fact, most of the nanofillers incorporated through single-step phase inversion technique tend to distribute within the porous support layer of MMM (Chung *et al.*, 2007; Khulbe, Feng and Matsuura, 2008; Tan *et al.*, 2019). This implied that a large portion of these precious nanomaterials would be wasted as they do not contribute actively in the separation process

In regard to this concern, the two-step approach involved in the fabrication of thin film nanocomposite (TFN) membrane is the key to overcome the constrains with fillers distribution (Seman, Khayet and Hilal, 2010). The selective skin layer of TFN is deposited on as pre-formed porous support layer. This two-step approach provides the flexibility to discretely manipulate the composite layers to attain the preferred properties (Albo, Wang and Tsuru, 2014; Misdan *et al.*, 2013). Through this approach, the distribution of nanofillers can be precisely controlled, i.e. deposited solely within the skin layer of the membrane or throughout the substrate. Among the coating techniques developed for the fabrication of the TFN skin, interfacial polymerization (IP) is very interesting because this technique is highly scalable and is already widely adopted in the industry (Liu *et al.*, 2011; Yu *et al.*, 2010; Yu *et al.*, 2011). Additionally, the availability of vast variety of monomers enables the thin film chemistry to be freely tuned (Espeso *et al.*, 2006; Song *et al.*, 2005).

Contemporary, much of the development on IP fabricated TFN centered around the field of water treatment. The deployment of IP to produce gas separation membrane still lags behind (Li *et al.*, 2013). In fact, polyamide (PA) layer synthesized through IP possesses many attractive characteristics for gas separation such as high mechanical strength, thermal stability and chemical resistivity (Albo *et al.*, 2014; Petersen and Peinemann, 1997). More importantly, PA thin film is found capable of promoting CO_2 transport across the membrane (Andrew Lee, Stevens and Kentish, 2013). Hence, there are enormous opportunities for in-depth research and development of IP fabricated TFN for gas separation application which can be explored in this work.

1.2 Problem Statements

Low energy consumption of TFN is conferred by the membrane low feed pressure requirement to afford separation while retaining a high output . This is made possible by the extremely thin skin of TFN. Apart from reducing the thickness of selective layer to ease mass transport for high gas permeability, alteration to the polymer network such as increasing the mobility of polymer chain, pore size and porosity could be made. However, doing so diminishes the sieving ability of the selective layer. This permeability-selectivity trade-off is the fundamental limitation to the separation performance of a polymeric material. The contemporary strategy to negate this trade-off is by incorporating filler materials with superior separation capability into the polymer network. CNT is one of such materials. Once embedded into the skin layer, CNT functions as rapid transport channel that drives the diffusion of gas molecules across the membrane and resulted in high productivity. Besides, the nanotube precise and narrow aperture ensures good selectivity of the nanocomposite materials. Additionally, the incorporation of inorganic nanofillers has been known to elevate the physical and thermal stability of the polymeric matrix which can improve resistant to aging and plasticization (Kanehashi *et al.*, 2018). In this study, multi-walled carbon nanotube (MWCNT) was employed as the primary filler to enhance the performance of TFN.

Despite the advantages offered through the incorporation of MWCNT, past studies have witnessed poor compatibility between this nanofiller and the polymer matrix (Shen *et al.*, 2013). Moreover, MWCNT is prone to agglomeration due to its high aspect ratio (Annala, Lahelin and Seppälä, 2012; Goh *et al.*, 2010). This is a major hiccup to the development of nanocomposite membrane as the overall transport properties of the membrane are critically dependent on the nanoscale interface morphology of the membrane (Goh *et al.*, 2011; Kim *et al.*, 2016; Swain *et al.*, 2017). Ideally, good inorganic-polymer interaction and homogeneous nanofillers dispersion are favorable for fabrication of defect-free nanocomposite membrane especially when the nanofillers are incorporated into the ultrathin skin of TFN. Amino functionalization and co-dispersion of MWCNT with graphene oxide (GO) were adopted in this study to circumvent this challenge. Introduction of amino functional groups on the nanotube

surface reduces the inter-tube Van de Waals interaction and sterically hinders entanglement of MWCNT. Insertion of highly dispersive GO nanosheets can aid with the debundling and dispersion of the nanotubes in aqueous solution. Moreover, the presence of CO_2-affinitive functional groups such as amino can enhance the membrane overall CO_2 permeance whereas GO sheets can alter the tortuosity of the composite matrix which lead to better sieving property. It is of great interest in this research to investigate the impacts of incorporation of MWCNT, GO and their amino-functionalized counterparts (individually or as nanotube-nanosheet combination) on the TFN formation and CO_2 separation performance.

1.3 Research Objectives

Based on the aforementioned issues, this study sets out with the following objectives:

1. To study the effects of amino functionalization and GO co-dispersion on the dispersibility of MWCNT in aqueous solution containing diamine monomers.
2. To develop TFN membrane with different nanofillers loading, combination and ratio in the PA skin layer.
3. To investigate the CO_2 separation performance of PA-based TFN incorporated with nanotube-nanosheet in term of permeance and selectivity.
4. To study the stability of CO_2 separation performance of TFN at elevated temperature and exposure to air.

1.4 Research Scope

The scopes of study have been identified and are listed below:

For Objective 1:

(a) Oxidizing MWCNT (OCNT) using a mix of sulfuric acid (H_2SO_4)/nitric acid (HNO_3) under reflux condition.

(b) Synthesizing GO from graphite via modified Hummer's method.

(c) Functionalizing OCNT and GO with amino groups under basic condition to obtained amino-functionalized CNT (ACNT) and amino-functionalized GO (AGO) respectively.

(d) Characterizing the chemical properties of pristine and modified nanofillers via Fourier transform infrared spectroscopy (FTIR).

(e) Studying the morphology of nanofillers via transition electron microscopy (TEM) and atomic force microscopy (AFM).

(f) Characterizing the crystallinity and degree of distortion in the nanofillers graphitic structure via X-ray diffraction spectroscopy (XRD) and Raman spectroscopy respectively.

(g) Determining the hydrophilicity of nanofillers using Washburn's equation.

(h) Evaluating the dispersibility of nanoparticles in aqueous solution containing diamine monomers using optical microscope and zeta potential (ζ) analyzer.

(i) Determining the diamine adsorptivity of nanofillers via ultraviolet-visible spectrophotometry (UV-Vis)

For Objective 2:

(a) Preparing polysulfone (PSF) dope solution containing polyvinyl pyrrolidone (PVP) and N-methyl-2-pyrrolidone (NMP) as additive and solvent respectively.
(b) Fabricating PSF support membrane via solvent-nonsolvent phase inversion technique.
(c) Characterizing chemical properties, morphologies and surface roughness of PSF support via FTIR, field emission scanning electron microscopy (FESEM) and AFM, respectively.
(d) Determining the molecular weight cut-off (MWCO) of PSF support based on rejection of various proteins.
(e) Forming PA skin layer which is embedded with nanofiller atop PSF support via IP technique to obtain TFN.
(f) Manipulating nanofiller loading (0.00 mg/mL – 1.00 mg/mL), nanotube-nanosheet combination (OCNT-GO, OCNT-AGO, ACNT-GO, ACNT-AGO) and weight (*wt*) ratio of nanotube-nanosheet (3:1, 3:2, 3:3, 2:3 and 1:3).
(g) Characterizing chemical properties, morphologies and surface roughness of TFNs via FTIR, FESEM and AFM, respectively.

For Objective 3:

(a) Evaluating the effects of nanofiller incorporation parameters (loading, type of nanofiller, combination and ratio) on PA formation and TFN CO_2 separation performance using custom-built testing rig.

For Objective 4:

(a) Evaluating the performance stability of TFN at different operating temperature (30 – 70 °C) and exposure to air for a period of 20 days.

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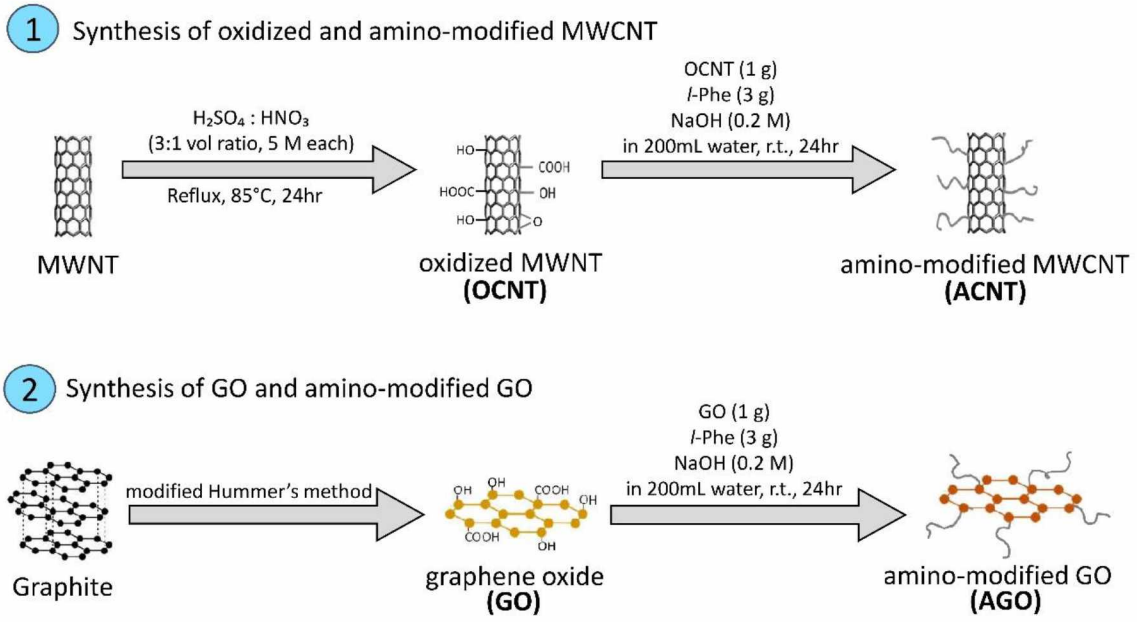
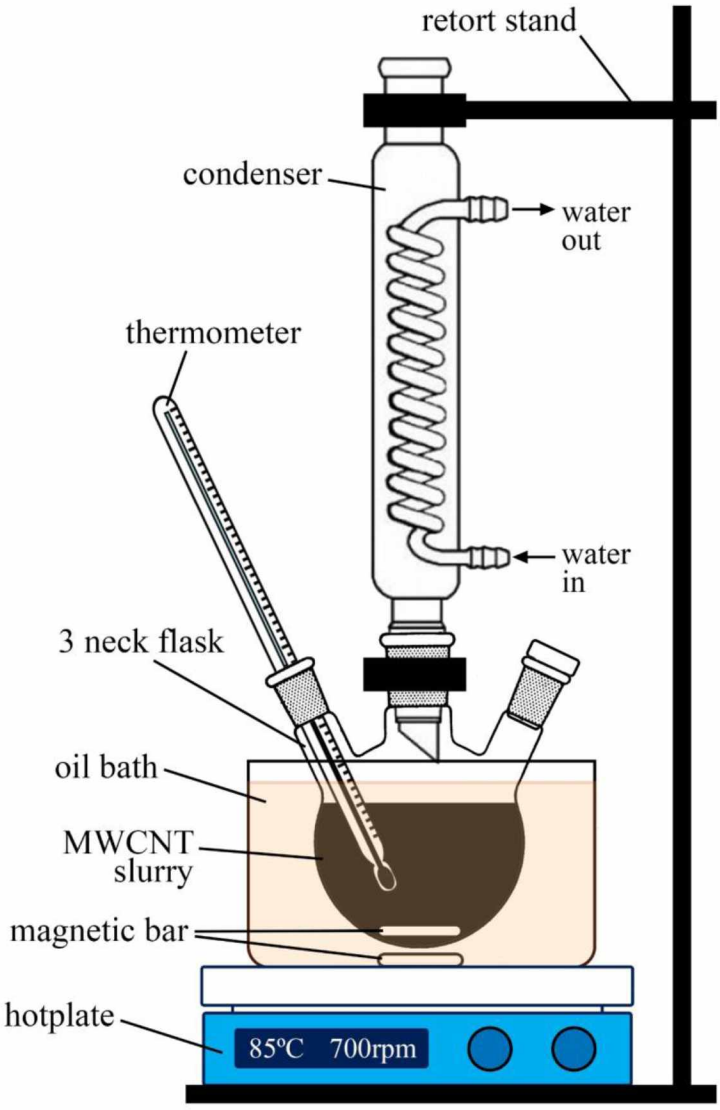
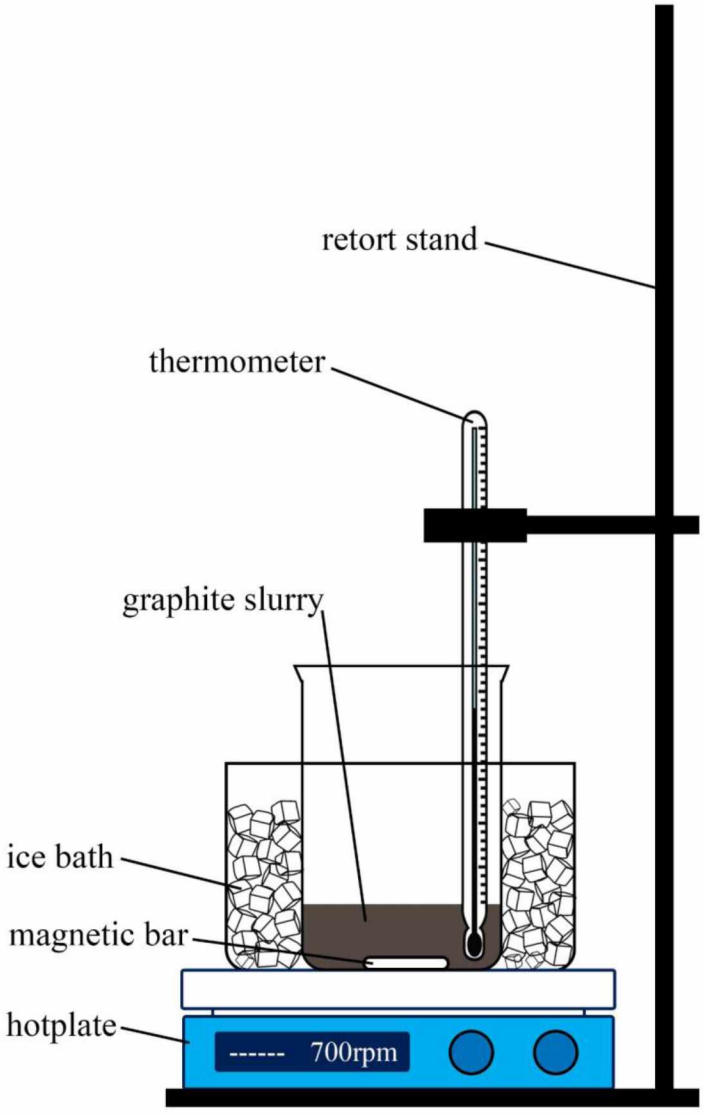
Appendix A Nanofillers Synthesis Procedures



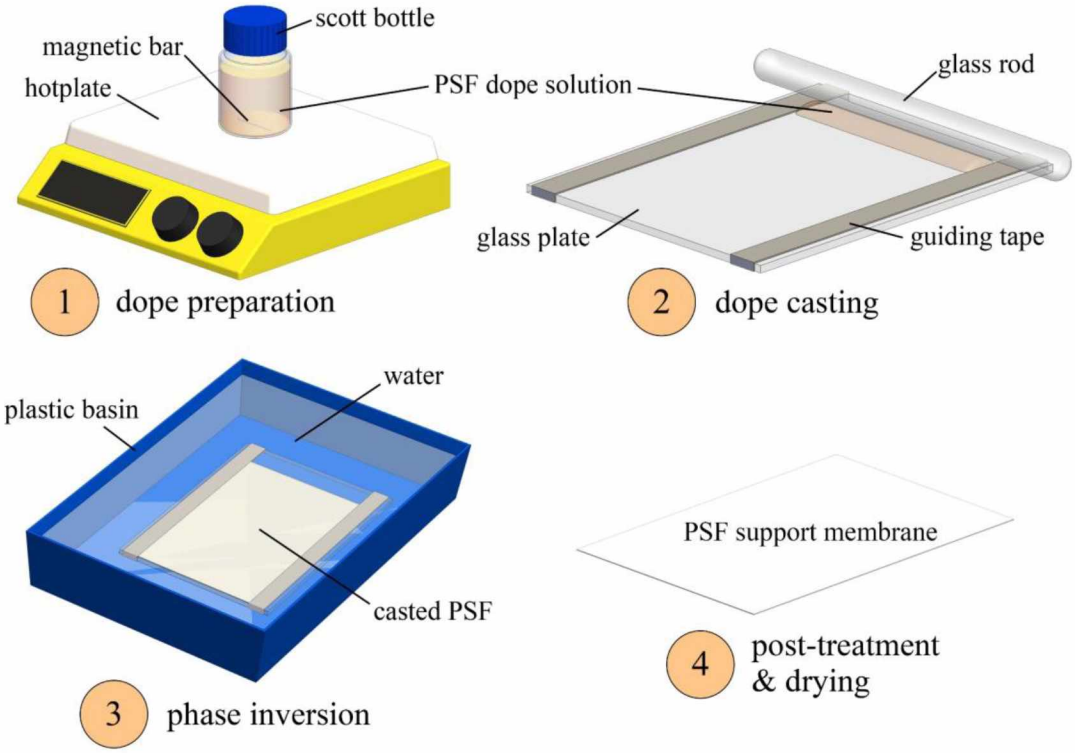
Appendix B Experimental Setup for MWCNT Oxidation



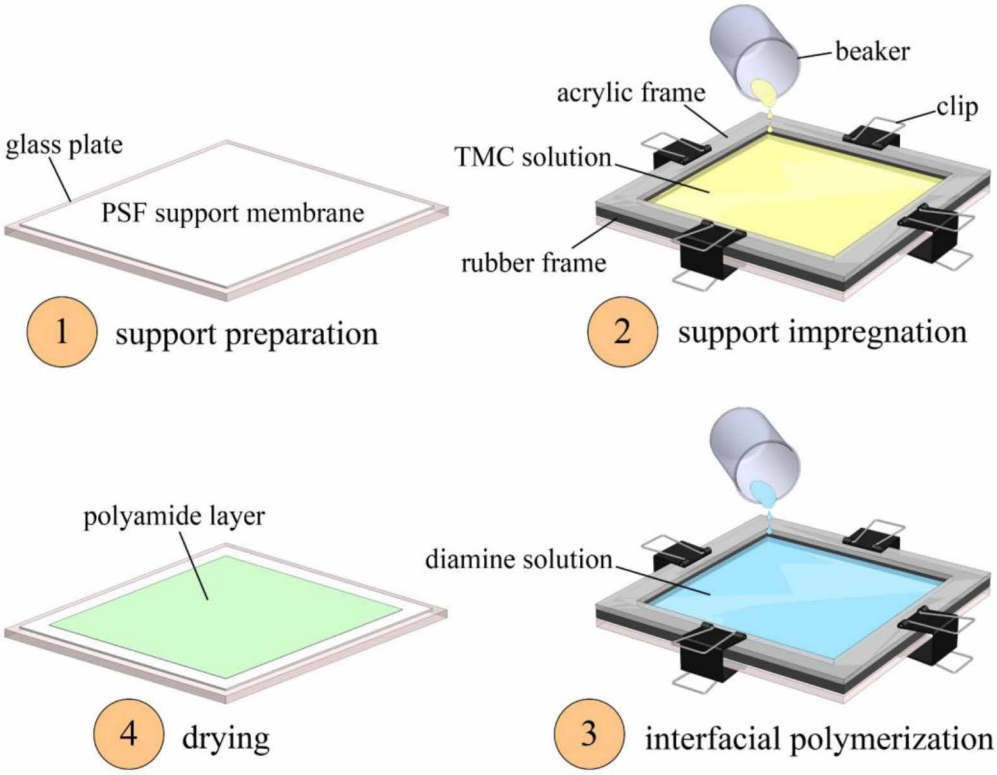
Appendix C Experimental Setup for Graphite Oxidation



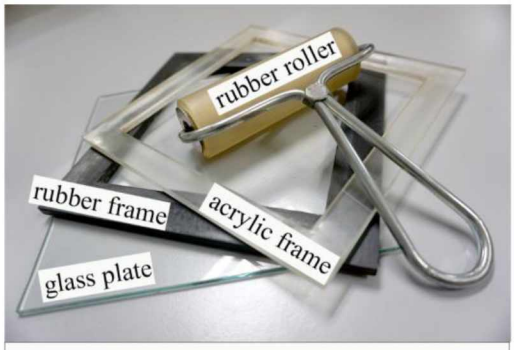
Appendix D Fabrication Steps of PSF Support



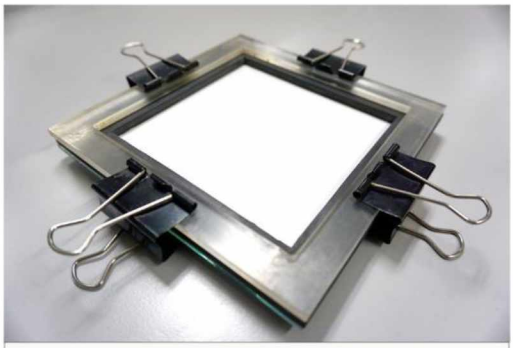
Appendix E Fabrication Steps of TFC or TFN



Appendix F Experimental Setups for Interfacial Polymerization



IP Setup

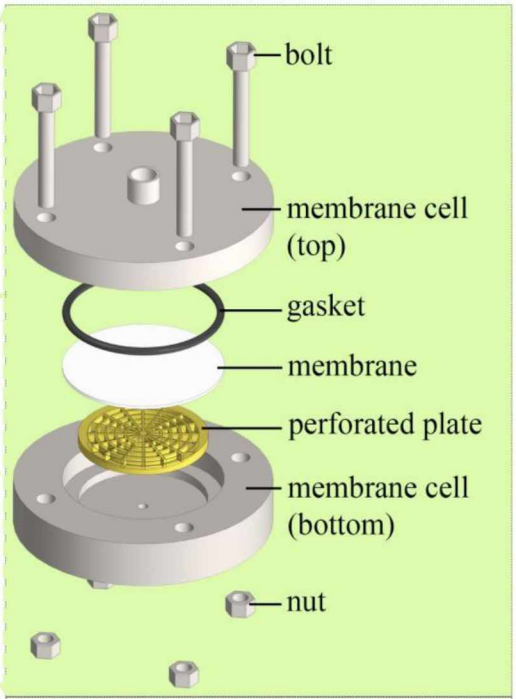


IP in opened environment

IP in closed environment

Appendix G Gas Permeation System

gas permeation test system



membrane cell setup

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