POLY(VINYLIDENE FLUORIDE)/MODIFIED CALCIUM CARBONATE NANOPARTICLE HOLLOW FIBER MEMBRANES FOR CARBON DIOXIDE REMOVAL

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ABSTRACT

The principal aim of this research work is to compensate for the shortcomings, especially pore wetting and mass transfer resistance, of polymeric membrane materials employed for carbon dioxide (CO_2) removal via gas-liquid membrane contactors. Calcium carbonate ($CaCO_3$) nanoparticles hydrophobically modified with octadecyl dihydrogen phosphaste were embedded in the polymer matrices to develop mixed matrix membranes (MMMs) with a well-tailored structure. Porous hydrophobic polyvinylidene fluoride (PVDF) mixed matrix hollow fiber membranes were fabricated via phase inversion method by incorporating $CaCO_3$ nanoparticles in various mixing ratios (10/100, 20/100 and 30/100 $CaCO_3$/PVDF). The effects of $CaCO_3$ nanoparticle loadings on the morphology, structure, and performance of the MMMs were investigated. The addition of $CaCO_3$ nanoparticles enhanced the surface roughness, permeation rate, porosity, and wettability resistance of the MMMs. Peak CO_2 absorption performance of 1.52×10^{-3} mol m^{-2} s^{-1} at 300 ml/min absorbent flow rate was achieved when 20/100 weight ratio of $CaCO_3$/PVDF was employed. However, further increase of the ratio resulted in MMMs with lower absorption performance. Moreover, a long-term stability study of the MMMs with the best CO_2 absorption flux showed no decline in performance in the initial 210 hours of operation, indicating the significant improvement caused by the addition of $CaCO_3$ nanoparticles into polymer matrix. From the physical CO_2 stripping tests point of view, similar trend of results were obtained. It was found that the CO_2 highest stripping flux of 1.8×10^{-2} mol m^{-2} s^{-1} and efficiency of 67% were achieved when 20/100 mixing ratio of $CaCO_3$/PVDF was employed, corresponding to its high gas permeation and effective surface porosity. For the purpose of further optimization, polymer concentration in dope solution was varied to study its effect on membrane characteristics. After the selection of MMM with the best CO_2 removal performance (in this case 20/100 $CaCO_3$/PVDF membrane), the MMMs with various polymer concentrations (16, 17, 18, and 19 wt.% PVDF) were prepared. Improvements in porosity and nitrogen permeance were recorded for P17, the MMM with 17 wt.% polymer concentration. Superior CO_2 absorption performance of 1.66×10^{-3} mol m^{-2} s^{-1} at 300 ml/min absorbent flow rate was also recorded. Moreover, testing P17 at various operating temperatures during CO_2 stripping process ranging from 27 °C to 100 °C was carried out. The maximum stripping flux of 2.57×10^{-2} mol m^{-2} s^{-1} at 2.3 m s^{-1} liquid velocity and 80 °C absorbent temperature was higher than the fluxes of the compared in-house and commercial membranes. Henceforth, the enhanced porosity of P17 coupled with its high wetting resistance suggests its potential for the efficient removal of CO_2 via gas-liquid membrane contactors.

ABSTRAK

Matlamat utama kajian ini adalah untuk mengatasi kekurangan, terutama dari segi pembasahan rongga dan rintangan pemindahan jisim, pada membran polimer yang digunakan dalam penyingkiran karbon dioksida (CO_2) menerusi penyentuh membran gas-cecair. Nanopartikel kalsium karbonat ($CaCO_3$) yang diubahsuai menggunakan oktadesil dihidrogen fosfat supaya bersifat hidrofobik diadun di dalam matriks polimer bagi menghasilkan membran matriks campuran (MMC) dengan struktur terubah suai yang lebih baik. Membran qentian berongga hidrofobik berpori dengan matriks campuran polivinilidena fluorida (PVDF) telah dihasilkan menerusi kaedah fasa songsangan dengan menggabungkan nanopartikel $CaCO_3$ pada pelbagai nisbah campuran (10/100, 20/100, dan 30/100 $CaCO_3$/ PVDF). Kesan muatan nanopartikel $CaCO_3$ terhadap morfologi, struktur, dan prestasi MMC telah dikaji. Penambahan nanopartikel $CaCO_3$ telah meningkatkan kekasaran permukaan, kadar telap, keliangan, dan rintangan kebasahan MMC. Prestasi penyerapan CO_2 puncak ialah 1.52×10^{-3} mol m^{-2} s^{-1} pada kadar alir bahan penyerap 300 ml/min yang dicapai apabila nisbah berat $CaCO_3$/PVDF pada 20/100 digunakan. Walau bagaimanapun, peningkatan nisbah terbabit telah menghasilkan MMC dengan prestasi penyerapan yang lebih rendah. Selain itu, kajian kestabilan jangka panjang terhadap MMC dengan fluks penyerapan terbaik CO_2 menunjukkan bahawa tiada penurunan prestasi ketika beroperasi selama 210 jam, menunjukkan peningkatan yang ketara berpunca daripada penambahan nanopartikel $CaCO_3$ ke dalam matriks polimer. Berdasarkan ujian CO_2 secara fizikal, keputusan yang serupa diperoleh. Fluks pelucutan CO_2 tertinggi pada kadar 1.8×10^{-2} mol m^{-2} s^{-1} dan kecekapan 67% dicapai apabila nisbah campuran $CaCO_3$/PVDF pada 20/100 digunakan, bersepadan dengan peresapan gas dan keliangan permukaan berkesan yang tinggi. Selanjutnya bagi proses pengoptimuman, kepekatan polimer dalam larutan dop telah diubahsui bagi mengkaji kesannya terhadap ciri-ciri membran. Selepas pemilihan MMC dengan prestasi terbaik penyingkiran CO_2 yang (dalam kes ini membran 20/100 $CaCO_3$/PVDF), MMC dengan pelbagai kepekatan polimer (16, 17, 18, dan 19 wt.% PVDF) disediakan. Peningkatan keliangan dan peresapan nitrogen direkodkan untuk P17, iaitu MMC dengan kepekatan polimer 17 wt.%. Prestasi unggul penyerapan yang terbaik CO_2 ialah 1.66×10^{-3} mol m^{-2} s^{-1} pada kadar alir serap 300 ml / min juga direkodkan. Selain itu, pengujian terhadap P17 pada pelbagai suhu operasi ketika proses pelucutan CO_2 dari 27 °C sehingga 100 °C telah dilaksana. Fluks pelucutan bernilai 2.57×10^{-2} mol m^{-2} s^{-1} pada halaju cecair 2.3 ms^{-1} dan suhu serap 80°C adalah lebih tinggi berbanding fluks membran dalaman dan komersial. Sehubugan itu, membran P17 dengan keliangan dipertingkat dan rintangan pembasahan yang tinggi menjadikannya berpotensi untuk menyingkir CO_2 secara berkesan menerusi penyentuh membran gas-cecair.

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LIST OF ABBREVIATIONS

AAG - 2-acrylamidogylycolic acid monohydrate
AAP - Acrylamido-2-methyl-1-propanesulfonic acid
AFM - Atomic force microscopy
AMP - 2-amino-2-methyl-1-propanol
CEPw - critical entry pressure of water
CO_2 - Carbon dioxide
$CaCO_3$ - Calcium carbonate
DCMD - Direct contact membrane distillation
DEA - Diethanolamine
DER - Dope Extrusion Rate
MDEA - Methyl diethanolamin
DMAC - Dimethylacetamide
DMF - Dimethylflomamide
DMSO - Dimethylsulfoxide
EDX - Energy Dispersive X-ray
FTIR/ATR - Fourier transform infrared/attenuated total reflectance
GPC - Gel permeation chromatography
HDI - Hexamethylene diisocynate
HFM - Hollow fiber membrane
IPCC - Intergovernmental panel on climate change
$LiCIO_4$ - Lithium percholarate
LiCl - Lithium chloride
$LiNO_3$ - Lithium Nitrate
MDI - Methylene bis-phenyl diisocyanate
MEA - Methylethanol amine
MMM - Mixed matrix membrane
MWCO - Molecular weight cut off
NIPS - Solvent induced phase separation
NMP - N-Methyl-1-pyrrolidone
nSMM - Novel (new) surafe modifying macromolecules

NVC	-	N-vinyl carprolactam
NVF	-	N-vinylformamide
NVP	-	N-vinyl-2pyrrodone
PCL	-	Polycaprolactone diol
PDMS	-	Polydimethylsiloxane
PE	-	Polyethylene
PEG	-	Polyethyl gycol
PEI	-	Polytherimide
PFA	-	Polytetraflouroethylene-co-perflourovinylether
PG	-	Potassium glycinate
PP	-	Polypropylene
PPO	-	Polyproylene diol
PSf	-	Polysulfone
PTFE	-	Polytetraflouroethylene
PTMO	-	Polytetramethylene diol
PVDF	-	Polyvinylidene fluoride
PVDF-HFP	-	Poly (vinylidene fluoride-co-hexaflouropropylene)
PVP	-	Polyvinyl pyrrolidone
PZ	-	Piperazine
RSM	-	Response surface methodology
SEM	-	Scanning electron microscopy
SMM	-	Surface modifying macromolecules
TEA	-	Triethanolamine
TIPs	-	Thermally induced phase separation
UF	-	Ultrafiltration
UV	-	Ultra-violet
VIPS	-	Vapor induced phase separation
VOC	-	Volatile organic compounds
XPS	-	X-ray photo-electron spectroscopy

LIST OF SYMBOLS

A - Mass transfer area

$C_{A,g}$ - Gas side concentration

$C_{A,l}$ - Liquid side concentration

d_i - Inside diameter of membrane

D_L - Diffusivity of CO_2 in a liquid

d_{lm} - Logarithmic mean diameter of membrane

d_o - Outside diameter of membrane

d_z - Mole balance across a segment

E - Enhancement factor

Gz - Greatz number

H - Henry’s constant

J_A - CO_2 absorption flux

J_i - Gas permeance

Jz - CO_2 absorption flux across a segment

K_0 - Intercept

k_l - Physical liquid-phase mass transfer coefficient

K_o - Overall mass transfer coefficient

Kz - Local mass transfer coefficient

L - Effective membrane length

Lp - Effective pore length

M - Gas molecular weight

n - Number of hollow fiber membranes

$\bar{P}$ - Mean pressure

P_0 - Slope

Q_L - Liquid flow rate

R - Universal gas constant

r_p - Membrane pore radius

$r_{p,max}$ - Maximum membrane pore radius

T - Temperature

T_g - Glass transition temperature

T_m - Melting temperature

w_1 - Weight of wet membrane

w_2 - Weight of dry membrane

ε, ε_m - Membrane surface porosity

γ - Surface tension

θ - Contact angle

ρ_w - Water density

ρ_p - Polymer Density

ΔP - Breakthrough pressure

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

There is a consensus among scientists that carbon dioxide (CO_2) is a major greenhouse gas contributing to global warming (Saidi, 2017). Continued emissions of CO_2 into the atmosphere by the excessive and unsustainable utilization of fossil fuels resulted in the formation of blanket-like layer of gases that trap heat, hence increasing average temperatures worldwide (Gómez-Coma *et al.*, 2017). Human-induced climate change is recognized as the most critical long-term global threat to future well-being and economy (Rahbari-Sisakht *et al.*, 2014). Therefore, Intergovernmental Panel on Climate Change (IPCC) urges the reduction of CO_2 emissions to the atmosphere by 50-80% until 2050 to avoid lasting damaging effects on global climate. Since the alternative sources of energy are not capable of replacing fossil fuels in the growing energy demand, low-cost efficient technologies to curb CO_2 emissions are essentially needed (Vogt *et al.*, 2011).

In the petroleum industry, natural gas is the fastest growing energy source in the world (Kang *et al.*, 2017). The primary element of natural gas is methane, however it also contains some serious contaminants that can cause detrimental environmental and economic effects if not efficiently removed (Rahim *et al.*, 2015). Table 1.1 contains detailed composition of a typical raw natural gas. Actually, natural gas composition varies from place to place. The composition in this table is the range obtained for Canada (Alberta), New Mexico (Rio Arriba County), Nigeria (Eleme), Southwest Kansas, Texas, Tunisia (Miskar), Vietnam (Bach Ho) and Western Colorado.

Table 1.1 Typical Natural Gas Compositions (Adewole *et al.*, 2013)

Component	**Composition Range (mol %)**
Helium	0.0 – 1.8
Nitrogen	0.21 – 26.10
Carbon dioxide	0.06 – 42.66
Hydrogen sulfide	0.0 – 3.3
Methane	29.98 – 90.12
Ethane	0.55 – 14.22
Propane	0.23 – 12.54
Butane	0.14 – 8.12
Pentanes and heavier	0.037 – 3.0

Apart from environmental reasons, the removal of CO_2 from natural gas has various economic and practical advantages for the industry. First, CO_2 as an acidic gas corrodes pipelines, facilitates hydrate formation and causes pressure drop during transmission (Ahmadpour *et al.*, 2014). Second, the presence of CO_2 also lowers the overall heating value of natural gas leading to customers to develop commercial specifications about the CO_2 concentration in natural gas. To meet all those practical, commercial and environmental demands necessitated the development for various technologies that reduce CO_2 in natural gas to manageable levels of concentration (Boributh *et al.*, 2011; Li and Chen, 2005).

Traditionally, a number of separation processes were employed for the removal of CO_2 from gas streams including physical or chemical gas absorption into a solvent, packed columns, spray columns and bubble columns (Figueroa *et al.*, 2008; Lee *et al.*, 2008; Meng *et al.*, 2007). For gas absorption, large scrubbing towers are implemented where the liquid and gas are dispersed into one another (Kosaraju *et al.*, 2004). Absorbent liquids are flown counter-currently to gases to remove CO_2. Before the absorbent liquid is sent for reuse, it is fed to a regeneration unit to strip out CO_2 (Shen *et al.*, 2013).

Albeit its technological maturity, chemical absorption through absorption tower is restricted by a few significant drawbacks such as low gas loading capacity and large equipment size. This process also requires high energy consumption and initial investment cost (Goh *et al.*, 2019). Furthermore, these traditional techniques are not easy to operate because of the frequent problems including foaming, flooding, channeling and entrainment (Li and Chen, 2005). Moreover, as those conventional methods usually involve substantially complicated equipment, higher energy consumption and capital cost, there is a growing need for alternative technologies with lower operational and capital costs (Mansourizadeh and Ismail, 2012).

The utilization of hollow fiber membrane contactor for capturing CO_2 has grown popularity as an attractive substitute to traditional methods (Zhang *et al.*, 2013). Basically, the membrane in contactor applications acts only as a physical barrier where the mass transfer of the gaseous and liquid phases is achieved by diffusion in non-dispersive mechanism. Hence, the solute gas and absorbent liquid are flown counter-currently in the lumen and shell sides of a hollow fiber, which enables independent control of gas and liquid flow rates (Atchariyawut *et al.*, 2008). In the meantime, the operational problems associated with conventional techniques can be easily avoided. Additionally, the modularity of the membrane modules makes membrane contactors highly flexible in operations. The performance of a membrane contactor can be easily predicted from the known interfacial surface area (Mosadegh-Sedghi *et al.*, 2014). The compact modular structure of membrane contactors together with sizable gas-liquid interfaces enhances the mass transfer efficiency on a small foot-print. It has been reported that the CO_2 absorption rate per unit volume of the membrane contactor was 2.7 times higher than that of the packed column, presumably caused by the increased interfacial area (Yeon *et al.*, 2005).

Overall, since gas-liquid membrane contactors combine both absorption (high selectivity) and membrane separation (modularity and compact structure), this technology presents significant advantages over the conventional absorption techniques.

1.2 Problem Statement

In contacting processes, membranes act as a barrier wall between the gas and liquid phases. The additional physical presence of the membranes pose auxiliary resistance to the mass transfer process (Belaissaoui and Favre, 2018). The phenomenon becomes more pronounced when the porous membranes are wetted by the liquid absorbents, resulting significant increase in the resistance to mass transfer mechanism (Li *et al.*, 2018). Generally, membrane wetting occurs when a stagnant liquid layer is introduced in the pores of the membrane either by penetration or capillary condensation (Mosadegh-Sedghi, *et al.*, 2014). Depending on the membrane material, the liquid absorbent nature and the pressure of the two phases, the membrane pores may be filled with gas or liquid which corresponds to the non-wetted mode and the wetted mode respectively (Rongwong *et al.*, 2009). The reduction of the overall mass transfer coefficient may reach up to 20% even if the membrane pores were 5% wetted (Wang *et al.*, 2005).

Wetting of the polymeric membranes deployed in gas-liquid contactor processes are determined primarily by the intrinsic properties of membrane materials, characteristics of spun membranes, operating pressure and temperature and the type of absorbent liquid used (Rezaei-DashtArzhandi *et al.*, 2016). Out of these parameters that influence membrane wetting, properties of materials selected to form the membranes cannot be molded by careful handling and experimental optimization. Consequently, the rational first step is to select membrane materials with the desired inherent attributes.

Typically, polymeric materials with hydrophobic property such as polypropylene (PP), polytetrafluoroethylene (PTFE) and polyvinylidene fluoride (PVDF) are widely used to fabricate membranes for gas-liquid contacting processes. Due to their high hydrophobicity, PP and PTFE have been utilized to fabricate commercially available membranes with symmetric structure. Nonetheless, the insolubility of these two materials in common solvents necessitates the use of inconvenient methods such as stretching or thermal techniques. In contrast, the easy processibility of PVDF polymer to fabricate porous membrane with asymmetric structure via simple phase inversion method have made it a common appealing substitute (Hosseini and Mansourizadeh, 2017). Notwithstanding its excellent chemical and thermal resistances, PVDF membranes tend to have low porosity owing to gradual phase inversion process.

To compensate for the shortcomings of polymeric membranes, several modification methods were proposed including blending of macromolecules (Rahbari-Sisakht, *et al.*, 2014), graft polymerization (Tanardi *et al.*, 2016), and ion beam irradiation (Rohani *et al.*, 2009). One recent method is the utilization of mixed matrix membranes (MMM) which involves the incorporation of inorganic fillers into the matrix of polymeric membranes. MMMs are lauded for their ability to bind the best properties of both polymeric materials and inorganic fillers, resulting in the production of highly permeable and mechanically stable membranes. MMMs can function as a structural and morphological director related to the improvement of interfacial polymer/inorganic properties.

Rezaei *et al.* (2014) investigated the performance of PVDF/montmorillonite (MMT) hollow fiber MMMs in absorption of CO_2 in membrane contactor. Their fabricated MMMs exhibited higher hydrophobicity, porosity and CO_2 absorption performance compared to neat PVDF.

In another work, Zhang and Wang (2014) used triple-orifice spinneret to incorporate fluorinated silica ($fSiO_2$) nano-particles in polyetherimide (PEI). They detected better wetting resistance, chemical compatibility and mechanical strength in modified PEI than the neat PEI membrane. Similarly, blending calcium carbonate ($CaCO_3$) nano-particles into the PVDF matrix has been reported to enhance membrane structure, narrow pore size distribution and increase membrane porosity (Hou *et al.*, 2014). Notably, unmodified $CaCO_3$ was also used to dramatically increase the hydrophilicity of PVDF (Zhi *et al.*, 2014).

In this study, hydrophobically modified $CaCO_3$ nano-particles will be employed as the inorganic filler for CO_2 removal in membrane contactors due to several important reasons. Firstly, as mentioned earlier, PVDF membranes tend to have low porosities which cause the unwanted mitigating factor of low CO_2 fluxes. The addition of $CaCO_3$ particles improves membrane porosity as demonstrated by Hou *et al.* (2012). Secondly, and most importantly, one desirable characteristic of membranes to be used in contactor applications is reduced pore diameter in order to have improved wetting resistance. Again, it has been shown that the impregnation of $CaCO_3$ nano-particles into PVDF matrix results membranes with smaller pore diameters (Hou *et al.*, 2012). In fact, the decreased pore sizes induced by $CaCO_3$ nano-particles is consistent with the need to tailor membrane pore sizes in order to be small enough to resist wetting and at the same time large enough to allow good mass transfer. Hence, the incorporation of modified $CaCO_3$ into PVDF membranes induces higher hydrophobicity and crucially narrower and numerous finger-like pores. As a result, its addition simultaneously improves pore wetting resistance and mass transfer, therefore, delicately balancing the parameters of the Laplace equation. Thirdly, to the best of our knowledge, no study has been done on the utilization of modified $CaCO_3$ nano-particles for the purpose of removing CO_2 in gas-liquid contacting processes, including the performance stabilization of long-term CO_2 absorption.

1.3 Objectives of the Study

Based on the above-mentioned problem statement, the objectives of the present study are as follows:

i. To fabricate PVDF mixed matrix membranes (MMMs) with well-tailored structures by incorporating modified hydrophobic calcium carbonate ($CaCO_3$) nano-particles in to the polymer matrix and examine the effects of nano-particle loadings on the membrane characteristics including pore size, porosity, hydrophobicity and surface roughness.
ii. To evaluate the performance of the fabricated membranes in CO_2 absorption and desorption.
iii. To evaluate the effect of polymer concentration variations on the membrane morphology and structural characteristics including pore size, porosity, hydrophobicity and surface roughness
iv. To investigate the effect of operating temperature on the membrane stripping performance

1.4 Scope of the Study

The principal aim of this study is to address the inadequacies of polymeric membrane materials by the dispersion of hydrophobic $CaCO_3$ nanoparticles in the polymer matrix. PVDF and lithium chloride were selected as the membrane material and non-solvent additive respectively. Polymer dopes containing various amounts of $CaCO_3$ nano-particle loadings (0, 10, 20, and 30 wt% of polymer) were prepared. The hollow fiber membranes were fabricated via non-solvent induced phase separation (NIPS). The effect of nano-particle on membrane properties including porosity, tortuosity and hydrophobicity was examined. The structure and morphology of the resultant membranes was studied using scanning electron microscope (SEM), atomic force microscopy (AFM), gas permeation test, critical water entry pressure (CEPw) and contact angle measurement. The performance of the fabricated membranes was tested in the CO_2 absorption/desorption and then the membrane with the best was chosen for further testing. Again, hollow fiber membranes with varying polymer concentrations (16, 17, 18 and 19 wt.% PVDF polymer concentration) with the selected $CaCO_3$ loading were fabricated. The effect of polymer concentration on the characteristics of the membranes and CO_2 absorption/desorption performance was investigated. Lastly, the effect of operating temperature (27 – 100 ^oC) on the performance of the selected membrane was investigated.

1.5 Significance of the Study

The utilization of mixed matrix membranes (MMMs) as a method of compensating for the inadequacies of polymeric membranes in contactor applications have gained attention recently. In this work, modified $CaCO_3$ nano-particles were selected as the inorganic fillers of PVDF MMMs for CO_2 removal in membrane contactors. This was a novel utilization of modified $CaCO_3$ nano-particles as they were previously employed in membrane distillation and not membrane in contactor applications. Since the modified $CaCO_3$ nano-particles are available commercially at low cost, this work provides the industry a cheap way to significantly raise the performance of polymeric membranes. For natural gas industry, this work offers a foundation for an inexpensive and efficient way of removing CO2 from natural gas to meet the customers’ demands. In addition, this study is beneficial for the community and public society at large as it involves the removal of CO_2 which is the culprit of global warming.

1.6 Limitations of the Study

There were few limitations in this study. First, during phase inversion method, the parameters that control the structure and morphology of the asymmetric hollow fiber polymeric membranes are too numerous. To name some, these parameters include polymer concentration, air gap, bore fluid type, bore fluid composition, dope extrusion rate and coagulation bath temperature. It was not conceivable to investigate the effects of all these parameters in this study. The effects of polymer concentration variations, the most important parameter according to the literature, and operating temperature were investigated. Second, there are two types of CO_2 absorption/desorption, namely using either physical or chemical absorbent solution. The focus in this study was the physical removal of CO_2 in membrane contactors using water as the neutral absorbent. In the future, the performance of the PVDF/$CaCO_3$ MMMs could be tested using chemical absorbent solutions.

1.7 Organization of the Thesis

This thesis consists of five chapters, which describes the fabrication of PVDF/$CaCO_3$ for CO_2 absorption through gas-liquid membrane contactors. Chapter 1 outlines a brief introduction of the membrane contactor for the capture of CO_2 and background of the research. It is followed by the problem statement, which identifies the research direction. Based on the problem statement defined, the objective, scope and significance of the study are explained in detail.

In Chapter 2, a general overview of the methods of CO_2 capture, and brief information about the advantages of membrane CO_2 capture in comparison with the other removal processes is provided. Afterwards, the challenges faced by CO_2 absorption membranes and the proposed prevention methods are also presented. Then, comprehensive studies about the use of mixed matrix membranes (MMMs) in membrane applications are given.

In Chapter 3, the research plan is outlined. The materials selected and the methodologies for the fabrication of porous plain membranes and PVDF/$CaCO_3$ MMMs are also discussed. Moreover, the related characterizations in membrane absorption/desorption processes are described in detail including membrane mass transfer resistance measurements.

In Chapter 4, fabrication of PVDF/$CaCO_3$ MMMs via wet phase inversion process by varying the nano-particle loading is presented. The effects of PVDF/$CaCO_3$ weight ratio on the phase inversion process, structure and performance of the membranes are discussed. The purpose of this study was to identify which nano-particle loading gives the best results in terms of structure and performance. After selecting the optimum loading, polymer concentration was varied and its effects on the structure and performance of the PVDF/$CaCO_3$ were studied. Polymer concentration was identified as the chief controlling parameter of non-solvent induced phase separation (NIPS). Additionally, the membrane with the CO_2 absorption flux was selected for further testing in different operating temperatures for CO_2 stripping via membrane contactors.

Finally, Chapter 5 concludes the thesis by discussing the achievements made on the objectives set for this study. It also highlights recommendations and future works and the possibility of building on this research.

1.8 Summary

In this chapter, the main objective and scope of the research were highlighted, namely the development of PVDF/$CaCO_3$ MMMs for the removal of CO_2 in gas-liquid contacting processes. Crucially, the reasons of selecting $CaCO_3$ nanoparticles as the inorganic filler are discussed in Section 1.2. It also presents the significance and limitations of the research. The next chapter has a detailed discussion of membrane contactors and the challenges encountered when employing them for the removal of CO_2, including why the employment of mixed matrix membranes to overcome the shortcomings of the membrane contactors for gas-liquid contactors is an attractive method.

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APPENDIX D

List of Publications

Indexed Journal

1. **Fosi-Kofal, M.**, Mustafa, A., Ismail, A. F., Rezaei-DashtArzhandi, M. and Matsuura, T (2016). PVDF/$CaCO_3$ composite hollow fiber membrane for CO_2 absorption in gas-liquid membrane contactor. *Journal of Natural Gas Science and Engineering*. 31: 428-436.

Non-indexed Journal

1. **Fosi-Kofal, M.**, Mustafa, A., Ismail, A. F (2016). Development of novel surface modified poly (vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) membrane contactor for CO_2 Absorption. *Journal of Scientific Research and Development*.

Conference presentation

1. **Fosi-Kofal, M.**, Mustafa, A., Ismail, A. F (2016). Experimental $_S$tudy of PVDF-$CaCO_3$ membrane contactor for CO_2 removal. Presented at IGCESH 2016, Johor Bahru, Malaysia, 15-17 August 2016.