

DEVELOPMENT OF POLYMER BLEND-BASED CARBON MEMBRANE FOR  
CARBON DIOXIDE SEPARATION FROM NATURAL GAS

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Dedicated to my beloved parents

*(Sazali bin Kandar, Soliah binti Adinan)*

Siblings

*(Norzalyana, Norsuhailizah, Mohammad Suzaima, and Mohammad Suzaidie)*

and friends for their continuous support and  
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## ABSTRACT

In membrane technology, polymeric membranes are the commonly used commercial membranes for carbon dioxide (CO<sub>2</sub>) separation. The limitations of commercial polymeric membranes have motivated researchers to study other alternatives, namely inorganic membranes, due to their higher thermal stability, good chemical resistance to solvents, higher mechanical strength and longer lifespan. In this study, novel carbon membranes supported onto porous alumina tubular with superior CO<sub>2</sub> separation performance were fabricated via carbonization of P84 co-polyimide (PI) blended with different types of additives such as polyvinylpyrrolidone, microcrystalline cellulose, and nanocrystalline cellulose (NCC). The preparation of this carbon membrane involved three main processes: (a) dope polymeric precursor membrane preparation, (b) coating step, and (c) heat treatment process. The influence of the dope formulation, carbonization conditions, and dip-coating parameters on the gas separation performance of the carbon membranes was evaluated. All membrane samples were characterized using scanning electron microscope, thermal gravimetric analysis, x-ray diffraction meter, raman analysis, mercury porosimetry, and Fourier transform infrared spectroscopy. Pure gas permeation tests of the resultant membranes were also conducted and evaluated by using CO<sub>2</sub>, and methane (CH<sub>4</sub>). The effects of additive loadings, carbonization conditions and dip-coating parameters have shown significant improvements toward the physiochemical and gas permeation properties. The incorporation of additives enhanced the gas separation performance in comparison to single polymer-based carbon membranes, where in this case NCC demonstrated the most promising additive with 36% improvement of CO<sub>2</sub> permeance. Manipulation of gas environments during carbonization process of argon, helium, and nitrogen have shown that argon environment gives 32% and 12% improvement of CO<sub>2</sub> permeance and CO<sub>2</sub>/CH<sub>4</sub> selectivity, respectively. These were due to higher order degree of carbon membrane and significant acceleration of the degradation reaction on carbon membranes. The influence of dip coating-carbonization cycles and dipping time during membrane fabrication resulted in a uniform membrane with pinhole-free particle layer and specific pore diameter. When the soaking time was increased from 30 to 120 min, CO<sub>2</sub> permeance and CO<sub>2</sub>/CH<sub>4</sub> selectivity increased up to 180% and 86%, respectively. The study also revealed that membrane PI-based carbon membrane with addition of 7 wt% of NCC featured excellent permeation properties with permeance of  $3.13 \pm 1.56$  GPU and  $213.56 \pm 2.17$  GPU for CH<sub>4</sub> and CO<sub>2</sub> gases, respectively. PI/NCC carbon membrane exhibited the highest CO<sub>2</sub>/CH<sub>4</sub> selectivity of  $68.23 \pm 3.27$  GPU under argon carbonization environment, 120 min thermal soaking time, two times coating-carbonization cycles, and 45 minutes coating duration.

## ABSTRAK

Dalam teknologi membran, membran polimer merupakan membran komersial yang biasa digunakan untuk pemisahan karbon dioksida ( $\text{CO}_2$ ). Keterbatasan membran polimer komersial telah mendorong penyelidik untuk mengkaji alternatif lain, iaitu membran bukan organik, disebabkan oleh kestabilan haba yang lebih tinggi, ketahanan kimia yang baik terhadap pelarut, kekuatan mekanikal yang lebih tinggi dan jangka hayat yang lebih lama. Dalam kajian ini, membran karbon baharu disokong oleh tiub alumina berliang dengan prestasi pemisahan  $\text{CO}_2$  unggul telah dihasilkan melalui pengkarbonan P84 ko-polimida (PI) diadunkan dengan jenis bahan tambah berbeza seperti polivinilpirolidona, selulosa mikrokristal, dan selulosa nanokristal (NCC). Penyediaan membran karbon ini melibatkan tiga proses utama: (a) penyediaan membran prapenanda polimer dop, (b) langkah salutan dan (c) proses rawatan haba. Pengaruh formulasi dop, keadaan pengkarbonan, dan parameter salut-celup terhadap prestasi pemisahan gas membran karbon telah dinilai. Semua sampel membran telah dicirikan dengan menggunakan mikroskop elektron imbasan, analisis termogravimetri, pembelauan sinar-X, analisis raman, porosimeter merkuri, dan infra-merah jelmaan Fourier. Ujian penelapan gas tulen membran yang dihasilkan juga telah dilakukan dengan menggunakan  $\text{CO}_2$ , dan metana ( $\text{CH}_4$ ). Kesan penambahan bahan tambah, keadaan pengkarbonan dan parameter salut-celup telah menunjukkan peningkatan yang ketara ke atas sifat-sifat fisiokimia dan penelapan gas. Penambahan bahan tambah meningkatkan prestasi pemisahan gas berbanding dengan membran karbon berasaskan polimer tunggal, di mana bahan tambah NCC didapati paling baik dengan peningkatan 36% untuk penelapan  $\text{CO}_2$ . Perubahan beberapa persekitaran gas semasa proses pengkarbonan dengan argon, helium, dan nitrogen menunjukkan bahawa persekitaran argon memberikan peningkatan 32% dan 12%, masing-masing terhadap penelapan  $\text{CO}_2$  dan sifat kememilihan  $\text{CO}_2/\text{CH}_4$ . Ini disebabkan oleh tahap membran karbon yang lebih tinggi dan pecutan ketara terhadap tindakbalas degradasi pada membran karbon. Pengaruh kitaran pengkarbonan celup-salutan dan masa celupan semasa fabrikasi membran menghasilkan membran yang sekata dengan tanpa serpihan lubang dan diameter pori tertentu. Penelapan  $\text{CO}_2$  dan sifat kememilihan  $\text{CO}_2/\text{CH}_4$  masing-masing meningkat sehingga 180% dan 86% apabila masa rendam haba dinaikkan daripada 30 hingga 120 minit. Kajian juga menunjukkan bahawa membran karbon berasas PI dengan 7% berat NCC menampilkan ciri-ciri penelapan gas yang sangat baik dengan nilai sebanyak  $3.13 \pm 1.56$  GPU dan  $213.56 \pm 2.17$  GPU untuk  $\text{CH}_4$  dan  $\text{CO}_2$ . Membran karbon PI/NCC mempamerkan sifat kememilihan  $\text{CO}_2/\text{CH}_4$  tertinggi, iaitu  $68.23 \pm 3.27$  GPU di bawah persekitaran pengkarbonan gas argon, 120 minit masa rendaman haba, dua kali kitaran pengkarbonan salutan, dan tempoh masa salutan 45 minit.

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**LIST OF ABBREVIATIONS**

CH <sub>4</sub>	-	Methane
C <sub>2</sub> H <sub>4</sub>	-	Ethene
C <sub>2</sub> H <sub>6</sub>	-	Ethane
CNT	-	Carbon nanotube
CMS	-	Carbon molecular sieve
CO <sub>2</sub>	-	Carbon dioxide
CA	-	Cellulose acetate
CMS	-	Carbon molecular sieve
CSD	-	Chemical solution deposition
DMAc	-	Dimethylacetamide
DSC	-	Differential scanning calorimetry
FESEM	-	Field emission scanning electron microscopy
FTIR	-	Fourier transform infrared spectroscopy
H <sub>2</sub>	-	Hydrogen
He	-	Helium
H <sub>2</sub> S	-	Hydrogen sulphide
ID	-	Inner diameter
OD	-	Outer diameter
MeOH	-	Methanol
MMM	-	Mixed matrix membrane
MMT	-	Montmorillonite
MOF	-	Metal organic framework
MWCNT	-	Multiwall carbon nanotube
N <sub>2</sub>	-	Nitrogen
NO <sub>2</sub>	-	Nitrogen dioxide
NMP	-	n-Methyl-2-pyrrolidone
O <sub>2</sub>	-	Oxygen

PAN	-	Polyacrylonitrile
PBI	-	Polybenzimidazole
PFA	-	Polyfurfuryl alcohol
PEG	-	Polyethylene glycol
PEI	-	Polyetherimide
PES	-	Polyethersulfone
PI	-	Polyimide
PLLA	-	Poly(L-lactide)
PPES	-	Poly (phthalazinone ether sulfone)
PPESK	-	Poly (phthalazinone ether sulfone ketone)
PPO	-	Poly(p-phenylene oxide)
PSf	-	Polysulfone
PSM	-	Particle size measurements
PVDC	-	Polyvinylidene chloride
PVP	-	Polyvinylpyrrolidone
RTIL	-	Room temperature ionic liquids
SEM	-	Scanning electron microscopy
SPAEK	-	Sulfonated aryl ether ketone
SO <sub>2</sub>	-	Sulphur dioxide
TEM	-	Transmission electron microscopy
TGA	-	Thermal behaviour analysis
THF	-	Tetrahydrofuran
TiO <sub>2</sub>	-	Titanium dioxide
WAXD	-	Wide angle x-ray diffraction
XRD	-	X-ray diffraction
ZIF-8	-	Zeolitic imidazolate framework



## LIST OF SYMBOLS

$A$	-	Effective membrane area (cm <sup>2</sup> )
$AR_{av}$	-	Average aspect ratio (dimensionless)
$D_{0.1}$	-	Free-path distance distribution (dimensionless)
$\frac{dV_i}{dt}$	-	Volumetric flowrate of gas permeated through the membrane (cm <sup>3</sup> /s)
$i$	-	Gas penetrant $i$ (dimensionless)
$j$	-	Gas penetrant $j$ (dimensionless)
$L_f$	-	Free-space length (nm)
$L_s$	-	Square length (nm)
MW	-	Molecular weight of repeat unit of polymer (g/mol)
$p$	-	Partial pressure (bar)
$P$		Permeance (GPU)
$T_g$	-	Glass transition temperature (°C)
$t_p$	-	Thickness of particle (nm)
Å	-	Angstrom
$\rho$	-	Density (g/cm <sup>3</sup> )
$\sigma_y$	-	Yield of strength (MPa)
$\lambda$	-	Wavelength of X-ray equal to 0.154056 nm
$\theta$	-	Angle at the maximum point of the first peak in the XRD pattern (°)
$\alpha$	-	Selectivity (dimensionless)
$\mu$	-	Mean spacing (nm)
$\Delta p$	-	Transmembrane pressure drop (bar)
$\Phi$	-	A plane angle

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Research Background

Natural gas is considered as an important source of fuel, as well as a primary feedstock for most petrochemical industries. It was reported the global consumption of natural gas is projected to increase up to 182 trillion cubic feet by the year 2030 from 95 trillion cubic feet recorded in 2003 (Yeo *et al.*, 2012). The separation of natural gas by thin barriers known as membranes has been verified to be economically and technically excellent technology by previous researchers (Adewole *et al.*, 2013a). In comparison with other sources of energy such as fossil fuel, natural gas is mostly considered as a clean source of energy. However, raw natural gas generally is not free of impurities. The large component constitutes of methane (CH<sub>4</sub>) and other hydrocarbons such as ethane (C<sub>2</sub>H<sub>6</sub>), propane (C<sub>3</sub>H<sub>8</sub>), butane (C<sub>4</sub>H<sub>10</sub>), CO<sub>2</sub> co-exists. Moreover, other impurities such as hydrogen sulphide (H<sub>2</sub>S), sulphur dioxide (SO<sub>2</sub>) and nitrogen (N<sub>2</sub>) are also present (Scholes *et al.*, 2012b). Removal of these types of impurities is crucial in minimizing issues related to fouling, corrosion, and pipeline rupture (Chua *et al.*, 2013). In addition, removal of CO<sub>2</sub> from natural gas is important to increase the calorific value and transportability of the natural gas in the pipeline (Adewole *et al.*, 2013a, Ahmad *et al.*, 2015).

The application of membrane in gas separation is a dynamic and rapidly growing field. They have drawn much attention by offering a number of advantages including ease of operation (no moving parts), low energy requirements and the overall economics of low-scale operation (Rufford *et al.*, 2012). Nowadays, gas separation is

one of the premier applications of membrane technology. The improvement of gas separation membranes in the past few years have accelerated in gas refining applications, especially natural gas. Membrane for gas separation technology can be marked as a competing industrial gas separation technique (Zhang *et al.*, 2013, Adewole *et al.*, 2013a). CO<sub>2</sub> capture from power plant flue gas and subsequent sequestration is expected to play a key role in mitigating global climate change. It is known that CO<sub>2</sub> is not only the main greenhouse gas but also an important potential carbon source, therefore, the capture and separation of CO<sub>2</sub> has attracted worldwide attention (Tena *et al.*, 2015). CO<sub>2</sub> has greater solubility coefficient than other light gases such as H<sub>2</sub>, N<sub>2</sub> and CH<sub>4</sub>, due to its high condensability. It has been pointed out that the polar groups such as carbonyl, ether, acetate groups etc have affinity for CO<sub>2</sub>. Thus, polymer containing these groups may achieve high CO<sub>2</sub> selectivity (Ho *et al.*, 2008).

Carbon membrane is one of the potential methods to remove CO<sub>2</sub> from natural gas. The interest on this membrane for gas separation has increased because of their molecular sieving properties that provide better selectivity and their higher thermal and chemical stability than the polymeric membranes. Due to the fragile characteristic of the thin carbon selective layer, the carbon membranes must be manufactured along with porous support, usually ceramic or metallic, for commercial purposes. Thus, a membrane with an improved mechanical resistance and thermal stability, supporting high temperatures and pressures, ideal characteristics has been fabricated for gas separation in several applications. Supported carbon membrane has attracted great attention due to its superior CO<sub>2</sub> separation and high mechanical strength. As mention by Hosseini and Chung (2009), carbon membrane is stable in several harsh environments such as under strong acid and base as well as at elevated pressure and temperature (Hosseini and Chung, 2009a). In the presence of high porosity and opening microspores that smaller than 0.5nm, high selective carbon membrane can be achieved. Carbon membranes are created from carbonization of polymeric precursor that consists of irregular microspores. During carbonization, thermally unstable polymer components would have evaporated and break the original polymer backbones. As a result, carbon membranes with highly porous, more open pores were created. The pores created were constructed in size and might allow molecular

separation by size and shape. These membranes offer great potential for operations with low maintenance, low energy demand, ease in scaling up and reliability.

## 1.2 Problem Statements

Polymer blend membranes of different polymer pairs have been reviewed and evaluated in terms of phase behaviour, permeability, and selectivity. The development of carbon membranes using polymer blend materials are among attractive strategies to improve their performances. The secondary polymer for blending are commonly possess low degradation temperature compared to primary polymer. Thus, during the carbonization process, the early degradation of the secondary polymer would create pore networks. This allows membrane to be more permeable compared to membrane without the additives, and potentially increase the gas pair selectivity. A variety of polymer blends has been explored in these recent years (Tiptipakorn et al., 2007, Hosseini et al., 2014b, Pirouzfard et al., 2014, Yong et al., 2016). In this current study, we incorporated this method in the application of carbon membranes by addition of polyvinylpyrrolidone (PVP), microcrystalline cellulose (MCC), and nanocrystalline cellulose (NCC) as an additives. All these three additives believed to exhibit enhanced performance in comparison to single polymer-based carbon membranes. However, there are still lack of comparative studies on those potential additives. Thus, choosing an appropriate polymer pairs for carbon membrane fabrication with an optimum composition is essential to overcome limitations in polymeric membrane applications (Itta et al., 2011). In the case of carbon membrane, the challenge factor is due to the issue of poor mechanical stability (He and Hägg, 2011). Hence, a membrane with a better separation properties and improved thermal stability and mechanical strength is needed to fully utilize the potential of this membrane technology in gas separation. Thus, membrane coated was prepared in this study. The purpose of choosing the membrane coated configuration is to overcome the poor mechanical strength that exhibited by unsupported carbon membrane.

On the other hand, it is reported that the gas permeation performance of the prepared carbon membrane are depending on the carbonization conditions during the

carbon membrane preparation steps (Salinas *et al.*, 2016a, Cipriani *et al.*, 2016, Abu El Hawa *et al.*, 2015, Llosa Tanco *et al.*, 2015a). There are two crucial process parameters which are gas environment and thermal soak time, that need to be considered to fine tune the final properties of the carbon membrane. Therefore, in this study, attempts are made to investigate the effect of gas environments and thermal soak times during the carbonization process on the physiochemical properties and gas permeation performance of the prepared coated membrane. By manipulating gas environments conditions, the production of higher order degree of carbon membrane can be achieved. In addition, a significant acceleration of the degradation reaction on carbon membranes can be achieved which lead to the formation of smaller pore size (Song *et al.*, 2010). Carbon membranes that carbonized from inert gasses shows superior gas separation performance with more open porous structure compared with those membranes that carbonized under vacuum environment; indicating that carbon membrane will possess smallest pores and low gas separation performance. Carbon membrane with a longer isothermal time (thermal soak time) results in more selective but less productive membranes (Favvas *et al.*, 2015).

Furthermore, in the preparation of coated membrane, there are several types of coating technique have been used by previous researches such as such as spin coating (Yong *et al.*, 2013), spray coating (Acharya and Foley, 1999), dip coating (Arabi Shamsabadi *et al.*, 2014, Qin *et al.*, 2015), slip-coating (Kim *et al.*, 2016, Lee *et al.*, 2016) and brush coating (Han *et al.*, 2016). Besides the types of coating technique, the effect of coating conditions is also play an important role in order to produce membrane with good separation performances. By comparing with all coating methods, dip-coating methods are most simple, inexpensive and most desirable choice for industrial applications. Dip-coating method will provide such good process parameters such as dip coating cycles and dipping time during membrane fabrication to obtain membrane with specific pore diameter. Therefore, these process conditions should be encouraged. Previous study by Kargari and co-workers, mentioned that by increasing the number of sequential dip-coatings, selectivity increased due to plugging or sealing the uncovered pores and defects in the membrane surface. The results indicate that two times coating-carbonization cycle would give superior performance on membrane selectivity (Kargari *et al.*, 2014). Therefore, the effect of dip-coating

process parameter such as coating-carbonization cycle and coating time towards the gas permeation performance will be investigated. Apart from author knowledge, by applying a tangential flow of suspension against the support during drying and dip-coating process, it will result in a dynamic particle deposition. The idea is to obtain a uniform and pinhole-free particle layer by the interaction of suspension tangential flow with the supported.

### **1.3 Objectives of Study**

Based on the problem statement discussed previously, the objectives of this research are outlined as follows:

- i. To investigate the effect of dope formulations (types of additives) on the morphological, thermal, and structural properties of the coated carbon membrane.
- ii. To investigate the effect of coated carbon membrane preparation conditions on the morphological, thermal, and structural properties of carbon membrane.
- iii. To evaluate the natural gas purification performance of the prepared tubular membrane.

### **1.4 Scopes of study**

In order to achieve the above-mentioned objectives, the following scopes of study are performed:

- i. Fabricating supported polymeric and carbon membrane using tubular alumina ceramic with 15wt% P84 co-polyimide as main precursor.

- ii. Fabricating flat-sheet polymeric and carbon membrane without support for characterization purpose as the presence of support would hinder most of characterizations used .
- iii. Fabricating coated polymeric and carbon membrane with different additives types (polyvinylpyrrolidone (PVP), microcrystalline cellulose (MCC) and synthesized nanocrystalline cellulose (NCC)) and composition (5 to 9 wt. %) via dip-coating technique.
- iv. Characterizing polymeric and carbon membranes by using Characterizing polymeric precursor and carbon membranes by using Thermogravimetric Analysis (TGA), X-ray Diffraction Analysis (XRD), Scanning Electron Microscopy (SEM), Mercury Porosimetry, Raman Spectroscopy, and Fourier Transform Infrared Spectroscopy (FTIR).
- v. Fabricating coated carbon membranes at different carbonization conditions (gas environment: N<sub>2</sub>, Ar, and He; thermal soak time: 30, 60, and 90 minutes).
- vi. Fabricating coated carbon membranes at different dip-coating parameters (coating-carbonization cycle: 1, 2, 3, and 4; coating time: 15, 30, and 45, and 60 minutes).
- vii. Conducting the pure gas permeation test using carbon dioxide (CO<sub>2</sub>), and methane (CH<sub>4</sub>) at room temperature with 8bars pressure.

## 1.5 Significance of the Study

It was acknowledged that polyimide embedded with additives have been studied in gas separation applications. However, a little attention has been given on the use of an additives in the preparation of supported carbon membranes. Therefore, attempts are made to investigate the potential of different types of additives such as PVP, MCC, and NCC in the preparation of supported carbon membranes for CO<sub>2</sub>/CH<sub>4</sub> separation. On the other hand, the used of tubular as supported on the preparation of carbon membrane would provide better alternative ways to enhance the mechanical



strength of the prepared carbon membrane. However, the characterization of all resultant membrane have been done by flat sheet configuration due to limitations of support membrane. The findings from this study would also provide a better understanding on the underlying principle on the fabrication of supported carbon membrane for CO<sub>2</sub> separation by considering physicochemical properties and gas permeation performance.

## **1.6 Organization of the Thesis**

The thesis consists of 5 chapters. Chapter 1 describes the brief information concerning the gas separation industries, current challenges in membrane technology and an introduction of carbon membrane. In addition, the problem statements, objectives and scopes of the study have also been provided in this chapter. Chapter 2 provides background information relevant to the gas separation technologies and their limitations as well as the past and current works on the development of membrane as a new approach for higher permeance and selectivity. Furthermore, the challenges and factors during fabrication of carbon membrane are also discussed. A comprehensive discuss on experimental methods and characterization techniques used throughout the study are explained in Chapter 3. In Chapter 4, the effect of dope formulation on the physicochemical and gas permeation properties of the tubular carbon membrane prepared from various additives were discussed. In this study, different thermally labile additives such as polyvinylpyrrolidone (PVP), microcrystalline cellulose (MCC) and nanocrystalline cellulose (NCC) were introduced in the P84 co-polyimide polymer solution. This chapter also includes the findings on the effects of carbonization conditions on the physicochemical and gas permeation properties of the tubular carbon membrane. In addition, the findings on the effects of coating conditions on the physicochemical and gas permeation properties of the tubular carbon membrane are briefly discussed. Finally, the general conclusions based on the findings and some recommendations for future works are provided in Chapter 5.

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