ZEOLITIC IMIDAZOLATE FRAMEWORKS BLENDED POLYSULFONE HOLLOW FIBER MEMBRANES FOR NATURAL GAS PURIFICATION

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For my beloved late mother and father,

my wife, children and family.

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ABSTRACT

Mixed matrix membranes (MMMs) have received world-wide attention for natural gas purification due to their superior performance in terms of permeability and selectivity. In this study, zeolitic imidazole framework (ZIF) based polysulfone (PSf) hollow fiber membranes were fabricated for natural gas purification. A new micron-sized leaf-like ZIF (ZIF-L) and hexagonal nano-sized ZIF-8 were synthesized in an aqueous basic solution at room temperature with the same molar ratio of reagents (Zn^{+2}/Hmim = 8). Furthermore, various moles of triethylamine (TEA)/total moles ratio of reactants ranging from 0–0.006 were used. Both ZIF powders were characterized by field emission scanning electron microscopy, X-ray diffraction, CO_2 temperature programmed desorption, Fourier transform infrared spectroscopy, thermogravimetric analysis, transmission electron microscopy, and surface area and pores textural properties using nitrogen adsorption-desorption analysis. ZIF-8 particles have shown improved thermal stability, textural properties, basic sites and CO_2 adsorption capacity compared to ZIF-L. The neat PSf membrane and mixed matrix hollow fiber membranes incorporated with the various loading of ZIF-8 ranging from 0–1.25% were fabricated at bore fluid rate of 1.5 and 1.8 ml/min. The prepared membranes were further investigated with respect to their structural morphology, thermal stability, functional groups, surface roughness and finally gas separation performance. The gas permeation results at room temperature showed that fabricated MMM at 1.8 ml/min of bore fluid and loaded with 0.5 wt% of ZIF-8 showed 28% higher CO_2/CH_4 selectivity at 6 bar (g) feed pressure compared to neat PSf membrane. High loading of ZIF-8 $\geq$0.75 wt% deteriorated the separation performances. However, CO_2/CH_4 selectivity decreased at elevated pressure (8 and 10 bar) due to CO_2-induced plasticization. The amine modification of ZIF-8 particles with 25 ml ammonium hydroxide solution at room temperature was found to significantly improve textural properties, basic sites strength and CO_2 desorption capacity. MMM prepared at 1.8 ml/min of bore fluid rate and loaded with 0.25 wt% of amine modified ZIF-8 showed 18% increase in CO_2/CH_4 selectivity compared to unmodified ZIF-8 based membrane. The amine modification was proven to be a membrane's anti-plasticization agent with superior gas separation performance at elevated pressure. In comparison to the neat PSf membrane, amine modified MMM prepared at the bore fluid rate of 1.8 ml/min has shown 50, 72 and 69% higher selectivity at 6, 8 and 10 bar (g) feed pressure respectively. Also, the selectivity of A-$M_{0.25}$ was 18% higher than unmodified ZIF-8 based MMM at 6 bar (g) feed pressure. The permeance of both gases decreased at an acceptable level with an increase of selectivity at elevated pressure. Hence, the promising results obtained in this study has demonstrated the potential of amine modified ZIF-8 based MMMs for natural gas purification.

ABSTRAK

Membran matriks campuran (MMMs) telah mendapat perhatian seluruh dunia untuk penulenan gas asli kerana prestasi unggulnya dari segi kebolehtelapan dan selektiviti. Dalam kajian ini, membran gentian geronggang polisulfona (PSf) berasaskan rangka imidazolat ziolitik (ZIF) telah dihasilkan untuk penulenan gas asli. ZIF berbentuk daun (ZIF-L) bersaiz-mikron dan ZIF heksagonal (ZIF-8) bersaiz-nano yang baharu telah disintesis dalam larutan berair pada suhu bilik dengan nisbah molar reagen yang sama (Zn^{+2}/Hmim = 8). Tambahan pula, pelbagai mol trietilamina (TEA)/jumlah mol ratio bahan tindak balas dari 0-0.006 telah digunakan. Kedua-dua serbuk ZIF ini dicirikan oleh analisis mikroskop elektron imbasan pelepasan medan, pembelauan sinar-X, penyahjerapan berprogram suhu CO_2, spektroskopi infra merah transformasi Fourier, analisis termogravimetrik, mikroskop elektron penghantaran dan sifat luas permukaan dan liang tekstur menggunakan analisis penjerapan penyahjerapan nitrogen. ZIF-8 menunjukkan peningkatan kestabilan terma, sifat-sifat struktur dan tekstur, tapak asas dan kapasiti penjerapan CO_2 berbanding dengan ZIF-L. Membran PSf yang asas dan membran serat berongga matriks campuran yang digabungkan dengan pelbagai muatan ZIF-8 dari 0-1.25% telah dihasilkan pada kadar bendalir penebuk 1.5 dan 1.8 ml/min. Membran yang disediakan telah disiasat dengan lebih lanjut mengenai morfologi struktur, kestabilan terma, kumpulan fungsi, kekasaran permukaan dan akhirnya prestasi pemisahan gas. Hasil ketelapan gas pada suhu bilik menunjukkan bahawa MMM yang dihasilkan pada 1.8 ml/min dengan bendalir penebuk dan dengan muatan 0.5 wt% daripada ZIF-8 menunjukkan selektiviti CO_2/CH_4 28% lebih tinggi pada tekanan suapan 6 bar (g) berbanding dengan membran PSf yang asas. Muatan tinggi ZIF-8 $\geq$0.75 wt% menjejaskan prestasi pemisahan. Walaubagaimanapun, CO_2/CH_4 telah menunjukkan penurunan selektiviti pada tekanan tinggi (8 dan 10 bar) akibat daripada pemplastikan teraruh CO_2. Pengubahsuaian amina yang lebih lanjut pada zarah ZIF-8 dengan 25 ml larutan amonium hidroksida pada suhu bilik didapati memperbaiki sifat-sifat struktur dan tekstur, kekuatan tapak asas dan kapasiti penjerapan CO_2 dengan ketara. MMM yang disediakan pada kadar bendalir penebuk 1.8 ml/min dan dimuatkan dengan ZIF-8 terubahsuai dengan 0.25% amina menunjukkan peningkatan 18% dalam selektiviti CO_2/CH_4 berbanding membran berasaskan ZIF-8 tanpa ubahsuai. Pengubahsuaian amina telah bertindak sebagai agen anti-pemplastikan membran dengan prestasi pemisahan gas unggul pada tekanan tinggi. Sebagai perbandingan dengan PSf yang asal, MMM diubahsuai amina yang disediakan pada kadar bendalir penebuk 1.8 ml/min telah menunjukkan selektiviti 50, 72 dan 69% lebih tinggi pada tekanan suapan masing-masing 6, 8 dan 10 bar (g). Juga, selektiviti A-$M_{0.25}$ adalah 18% lebih tinggi daripada membran berasaskan ZIF-8 tanpa ubahsuai pada tekanan suapan 6 bar (g). Ketelapan bagi kedua-dua gas menurun pada tahap yang boleh diterima dengan peningkatan selektiviti pada tekanan tinggi. Oleh itu, keputusan utama yang **diperoleh** dalam kajian ini telah menunjukkan potensi penulenan gas asli bagi MMM yang berasaskan ZIF-8 diubahsuai amina.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
AMDEA	-	Activated methyl diethanolamine
AS	-	Amine scrubbing
ATBC	-	Acetyl tributyl citrate
ATR-IR	-	Attenuated total reflectance infrared
BET	-	Brunauer-Emmett-Teller
CA	-	Cellulose acetate
CCM	-	Carbon cryogel microspheres
CM	-	Carbon membrane
CMS	-	Carbon molecular sieve
CNT	-	Carbon nanotubes
CS	-	Cryogenic separation
CSP	-	Chemical scrubbing process
CXM	-	Carbon xerogel microspheres
DEA	-	Diethanol amine
DEF	-	Diethylformamide
DER	-	Dope extrusion rate
DMAc	-	Dimethylacetamide
DMF	-	Dimethylformamide
DMSO	-	Dimethyl sulfoxide
DSC	-	Differential scanning calorimeter
EDX	-	Energy dispersive X-ray spectrometer
EIPS	-	Evaporation-induced phase separation
ESA	-	Electrical swing adsorption
FAU	-	Faujasite
FESEM	-	Field emission scanning electron microscopy

FTIR	-	Fourier transmission infrared
GHG	-	Greenhouse gas
GS	-	Gas separation
HD	-	Hemodialysis
HF	-	Hollow fiber
Hmim	-	2-methylimidazole
HPWS	-	High pressure water scrubbing
ISS	-	Inorganic solvent scrubbing
JS	-	Jet strength
LBM	-	Liquefied biomethane
LNG	-	Liquid natural gas
MDEA	-	Methyl diethanol amine
MEA	-	Monoethanol amine
MF	-	Microfiltration
MMM	-	Mixed matrix membrane
MOF	-	Metal-organic framework
MS	-	Membrane separation
NIPS	-	Nonsolvent induced phase separation
NMP	-	N-methyl pyrrolidone
NYT	-	Neapolitan yellow tuff
OPS	-	Organic physical scrubbing
PC	-	Polycarbonate
PDMS	-	Polydimethyl siloxane
PEG	-	Polyethylene glycol
PEI	-	Polyetherimide
PI	-	Polyimide
PSA	-	Pressure swing adsorption
PSf	-	Polysulfone
PVDF	-	Polyvinylidene difluoride
PZ	-	Piperazine
RO	-	Reverse osmosis
SAPO-34	-	Silicoaluminophosphate-34
SBUs	-	Secondary building units

SOD	-	Sodalite
STP	-	Standard temperature and pressure
TEA	-	Triethylamine
TEM	-	Transmission electron microscopy
TGA	-	Thermogravimetric analysis
THF	-	Tetrahydrofuran
TIPS	-	Thermally induced phase separation
TPD	-	Temperature programmed desorption
TSA	-	Temperature swing adsorption
UF	-	Ultrafiltration
VIPS	-	Vapor induced phase separation
XRD	-	X-ray diffraction
ZIF-L	-	Leak-like zeolitic imidazolate framework
ZIFs	-	Zeolitic imidazolate frameworks
ZSM-5	-	Zeolite socony mobil-5

LIST OF SYMBOLS

(P_i/l)	-	Pressure-normalized flux or permeance
P_i	-	Permeability of gas *i*
P_j	-	Permeability of gas $_j$
Q_i	-	Volumetric gas flow rate of gas *i*
€	-	Euro
Δ	-	Change
2D	-	Two dimensional
3D	-	Three dimensional
A	-	Effective membrane area
Å	-	Angstroms
B	-	Full-width at half maximum of the peak in radian
D	-	Crystal size, nm
Da	-	Molecular weight
$P_{plasticization}$	-	Plasticization pressure
R_a	-	Average roughness, nm
R_{ms}	-	Root mean squared roughness, nm
T_c	-	Crystallization temperature
T_g	-	Glass-transition temperature
T_p	-	Derivative peak of temperature
V_m	-	Molar volume
α	-	Gas pair selectivity
ΔP	-	Pressure difference
λ	-	X-ray wavelength
ϕ	-	Elongational draw ratio
θ	-	Diffraction angle

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Natural gas is formed due to decomposition of plants and animal materials by heat and high pressure under the surface of the earth over millions of centuries. It is usually composed of hydrocarbon gas mixtures mainly consisting of methane (CH_4) and carbon dioxide (CO_2) (Baker and Lokhandwala, 2008). CH_4 is a world's energy source that provides energy for heating, electricity generation, cooking and also as a fuel for vehicles but it requires purification from impurities. Furthermore, environmental problems have become a big issue due to the greenhouse gas (GHG) emission as the result of fossil fuel combustion (Hosseini and Wahid, 2013). Among impurities, the removal of CO_2 is more crucial and important due to its corrosive nature. In addition, the uncontrolled CO_2 emission to the atmosphere has become great concern worldwide that would lead to climate change, flooding, acid rain, hazards for human health and many undesirable effects (Permpool and Gheewala, 2017, Sun *et al*., 2015 and Nojedehi *et al*., 2016). There are several technologies that were commercially available on an industrial scale for purification of natural gas to ensure the elimination of CO_2. Adsorption, absorption, membrane separation and cryogenic technique were well-established processes with their own advantages and disadvantages. In general, membrane-based gas separation technique offers various advantages over the conventional processes such as: 1) Simple operation with easy maintenance (Kárászová *et al*., 2015). 2) An economical process with low capital investment cost (Bekkering *et al*., 2010). 3) Low energy demands with minimal space and supervision requirement (Samarasinghe *et al*.,2018), and 4) Environmentally friendly process.

Metal-organic framework (MOF) has shown to have an excellent affinity towards many polymer matrices even without surface modification (Wen *et al*., 2009). MOF is a crystalline compound with metal ions and organic ligands as a repetitive unit arranged systematically as a framework (Samarasinghe *et al*., 2018). The organic ligands within its structure offers exceptional interaction with polymer matrices, hence minimize the interfacial defects. Zeolitic imidazolate frameworks (ZIFs), a subclass of MOFs are emerging as a new family of molecular sieves with low cost, and highly diversified and tunable structural properties (Lee *et al*., 2015, Wang *et al*., 2008). Therefore, leaf-like zeolitic imidazolate framework (ZIF-L) and zeolitic imidazolate framework 8 (ZIF-8) particles are synthesized and characterized. The selection of polymer and dispersed phase are very important factor to produce defect free and high-performance mixed matrix membrane (MMM). Hence, current research is concentrated on the fabrication of MMM having polysulfone (PSf) as a continuous phase and ZIF based inorganic filler as a dispersed phase. The basic objective is to improve gas separation performance of the existing pristine PSf membrane. PSf is the best option as polymer phase due to its advantages such as: (1) High mechanical and thermal strength (Nordin *et al*., 2014). (2) One of the most studied polymer membrane materials due to its low cost compared to other polyimide materials (Julian and Wenten, 2012). (3) Excellent equilibrium between permeability and selectivity towards CO_2 with high plasticization pressure ($P_{plasticization}$ = 34 bar) (Bos *et al*., 1999). (4) Reasonably high separation factor for gas separations (Intrinsic CO_2/CH_4 selectivity = 28.1 (Chern and Koros, 1985), and (5) good stability against environmental oxidation due to its high glass-transition temperature (T_g) approximately 185°C (Kesting *et al*., 1990).

1.2 Problem Statement

Membrane technology has proven to have numerous advantages over commercial adsorption and absorption processes. But, recent developments in this field are still at the experimental stage and long-term stability at elevated pressure has rarely been investigated in the literature. Selection of suitable membrane materials is a major requirement to fabricate high-performance defect-free membranes with low cost, high thermal stability and plasticization resistance at elevated pressure. Most of the inorganic fillers are not compatible with the polymer phase and cause the occurrence of non-selective interfacial voids that leads to reducing the gas separation performance due to unselective pathways at the filler interfaces. Hence, it is necessary to investigate the common problems such as filler size and loading, compatibility with polymers, modification and gas seaparation performance in the MMM. However, the ZIF-L is still not commercially produced and available ZIF-8 possesses large particle sizes (particle size of ~500nm) and this would rise as challenges to incorporate the particles within the thin-selective layer of membrane. In addition, ZIF-8 is considerably expensive and would certainly increase the cost of prepared membrane. Nordin et al. (2015) has reported the cost of ZIF-8 particles around RM 25,551.87 for 500g, relatively expensive compared to synthesis materials provided by Sigma Aldrich in this research such as 2-methylimidazole (RM 852/kg), zinc nitrate hexahydrate (RM 802/kg) and base-type additive triethylamine (RM 361/500ml). Hence, research to produce smaller ZIFs particle with economical method would be of special interest to this study. The addition of base-type additives such as triethylamine (TEA) during synthesis process is beneficial in various ways such as minimizing the usage of organic ligands, shorter time of synthesis due to rapid deprotonation of organic ligands which further reduce the particles size. Though ZIF-L and ZIF-8 are compatible with different polymer materials but their intrinsic separation factor is not satisfactory. The intrinsic CO_2/CH_4 selectivity of ZIF-L and ZIF-8 is 7.2 (Chen *et al*., 2013) and 5 (Chen *et al*., 2014; He *et al*., 2014; Yao *et al*., 2013) respectively which is significantly lower than zeolite (CO_2/CH_4 = 80 (Yeo *et al*., 2014)) and carbon membrane (CO_2/CH_4 = 80 (Salleh and Ismail, 2011)). It is essential to modify the filler surface to enhance CO_2 adsorption capacity. Furthermore, ZIFs based membranes have low plasticization resistance at elevated operating pressure. Hence, amine modification of ZIFs particles is used to improve its functional properties and compatibility with polymer. Subsequently, reducing the segmental mobility of polymer matrix and improve the plasticization resistance. So, these factors can be regarded as the main hindrances to the potential application of ZIFs based MMM for gas separation. Unless these limitations are addressed, the advantages offered by ZIF based MMM are likely to be neglected.

Therefore, this study is aimed to fabricate MMMs for CO_2 separation using various loading of synthesized and amine modified ZIFs nanoparticles. Furthermore, the effect of the different loading of fillers and feed pressures on the gas separation performance of prepared MMM is evaluated. Moreover, to date, the incorporation of ZIFs particles has primarily been subjected to the preparation of flat sheet membranes, whereas studies on ZIFs based hollow fiber membranes are rarely investigated.

1.3 Objective of the Study

The major goal of this research was to produce asymmetric mixed matrix hollow fiber membranes with ZIF based materials as the filler via dry-wet phase inversion process with high gas separation performance and improved plasticization resistance. Therefore, based on the above mentioned challenges and issues, the specific objectives of this research are as follows

i. To synthesize and characterize the ZIF-L and ZIF-8 particles with various moles of TEA/total moles ratio of the reactants with the aim to evaluate their potential for the use as filler in the fabrication of MMMs.
ii. To investigate the effect of different loading of selected ZIF based filler on the resultant mixed matrix hollow fiber membrane gas separation performance at various feed pressures.
iii. To evaluate the effect of amine modification of selected ZIF based filler on CO_2/CH_4 selectivity and plasticization resistance at various feed

pressures of gas permeation operations of the mixed matrix hollow fiber membranes.

1.4 Scope of the Study

The following activities of research were selected as the scope of this research to achieve the above mentioned objectives:

i. Investigating the effect of moles of TEA/total moles ratio ranging from 0−0.006 in the formation of ZIF-L and ZIF-8 at room temperature.
ii. Characterizing the ZIF-L and ZIF-8 particles by X-ray diffraction (XRD) analysis, transmission electron microscopy (TEM), field emission scanning electron microscopy (FESEM), Fourier transmission infrared (FTIR), Attenuated total reflectance infrared (ATR-IR), thermogravimetric analysis (TGA), surface area using the Brunauer-Emmett-Teller (BET) equation and CO_2 temperature programmed desorption (CO_2 -TPD).
iii. Formulating polymer dope solutions comprised of polysulfone (Udel® P−1700, 30%), N, N-dimethylacetamide (DMAc, 35%), tetrahydrofuran (THF, 35%) with the various loading of selected filler ranging from 0 wt% to 1.25 wt%.
iv. Fabricating mixed matrix hollow fiber membranes using bore fluid rate of 1.5 and 1.8 ml /min.
v. The potted fibers were externally coated using 3 wt% of PDMS dissolved in n-hexane.
vi. Amine modification of selected filler by using 25 and 50 ml ammonium hydroxide solution.
vii. Characterizing the membranes using atomic force microscopy (AFM), energy dispersive X-ray spectrometer (EDX), and differential scanning calorimeter (DSC), FESEM and TGA.

viii. Evaluating the gas separation performance and plasticization resistance of the fabricated mixed matrix hollow fiber membranes using pure gases (CO_2 and CH_4) at three different pressure (6, 8 and 10 bar).

1.5 Significance of the Study

The recent formation of ZIF-L and ZIF-8 particles through aqueous condition has emphasized the significance of this process. Particularly, this method gives rapid reaction between metal source and an organic ligand, promote yield due to the presence of base type additive TEA. Till date, aqueous condition synthesis needed high metal to solvent ratio but TEA induce the deprotonation of the organic ligand, subsequently reduce the excessive solvent usage and synthesis time. This alternative method offered in this research is very productive, environmentally friendly and economical with improved morphology and gas separation performance compared to other available methods.

Generally, one common problem encountered during fabrication of MMMs is filler-polymer incompatibilities that affect the gas separation performance. The unique advantage of incorporating ZIFs over many nonporous materials is its organic components that enhanced the filler-polymer compatibilities and also improved the separation performance and plasticization resistance of resulted membranes. Furthermore, amine modification of ZIFs particles is carried out to improve its functional properties before being dispersed into PSf. Therefore, MMMs with high gas separation performance and plasticization resistance at elevated pressure is expected to produce for natural gas purification. The gas separation performances of the amine modified MMMs explores the new perspective of membrane for natural gas purification. The CO_2 plasticization phenomena was yet to be investigated for MMMs. The incorporation of amine modified ZIFs nanoparticles into polymer matrix has offered superior gas separation performance with improved plasticization resistance compared to the virgin ZIFs based and neat membrane. This research contribution will offer guidelines to future researchers to select suitable organic and inorganic membrane materials for high performance mixed matrix membranes with economical, safer and environmentally friendly unit operation.

1.6 Thesis Organization

This thesis consisted of eight chapters which describe original and novel research on mixed matrix membranes for natural gas purification. Chapter 1 briefly explores the ideas of membrane separation processes. The research background of membrane technology and the issues that lead to the current study were discussed. The four research objectives were identified, followed by the scopes of study used to attain these objectives. Chapter 2 describes the scientific literature review of all available separation processes with focused on membrane technology for gas separation. All basic principles of gas separation through hollow fiber membranes, the materials selection, morphology, spinning parameters and various fabrication techniques were critically reviewed and explained. The concepts and development of novel MMM were comprehensively explained. Also, advantages and limitations of ZIFs nanoparticles for membrane application were described in detail. Finally, different pre and post treatment methods for synthesized nanoparticles and MMM were discussed in detail. Chapter 3 provides the methodology for the fabrication of PSf and ZIFs mixed matrix membranes, procedures for material synthesis, modification, characterization techniques and finally gas separation performance. Also, complete research operational framework was provided in this chapter. Chapter 4 explains the synthesis of ZIF-L at aqueous room temperature with various concentrations of TEA in the synthesis mixture. The effect of various concentration of TEA on the yield, particle size, crystal growth, microposity, CO_2 desorption performance and thermal stability were investigated and discussed in detail. Chapter 5 elaborates on the determination of the critical loading of TEA that was used for intermediate structure between ZIF-L and ZIF-8 during the synthesis process. Chapter 6 aims to fabricate and characterize the defect-free high performance hollow fiber membranes using various loading of ZIF-8 nanoparticles and bore fluid rate. The effect of the different loading of ZIF-8, bore fluid rate and feed pressure on the gas separation performance was evaluated. Chapter 7 has investigated the effect of amine modification on the structural, thermal, pore textural and desorption properties of ZIF-8 nanoparticles. Defect-free high performance hollow fiber membranes using various loading of amine modified ZIF-8 nanoparticles and bore fluid rate has been fabricated and characterized. Also, it further explains the plasticization effects of CO_2 using amine modified ZIF-8 nanoparticles as dispersed phase into PSf polymer matrices at high feed pressures. The plasticization reduction with high gas separation performance is the major objective of this chapter. The gas separation performances of the amine modified ZIF-8 based membranes are evaluated and compared with ZIF-8-based membranes reported in the previous chapter (Chapter 6). Finally, Chapter 8 presents the general conclusions from present work and providing a list of some recommendations for future researches.

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