EFFECT OF CASTING PARAMETERS ON CELLULOSE ACETATE MEMBRANE MORPHOLOGY AND OXYGEN/NITROGEN SEPARATION

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DEDICATION

To my beloved husband, Mohd Hafis Maswir,

dearly daughter and son, Khalisah and Ihsan,

my late father (Jami'an Ahmad) and mother (Sayeah Sinan),

my late parent in laws (Maswir and Raemah),

my brothers and sisters.

Thank you for your eternal love and never ending support

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ABSTRACT

Membrane for oxygen purification have been reported to be competitive for a small scale oxygen production plant (10 to 25 tonnes/day) at 25 to 40 % oxygen (O2) purity. In order to overcome the trade-off between permeability and selectivity of the membrane, industries are always looking for ways to find a cost effective membrane system. Reducing and minimising the use of chemicals in membrane production will lead in achieving this intention. Therefore in this study, binary dope solution which involved only two chemicals (cellulose acetate (CA) and N-methyl-2-pyrrolidone (NMP)) were used. The aim of this study was to evaluate the effect of casting parameter towards membrane morphology and gas separation performance of CA membrane. For the methodology, CA polymer was dissolved in NMP solvent and fabricated using dry/wet phase inversion technique with three different parameter, first, at the different coagulation bath temperature, second at different solvent evaporation time, and at different coagulation bath composition. The membrane produced was coated with low-viscosity poly(dimethylsiloxane) to cover defects. The prepared membrane morphology was analysed using scanning electron microscope and gas separation performance was tested using soap bubble meter with pure oxygen and nitrogen gasses. Coagulation bath temperature, solvent evaporation time and coagulation bath composition involved in the phase inversion process were significantly influenced the structures and transport properties of the resultant membranes. It was found that, the highest selectivity obtained in this study was 14.95 at optimum conditions of 25 °C, 10 % NMP coagulation bath with 5 seconds solvent evaporation time and 10 % NMP in coagulation bath. This optimised membrane had successfully produced 29.38 % of oxygen purity when tested with compressed air. Hence, it can be said that, this membrane for oxygen purification will be suitable for industrial.

ABSTRAK

Membran untuk penulenan oksigen telah dilaporkan berdaya saing untuk loji penghasilan oksigen berskala kecil (10 hingga 25 tan/hari) pada ketulenan oksigen sebanyak 25% hingga 40%. Selain daripada untuk mendapat keseimbangan antara kebolehtelapan dan kememilihan membran, kebanyakan industri mencari jalan untuk menghasilkan sistem membran yang menjimatkan. Salah satu cara untuk mencapai matlamat ini adalah dengan mengurangkan penggunaan bahan kimia semasa proses penyediaan membran. Oleh itu, dalam kajian ini sistem larutan dop perduaan yang menggunakan dua bahan kimia (selulosa asetat (CA) dan N-metil-2-pirolidon (NMP)) telah digunakan. Tujuan utama kajian ini adalah untuk mengkaji kesan parameter penghasilan membran terhadap morfologi dan prestasi pemisahan gas membran CA. Metodologi kajian adalah dengan polimer CA dilarutkan di dalam NMP dan membran dihasilkan menggunakan teknik proses songsangan fasa kering/basah dengan tiga parameter yang berbeza, pertama, pada suhu medium pengentalan yang berbeza, kedua pada masa penyejatan pelarut dan komposisi medium pengentalan yang berbeza. Membran yang terhasil telah disalut dengan poli(dimetillsiloksana) berkepekatan rendah untuk menutup kecacatan. Morfologi membran yang terhasil telah dianalisa dengan menggunakan mikroskop elektron imbasan, dan prestasi pemisahan gas telah diuji menggunakan meter buih sabun dengan gas oksigen dan nitrogen tulen. Suhu medium pengentalan, masa penyejatan pelarut dan komposisi medium pengentalan yang terlibat dalam proses songsangan fasa telah memberi kesan yang ketara terhadap struktur dan prestasi membran yang terhasil. Hasil kajian, kememilihan tertinggi yang diperoleh adalah 14.95 pada keadaan optimum suhu 25 °C, rendaman pengentalan 10 % NMP dengan masa penyejatan pelarut adalah 5 saat dan 10 % NMP dalam rendaman pengetalan. Membran optimum ini berjaya menghasilkan ketulenan oksigen setinggi 29.38 % apabila diuji dengan udara termampat. Oleh yang demikian, membran untuk penulenan oksigen ini sesuai digunakan pada industri.

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LIST OF ABBREVIATIONS

OEC	-	Oxygen-Enhanced Combustion
O_2	-	Oxygen
PSA	-	Pressure Swing Adsorption
PIM	-	Polymers Of Intrinsic Microporosity
N_2	-	Nitrogen
CA	-	Cellulose Acetate
CO_2	-	Carbon Dioxide
SEM	-	Scanning Electron Microscopic
OEC	-	Oxygen-Enhanced Combustion
NOx	-	Nitrogen Oxide
H_2O PI	-	Polyimide
PSF	-	Polysulfone
PPO	-	Poly(2,6-dimethyl-1,4-phenylene oxide)
PVC	-	Polyvinyl Chloride
PEEK	-	Poly(ether ether ketone)
MMM	-	Mixed Matrix Membrane
CMS	-	Carbon Molecular Sieve
MOF	-	Metal Organic Framework
RO	-	Reverse Osmosis
PU	-	Polyurethane
UF	-	Ultrafiltration
PEG	-	Poly(ethylene glycol)
NMP	-	N-Methyl-2-Pyrrolidone
PES	-	Polyethersulfone
PEI	-	Polyetherimide
H_2O	-	Water
EtOH	-	Ethanol
PDMS	-	Polydimethylsiloxane
PBS	-	Poly(butylene succinate)

PVP	-	Polyvinylpyrrolidone
CH_4	-	Methane
DMAc	-	Dimethyl acetamide
GC	-	Gas Chromatography
ET	-	Evaporation Time
CBC	-	Coagulation Bath Composition
T_g	-	Glass Transition Temperature
CH_3OH	-	Methanol
C_6H_{14}	-	n-hexane
PMP	-	poly(4-methyl-1-pentene)
NOx	-	Nitrogen oxide
PI	-	polyimide
PU	-	polyurethane
GC	-	Gas Chromatorgaphy
STP	-	Standard Temperature Pressure

LIST OF SYMBOLS

%	-	Percentage
ºC	-	Celsius
ºF	-	Fahrenheit
K	-	Kelvin
Ω	-	Omega
atm	-	Standard atmosphere
s	-	second
Å	-	Angstrom

CHAPTER 1

INTRODUCTION

1.1 Introduction

The advance in membrane fabrication technology for O_2/N_2 separation began with the development of asymmetric membrane in the 1970s. In 1982, Generon produced the first air separation system based on poly(4-methyl-1-pentene) (PMP) membranes with an O_2/N_2 selectivity about 4. Then, the milestones in the industrial application of membrane O_2/N_2 separation systems are follows (Bernardo and Clarizia, 2013): PSf hollow fiber membranes for O_2/N_2, composite hollow fiber membranes for O_2/N_2 and polyphenyl oxide membrane for air separation. Recently, Air Products- supported in part by the U.S Department of Energy – has developed ion transport membrane for air separation. Concurrently with the development in the large scale, intensive research in the laboratory scale also continues to grow since there are still many challenges to be addressed to obtain highly efficient membrane-based O_2/N_2 separation.

In large scale production of O_2/N_2, the use of membranes can be classified as the following: (1) membranes with selectivities of 3–4 combined with high permeabilities (~600 barrers) for O_2 production with low purity, i.e. <50%; (2) membranes with selectivity of 8–10 and permeability of 10 barrers for N_2 production with high purity; and (3) membranes with very high selectivity (> 30) and very high permeabilities (>50 barrers) for O_2 production with high purity, i.e. over 90%. Thus, from the available data of permeability and selectivity of polymer membranes, it can be concluded that most of the polymer membranes are suitable for the production of low to moderate purity O_2 and for production of high-purity N_2. Although it is not impossible, high-purity O_2 production using a polymer membrane is still a huge challenge to be addressed. For example, new material such as perovskite. This material has already shows to produces high O_2 purity at elevated temperature (800-900 °C) (Belaissaoui *et al.,* 2014). However, in term of cost, it still high.

There are three methods that can produce oxygen: cryogenic distillation pressure swing adsorption (PSA) and membrane separation. The suitable methods depends on the operation size and O_2 purity needed. Among these three method, cryogenic distillation and adsorption have been established for medium to large scale industry h the plant sizes up range up to 4000 tonne/day oxygen (Chong *et al.,* 2016; Dorris *et al.,* 2016; Allam, 2009). However, these technology still high in term of energy demanding. Due to this, introduction of membrane including polymeric and high temperature ion transport membrane into oxygen production application is an alternative to overcome the problem. Polymeric membrane can produce oxygen enriched air of various concentration, while ion transport membranes can produce purities of close to 100%.

In addition, membrane technology also offered several advantages in term of modular design, ease to scaling up and controlling and low energy requirementswhich leads to lower operating cost (Baker, 2002; Belaissaoui et al., 2014; Delaney et al., 1990; Fernández-Barquín et al., 2016; Konietzny et al., 2011; Murali et al., 2012).

1.2 Problem Statement

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In recent times, many industries use oxygen for a variety reasons, such as in the glass, gasification, and gas-to-liquid industries. The use of high concentration of oxygen offers several advantages such as can reduce heating time, while CO concentration is increased almost linearly with the oxygen concentration. However, the oxygen that usually use in oxygen burner is often pure. This will cause a limitation of widespread application including in combustion industry. Furthermore, the existent combustion system have to be retrofit damage and the retrofitting is very high. One way to reduce the retrofitting and oxygen cost is by decrease the oxygen concentration during the application of oxygen combustion. Previous study reported that minimum requirement of oxygen purity are in the range of 21 % to 28 % in combustion application (Wu et al., 2010). Qiu and Hayden (2009) carried out oxygen-enriched combustion of natural gas in porous ceramic radiant burners, at oxygen concentration varying between 21% and 28 %. The experimental results indicated that the saving in natural gas was about 22 % when oxygen concentration was increased to 28 %. Czakiert et al., (2006) investigated combustion in an oxygen-enriched atmosphere using a circulating fluidized bed combustor. They used brown coal as the fuel, and the oxygen concentration was set as 21 %, 40 % and 60 %. The experimental results showed that the ratio of carbon conversion increased with oxygen concentration. Moreover, oxygen enriched-conditions promoted an increase in the conversion of sulphur present in the fuel.

Cellulose acetate (CA) is a recognized environmental friendly material for making membranes since it is a non-toxic and low cost material. It is widely used as fibres, plastics and membranes in many industrial applications (Lucena et al., 2003). Furthermore, in gas separation technology, CA has been recognised as commercialized material for membrane preparation due to its unique properties of high CO2 and hydrogen sulphide (H2S) (Chen et al., 2015). Nevertheless, the number of plant that used this CA membrane has been decreasing due to its modest CA membrane and consequently reducing CA membrane performance under mixed gas condition (Scholes et al., 2012; Scholes et al., 2009). In addition, the trade-off between the gas permeability and separation performance causing it to be replaced by membrane that is more selective and permeable such as polysulfone, polyimides and polyethylene.

Therefore, several method has been implemented in order to produce CA membrane with high permeability and selectivity including polymer blending, mixed-matrix membrane, polymer modification, composite membrane and many more. Thus, this study is focus on the effect of casting parameters on the morphology and performance of cellulose acetate membrane for low-level of oxygen enrichment.

1.3 Objective

Based on the problem statement, the main objective is fabricate CA asymmetric membrane based on the binary system for gas separation application. Moreover, this study was carried out for several objectives:

(a) To evaluate the effect of varying coagulation bath temperature and solvent evaporation time on physiochemical properties of CA membrane.

(b) To study the effect of NMP addition into the membrane coagulation fluid on the oxygen purification performance.

1.4 Scope of Study

(a) Varying binary dope solution viscosity to determine CA membrane solution critical concentrationTo estimate the parameters

(b) Fabricating an asymmetric membrane based on dry/ wet phase inversion process using manual casting. Manipulation solvent evaporation time (dry phase) between 5 s to 60 s.

(c) Immersing membrane in various coagulation bath conditions (wet phase), including different water bath temperature (5°C to 60 °C) and various NMP composition (0 to 50 %) in the membrane coagulation fluid.

(d) Measuring the permeate gas volumetric flow rate through CA asymmetric membrane using bubble flow meter with pure oxygen and nitrogen gases.

(e) Examining the morphological structure of the produced membrane using Scanning Electron Microscopic (SEM).

(f) Determining the produced CA membrane mechanical properties including tensile strength and young's modulus using tensile test machine.

(g) Measuring the purified oxygen concentration from compressed air (mixed gas) using oxygen concentration tester analyzer.

1.5 Significance of Study

The development of membrane gas separation have gained much attention, due to the advantages such as simplicity and low energy consumption, especially for CA membrane that have been commercialized. However, the study effect of water bath temperature, solvent evaporation time and addition of solvent in coagulation fluid on membrane gas separation application based on binary system was limited and until now, there is no publication regarding binary CA membrane for O_2/N_2 separation. In addition, this research will study the effect of solvent addition in coagulation fluid not only on pure gas, but also with mixed gas. With this regard to the above problem, this study proves by manipulation of above parameters is an alternative for the improvement of cellulose acetate gas separation membrane, thus does not required any additional material on the system, just polymer and solvent. Moreover, the dependent on non-renewable synthetic polymer can be reduced due to the cellulose acetate material itself is from renewable resources.

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LIST OF PUBLICATIONS

Indexed Journal

1. **Jami'an, W. N. R**., Hasbullah, H., Mohamed, F., Wan Salleh, W. N., Ibrahim, N. and Ali, R. R. (2015). Biodegradable gas separation membrane preparation by manipulation of casting parameters. *Chemical Engineering Transaction*, *43,* 1–6. https://doi.org/10.3303/CET1543185. **(Indexed by SCOPUS)**

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Indexed Conference Proceedings

1. **Jami'an, W. N. R**., Hasbullah, H., Mohamed, F., Yusof, N., Ibrahim, N. and Ali, R. R. (2016). Effect of evaporation time on cellulose acetate membrane for gas separation. *IOP Conference Series: Earth and Environmental Science*, *36* (12008), 1-6. http:doi:10.1088/1755-1315/36/1/012008. **(Indexed by SCOPUS)**

2. Mohamed, F., Hasbullah, H., **Jami'an, W. N. R**., Salleh, W. N. H. W., Ibrahim, N. and Ali, R. R. (2016). Effect of coagulant bath on the gas permeation properties of cellulose acetate asymmetric membrane. *IOP Conference Series: Earth and Environmental Science*, *36* (12009), 1-6. http: 10.1088/1755-1315/36/1/012009. **(Indexed by SCOPUS)**

3. Mohamed, F., Hasbullah, H., **Jamian, W. N.** R., Rani, A. R. A., Saman, M. F. K., Salleh, W. N. H. W, and Ali, R. R. (2015). Gas permeation performance of poly(lactic acid) asymmetric membrane for O2/N2 separation. *In ICGSCE 2014. Singapore: Springer Singapore*, 149–156. http: 10.1007/978-981-287-505-1_18. **(Indexed by SCOPUS)**