PRODUCTION OF MEMBRANE FOR CO$_2$/N$_2$SEPARATION EFFECT OF DILUENTS EXTRACTION TIME ON PP-DPE MEMBRANE

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**BACHELOR OF CHEMICAL ENGINEERING (GAS TECHNOLOGY)
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AHMAD FADHIL HANAFI BIN ABDULLAH

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering (Gas Technology)

Faculty of Chemical & Natural Resources Engineering
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<center>**SUPERVISOR'S DECLARATION**</center>

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering (Gas Technology).

Signature   :
Name of supervisor   : DR. SUNARTI BINTI ABDUL RAHMAN
Position   : LECTURER
Date   : 28 JANUARY 2014

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

Signature   :
Name   : AHMAD FADHIL HANAFI BIN ABDULLAH
ID Number : KC10059
Date   : JANUARY 2014

Dedication

THANK GOD ☺

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ABSTRACT

The main factors that contribute for the raising of global warming are CO_2 gas. Production of membrane is actually one of the methods for the separation of CO_2 gas. Membrane technology is continuity and simplicity process compared to the others conventional separation technology. The membranes were prepared from thermal induced phase separation method with 80 wt% of diphenyl ether using the hot steel as a casting with thickness of 500 μm. The membranes were selected to extract into the methanol within 1 hour, 2 hours, 3 hours, and 4 hours of immersion. For the physical and chemical characterization membrane, Scanning Electron Microscopy (SEM) and Fourier Transform Radiation (FTIR) will be used. This research need to calculate the gas permeability and selectivity after single gas permeation is tested with different of pressure. Refer to SEM analysis, the figure for the 4 hours time diluents extraction gives the better pore production compare to 3 hours, 2 hours and 1 hour diluents extraction as the methanol completely remove all the air inside the membrane. If the methanol completely removed the air inside the membrane, the image capture becomes clear and better pore will produced. For FTIR analysis, there have no different functional group inside the membrane after immersed into the methanol. From 1 hour until 4 hours of diluents extraction of membrane have the same functional group of alcohol with the structure of (O-H) stretch that mean the presence of methanol inside the membrane and functional group of ether (C-O-C) stretch and alkanes (C-H) stretch since the membrane is produced by polypropylene and diphenyl ether as a solvent. For the gas permeation, the diluents extraction into methanol for 4 hours gives the highest permeability for the CO_2 and N_2 single gas permeation test. The relation between gas permeation tests with SEM morphology test for the better pore on cross section membrane will cause the good permeability on CO_2/N_2 gas. However, it is difficult to ensure the selectivity of the membrane increase when the permeability increase since the permeability is inversely proportional to selectivity.

Keywords: Polypropylene membrane; methanol immersion; diphenyl ether; gas permeation

ABSTRAK

Faktor utama yang menyumbang kepada peningkatan pemanasan bumi adalah berpunca daripada gas CO_2. Penghasilan membran adalah salah satu cara untuk memisahkan gas CO_2. Teknologi penghasilan membran adalah proses yg lebih mudah berbanding teknologi proses pemisahanan yang lain. Membran telah dihasilkan melalui proses therma pemisahan fasa dengan kepekatan 80 wt% eter diphenyl dengan menggunakan besi panas sebagai alat pemutus dengan ketebalan 500 µm. Membran telah dipilih untuk direndam kedalam methanol selama 1 jam, 2 jam, 3 jam dan 4 jam masa rendaman. Untuk menguji sifat fizikal dan sifat kimia membran, pengimbasan electron mikroskop (SEM) dan fourier transform spectroskopi inframerah (FTIR) akan digunakan. Kajian ini perlu mengira kebolehtelapan dan pemilihan selepas penyerapan gas tunggal diuji dengan perbezaan tekanan. Merujuk kepada SEM, gambar yang mempunyai masa rendaman selama 4 jam mempunyai liang yang lebih baik berbanding 3 jam, 2 jam dan 1 jam rendaman pengekstrakan bahan pencair kerana metanol telah mengeluarkan kesemua udara di dalam membran. Untuk FTIR analisis, tiada perbezaan antara kumpulan berfungsi di dalam membran selepas direndam kedalam metanol. Dari 1 jam hingga 4 jam rendaman, membran mengandungi kumpulan berfungsi seperti alkohol dengan struktur (O-H) regangan membuktikan kewujudan metanol di dalam membran, kumpulan berfungsi seperti eter (C-O-C) regangan dan alkana (C-H) regangan kerana membran diperbuat daripada polipropilena dan diphenyl eter sebagai bahan pencair. Untuk kebolehtelapan gas, rendaman kedalam metanol selama 4 jam menghasilkan kebolehtelapan yang paling tinggi untuk gas CO_2 dan N_2. Bagaimanapun, amatlah sukar untuk memastikan pemilihan membran meningkat apabila kebolehtelapan meningkat kerana hubungan antara mereka adalah berkadar songsang antara satu sama lain.

Kata kunci: membran polipropilena; metanol rendaman; Diphenyl eter; gas penyerapan

.

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LIST OF ABBREVIATIONS

α Selectivity

Δp Pressure different across membrane

LIST OF ABBREVIATIONS

PP	Polypropylene
CO_2	Carbon Dioxide
N_2	Nitrogen
CH_3OH	Methanol
DPE	Diphenyl Ether
SEM	Scanning Electron Microscope
FTIR	The Fourier Transform Infrared Spectroscopy
TIPS	Thermally Induced Phase Separation
PA	Permeability of desired gas (CO_2)
PB	Permeability of desired gas (CH_4)
Pi	Permeability coefficient of gas
Qi	Volumetric flowrate of gas i
A	Membrane effective surface area
l	Membrane skin thickness
(p/l)i	Pressure normalize flux or permeability of gas i
Q	Permeate volumetric rate

1 INTRODUCTION

1.1 Background

In our life, carbon dioxide gas already release to the surrounding naturally. At the same time, many industrial remove the CO_2 gas as a waste product. As a result, the composition of CO_2 in the air will be increase. Unfortunately, this CO_2 gas is not good for human health. Back to the origin, CO_2 gas is a chemical compound of two oxygen atoms that have a covalent bond with a single carbon atom. As a gas, CO_2 is colorless and odorless that is harmful to human health in a concentration higher than 5000 ppm. The first person to discover CO_2 in gas form was Flemish Chemist Jan Baptist Van Helmond in the 17th century, and the properties of CO_2 were further researched by Scottish physician Joseph Black in the 18th century (Clem et al., 2006).

One of the effects that were created by CO_2 gas is the global warming and has been identified as the world's major environmental issues. This problem is made from the greenhouse effect. Actually, the greenhouse's function is to warm the earth by trapping the CO_2 gas inside the atmospheric layer. According to the increasing of CO_2 gas composition in the air that are release from the industries, the greenhouse gives the negative impact for the earth because it trapped too many CO_2 gas inside the atmospheric layer. As a result, the earth's temperature will be increase. Besides that, the increasing of the earth temperature also gives a side effect such as snow melt in the arctic, rise of sea level and shrinking the size of an area of the land (Nomura et al., 2010).

In order to separate this CO_2 gas, there are various types of CO_2 gas removal process such as solvent absorption, solid adsorption, direct conversion, cryogenic fractionators and membranes. This research will focus on membrane separation process. Membrane technology is continuity and simplicity process compared to conventional separation technology. Besides, this technology is flexibility in designed because it can be combined with each other and with other separation technologies to meet complex demand in separation technology. The other characteristic of membrane technologies

that give significant advantages to the industries is the compactness of it design that suitable for the plant that limited in area (Baker, 2002).

Polymer is the substances that can be used to prepare the membrane. In order to produce the membrane, it is important to select the suitable types of polymer. polyvinylidene fluoride (PVDF), polypropylene (PP), polyethylene (PE) and polyvinylchloride (PVC) are an example of the polymer that are suitable to produce the membrane. This research will select the PP for the membrane production. The advantages of choosing PP rather than other polymers is because PP is a plastic material known for its ability to withstand very high temperatures without warping, its general sturdiness, and its water resistance. Normally, CO_2 gas release from the chemical plant in a high temperature. So, it is relevant to choose polypropylene as a material to produces the membrane.

1.2 Problem statement

Separation of CO_2 is one of the important processes in many industrial areas. Membrane gas separation was chose as a CO_2 capture technique. As we know, the main problems is the used of membrane based separation process in a wide range of application is the lack of membrane with high selectivity and permeability. Usually, membrane with high selectivity gravitate to exhibit less permeability. It is difficult to search the membrane with high selectivity and permeability at the same time as the selectivity is inversely proportional to the permeability. It is important to select the most compatible combination of polymer and solvent formulation to get the best performance of the membrane. The diluents extraction time of the membrane into the methanol is one of the significant factors in determining the membrane performance. The methanol actually can be used as an agent to remove the air inside the membrane. When the air inside the membrane is removed, the pore can be produced easily. Therefore, membrane formation based on the formation of the pore is an important part to get a good membrane performance for gas separation.

1.3 Objective

To study the performance of produced polypropylene membrane using gas separation.

1.4 Scope

1. Preparation of membrane using TIPS method from polypropylene and diphenyl ether with percentage 80 wt% of polypropylene with four different times of diluents extraction of membrane into methanol (1 hour, 2 hours, 3 hours and 4 hours times of immersion).
2. Physical and chemical properties of produced membrane are characterized using SEM and FTIR.
3. Test for CO_2/N_2 gas separation using single gas permeation set in FKKSA lab.

2 LITERATURE REVIEW

2.1 Membrane Separation Process

There are many uses of membranes in the industries such as ultrafiltration, reverse osmosis, gas separation and many more. Table 2.1 shows that approximate data for commercialization of membrane technology for various applications according to the year (Perez and Zhang, 1997).

Table 2-1: Commercialization of the membrane technology for various applications according to the year

Technology	Industrial application	Commercialization
Electrodialysis	Desalination of bracklish water	1952
Reverse Osmosis	Desalination of bracklish seawater	1965
Ultra	Paint recovery (electrocoat)	1965
Electrosynthesis	Chlorine/caustic production	1972
Gas separation	Hydrogen recovery	1979
Pervaporation	Alcohol removal from water	1979
Nanofiltration	Softening of hard water	1990
Microfiltration	Filtration of portable water	1994

2.1.1 Ultrafitration

Ultrafiltration is one of the processes that attribute on the separating of small particles and diffuse molecules from fluids. Ultrafilters are the process of removing or rearrangement of sugars, non aqueous solvents, the separation of free from protein-

bound ligands, the elimination of materials of low molecular weight, of the rapid change of ionic or pH environment (Munir, 2006). The factor that can affect the separation process is the molecular size of the molecule. If the molecules have the same molecular size, the process cannot be removed. From the theory, the smaller pore size of the molecules will pass through the filter while the bigger molecules are sustained by the filter then it will be concentrated. The amplitude of material from 1K to 1000K (MW) is contains by certain ultrafiltration membrane, while salt and water will pass through (Maatsura, 1996).

2.1.2 Reverse osmosis

Reverse osmosis is the process where the salts and smalls molecules from lower molecular weight solutes are separated at high pressure (hydrostatic pressure). Usually, reverse osmosis rated by reservation of sodium chloride. The main function of the reverse osmosis is used to purify tap water to purities that excel distilled water quality. Figure 2.1 show the reverse osmosis process.
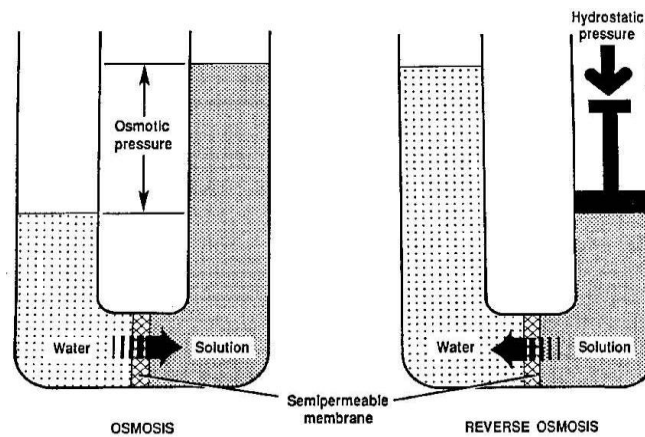
Figure 2-1: Reverse osmosis process

2.1.3 Gas Separation

Membrane gas separation normally used hydrostatic pressure and concentration gradient as a driving force. There are many applications of the membrane gas separation especially for the hydrogen gas mixture. Hydrogen recovery for synthesis gas ration

adjustment for H_2/CO_2 gas, H_2 recovery from hydroprocessing purge stream and H_2 recovery from ammonia plant purge streams and other petrochemical plant stream. Other than that, it also can be used to separate oxygen/nitrogen mixtures, recovery for helium gas and biogas processing (Scott, 1998).). Table 2.2 below shows application of membrane gas separation units.

Table 2-2: Application of membrane gas separation units (Nunes and Peinemann, 2006)

Gas Separation	Application
O_2/N_2	Nitrogen generation Oxygen enrichments
H_2/hydrocarbon	Refinery hydrogen recovery
H_2/CO	Syngas ration adjustment
H_2/N_2	Ammonia purge gas
CO_2/CH_4	Acid gas treating enchanced oil recovery landfill gas upgrading
H_2S/hydrocarbon	Sour gas treating
H_2O/hydrocarbon	Natural gas dehydration
H_2O/air	Air dehydration
Hydrocarbon/air	Pollution control hydrogen recovery
Hydrocarbon from process streams	Organic solvent recovery monomer recovery

2.1.3.1 History of membrane in gas separation system

In 1829, Thomas Graham found the gaseous osmosis for the air carbon dioxide system through a wet animal bladder. (Kesting and Fritzsche,1993). After that, in 1831 J.K. Mitchell noted that CO_2 gas was observed by rubber film to a larger degree than other gases, and was led to infer, accordingly, noticed that rubber expand with volume hence porosity was induced in solid sample which provide awa of penetration of CO_2 molecules. Then, 1866 Graham's law of diffusion was found. He describe about "sorption diffusion" theory of gas transport through membrane. In his researched, gas is permeated through a film (natural rubber) into vacuum not into air. He set up a series of relative permeation rates across the film for a number of gases that is amazing lose to modern estimates of the corresponding properties then state that no relation between these values and known diffusion coefficients in gases. Graham then, test for the first membrane gas separations and acquire oxygen riched air containing 46.6% oxygen. He discover that changes in the thickness of films affects the flux but not the composition of permeate gas. On 1891, Kayser show the validity of Hendry's law for adsorption of CO_2 in natural rubber (Raul and Yampol' ski, 1994). The information and knowledge about the gas separation in membrane continues discovered until nowadays.

2.1.3.2 Absorption

Absorption application was found to remove the acid gas such as carbon dioxide and hydrogen sulphide (Maclean et al, 1986). This process actually is a physical process where a gas selectively diffused in a liquid and subsequently recovered through the effect of heat, pressure or chemical. The absorption take place when normal boiling point of component operated or one or more of component have strong affinity for a particular solvent. One of the examples the application of absorption is CO_2 removal for synthesis gas and for scrubbing CO_2 and sulphur compound from natural gas.

2.1.3.3 Adsorption

Adsorption process apply the technique porous solid such as zeolite, carbon molecular sieve and aluminosilicates material to prefer adsorb one gaseous species versus others. The adsorbent is packed in carbon steel vessels and a higher pressure is used to adsorb while a lower pressure is used to desorb.

2.1.3.4 Membrane

Membrane is a thin barrier between feed and permeate gas stream and had been used to carry fluids. Thin asymmetric membranes was first assembly which contain of a thin, dense, outside layer was formed on a porous base layer, thick and developed from cellulose acetate (Loeb and Sourirajan, 1963). Gas separation is used to separate the mixture of gases using porous or non- porous membrane. This mechanism is quite different of transport involved. Separation of porous membrane is through the difference in Knudsen flows of component in the pores which are less size than mean free path of the molecules. Meanwhile, separation of gas through non-porous depend on different in permeability's of gases.

2.1.3.5 Comparison between gas separation

Summary of the gas separation system with their performance as shown in Table 2.3

Table 2-3: Comparison of Gas Separation Systems

Process	Advantages	Disadvantages
Cryogenic Distillation	• Low power consumption • Could produce high purity products. • Could achieve higher recovery compared to other process.	• Unable to economically scale down to very small size. • Consist of highly integrated, enclosed system which does not permit easy handling of widely varying feed streams.
Adsorption	• Could obtain high purity of products. • Can be supplied to remote locations where equipment size is critical.	• Lower recovery of products. • Single relatively pure product.

Absorption	• Excellent for CO2 and H2S Removal	• High partial pressure needed for physical solvents • Low partial pressure needed for chemical solvent slow purity of acid gas
Membrane	• Versatility • Simplicity • Stable at high pressure • Could achieve high purity of product.	• Possible recompression of permeate. • Medium purity.

2.2 Membrane Module

Membranes actually need a module for large surface area. Many types of module are used in the industries such as plate and frame module, tubular module, hollow fiber module and flat sheet module. There are many type of membrane that can be used as a good separator such as flat sheet into spirally wound modules or bundles of hollow fibers and casting epoxy resins on both ends and then encasing the bundle in carbon steel shells with appropriate entrance and exit nozzles (Maclean et al, 1986).

2.2.1 Plate and Frame

Plate and frame module is the uncomplicated module's structure and not arrogation for membrane replacement. This module has its own advantages such as low volume hold up per unit membrane area and ability to process highly viscous solution because of thin channel height (0.3-0.6 mm) (Belfort, 1988). This plate support a configuration which is closest to the plate membranes used in laboratory. The skills to apply this module firstly, sets of the two membranes are placed in a sandwich like with their feed sides facing each other, in each feed and permeate compartment thus obtained a suitable spacer is placed. The number of sets needed for a given membranes are furnished with sealings rings and the two end plates then builds up to a plate and frame stack.

2.2.2 Tubular

This module process need a number of membranes of tubular shape are closed in a container. The feed will flow through the center of the tubes while the permeate flow through the porous supporting tube into the module housing. Tubular module configuration commonly introduced by ceramic membrane. Energy consumption increases to per unit amount of liquid. Unfortunately, this module cannot operate by itself and need to be supported by a tube from outside.

2.2.3 Hollow Fiber

This module contains of a large number of fibers join together in a module. The self supporting membrane is suitable to use in this module. There are two arrangement of this module firstly, inside out where the feed solution passes through the bore of the fiber and the permeated is collected on the outside of the fiber. Secondly, outside in where the feed solution enters the module on the shell side of the fibers and the permeated passes into the fiber bore. Common diameters of hollow fibers are 25-200 μm. Since no breaks or defects are allowed in a module, this requires very high standards of reproducibility and quality controls (Baker et al., 1991).

2.2.4 Flat Sheet

The flat sheet membrane is different among others because it is a simplest membrane module and easy for replacement. It is easy to prepare, handle and mount. Two membranes are placed in a sandwich-like fashion with feed side was feed each other (Teo et al., 2008). Figure 2.2 shows the flat sheet membrane.
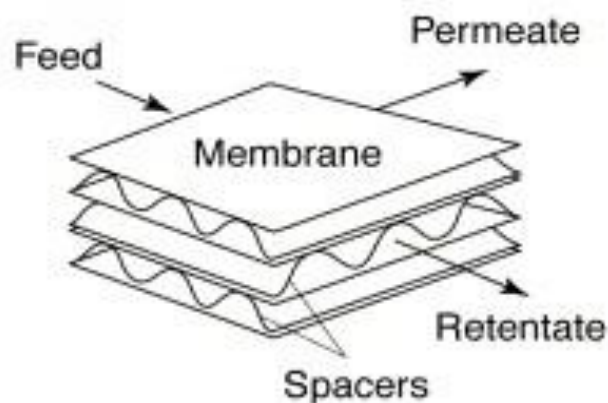
Figure 2-2: flat sheet module

2.3 Membrane Classification

There have a few sample of membrane listed that had been found but most of them still have the similar main principle and configuration.

2.3.1 Microporous Membrane

Membrane react nearly such a fiber filter and separates by a sieving mechanism detected by the pore diameter and particle size. Substances such as ceramics,graphite, metal oxide and polymers were used in making such membranes (Scott et al.,1998).

2.3.2 Electrically Charged Membrane

An ion changing membranes consist of highly swollen gels conducting a fixed positive or negative charges. These are generally used in the electro dialysis.(Nunes *et al.*, 2003)

2.3.3 Assymmetric Membrane

Asymmetric membranes are consumed primarily for pressure driven membrane processes, such as ultrafiltration and gas separation. Their structure consist of a very thin (0.1 to 2.0μm) polymer layer on highly porous 100 to 200μm thick sublayer (Strathmann, 1986). The sublayer only acts as a support and does not affect the separation characteristics or the permeation rate of the membrane in pressure driven processes. To obtain high permeation rates, the selective layer of gas separation membranes must be extremely thin (Baker, 2002). The advantage of asymmetric membranes is the membranes are surface filters retaining all the rejected materials at the surface where they can be release by shear forces applied by the feed solution moving parallel to the membranes surface (Costello, 1994)

2.4   Method to Produce the Membrane

2.4.1 Phase Inversion

This process is a casting solution consisting of polymer and solvent is immersed into a non-solvent coagulation bath. The mixtures then dissolved causes the casting solution will go through a phase transition by which the membrane then formed. The phase inversion process involves two different types of phase transition which is liquid-liquid phase, two-phase region and solidification phase (Pinnau 1991). There are many type of phase inversion method such as thermal precipitation, air casting of dope solution, precipitation from the vapor and immersion precipitation.

2.4.1.1 Immersion Precipitation

Immersion precipitation casting of a polymer as a thin film on a support or extruded through a die, and is subsequently immersed in a non-solvent bath. Precipitation can occur because of the good solvent in the polymer solution is exchanged with non-solvent in the coagulation bath (Fleming, 1998).

2.4.1.2 Air Casting of Dope Solution

Air casting technique process used the polymer to dissolve in a mixture of a volatile solvent and less volatile non solvent. During the evaporation of the solvent, the solubility of the polymer decreases and then phase separation will occur.

2.4.2 Thermal Phase Induce Separation

Membrane preparation using thermal induce phase separation (TIPS) is the preparation of membrane includes phase separation of polymers. In the late 1970s and early 1980s, TIPS was assembly by Castro and then patented by several people for the preparation of microporous polymeric membranes and used by various companies (Chen et al., 2009). The advantage of TIPS is suitable for wide applications for semi-crystalline polymer. On the other hand, the process is flexible, good mechanical properties, thermal stability, chemical resistance and low cost. TIPS method is operate to formed pore in the membrane layer. One efficient way to control pore size in the layer by cooling both side of the uniform solution. Using TIPS the polymer must be mix with diluents. After that, the membrane will produced and started to be characterized and tested its performance.

3 METHODOLOGY

3.1 Research Design

Figure 3.1 shows the flowchart of the workflow in this research.

Membrane dope solution	
Diphenyl ether (80%)	Polypropylene (20%)

↓

Casting process	
Hot Steel	500 micro metre

↓

diluents extraction	
methanol	1 hour - 4 hours

↓

membrane characterization	
SEM	FTIR

↓

gas permeation test	
CO_2 gas	N_2 gas
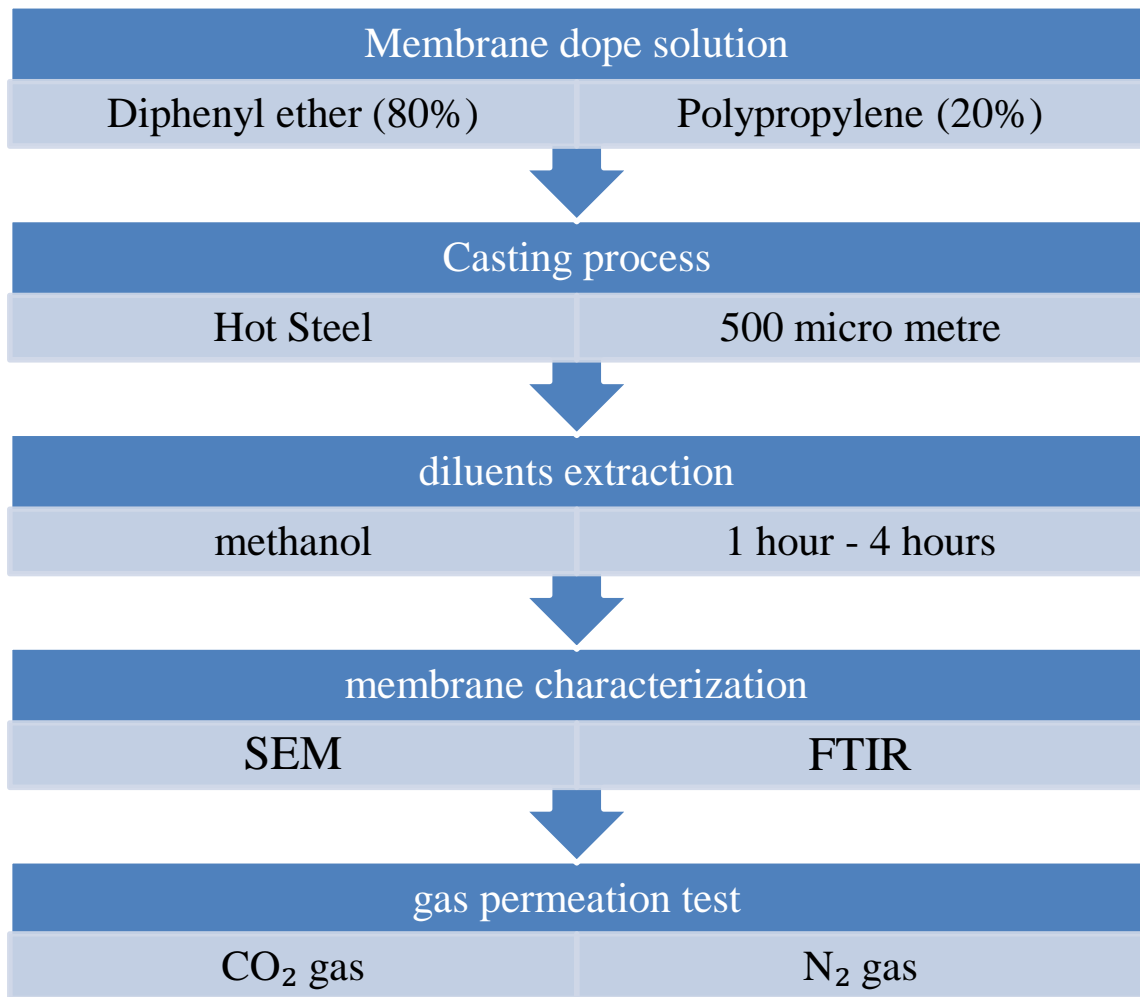
Figure 3-1: the flowchart of the workflow

3.2 Material Selection

3.2.1 Isotactic Polypropylene (iPP)

Isotactic Polypropylene (iPP) from Sigma Aldrich was used in this experiment. Figure 3.2 below shows the chemical structure of iPP.

Figure 3-2: the chemical structure of iPP

Table 3-1: basic Properties of (iPP).

Properties	Values
Melt index	12g/10min(230°C/2.16kg)
Mol wt(g/mol)	Average Mw= 250 000
Hardness	100 (Rockwell R, ASTM D 785-A)
Transition Temperature (°C)	160-165
Density (g/mL)	0.9
Melting Point (°C)	160-166

3.2.2 Diphenyl Ether

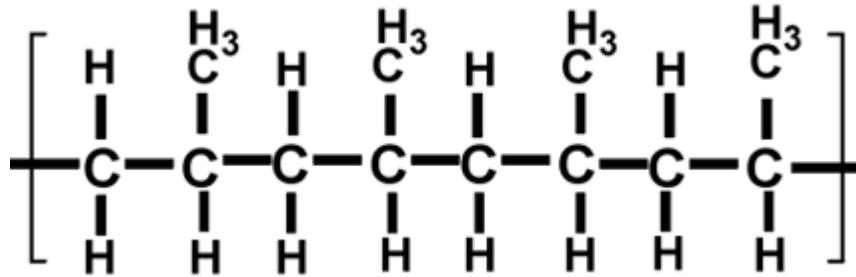Diphenyl ether which also supplied from Aldrich was used as a diluent without any further purification with CAS number 101-84-8. Figure 3.3 shows the chemical structure of diphenyl ether (DPE).
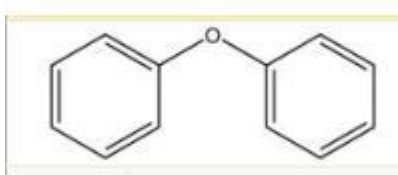

Figure 3-3: the chemical structure of diphenyl ether (DPE).

Table 3-2: basic properties of DPE

Properties	Values
Vapour density	>5.86(25°C, vs air)
Vapour pressure	<1 mmHg (20°C)
Refractive index	n20/D 1.579 (litre)
Boiling point	259°C (litre)
Melting point	25-27°C (litre)
Density	1.073g/mL, 25°C (litre)

3.2.3 Carbon Dioxide and Nitrogen Gas

The membranes were tested using pure carbon dioxide and nitrogen gas as test gases. Table 3.3 shows the properties of pure carbon dioxide and nitrogen.

Table 3-3: the properties of pure carbon dioxide and nitrogen

	Carbon Dioxide	**Nitrogen**
Molecular Formula	CO_2	N_2
Molar Mass	44.01 g/mol	16.043 g/mol
Kinetic Diameter	-	-
Viscosity	-	-
Appearance / Odor	Colourles/ odorless gas	Colourless gas, liquid, or solid
Melting Point	-78˚C	-210˚C
Boiling Point	-57˚C	-195.79˚C

3.3 Preparation of Casting Solution

Polypropylene was first dried in a vacuum oven overnight at temperature of 60˚C in order to eliminate all absorbed water vapor before used in making dope solution. The polypropylene and solvent were weighted according to the required concentration 80wt% of DPE and 20wt% of PP with basic of total mass 20g. The polymer and diluent were mixed in a beaker, which was first sealed with aluminium foil to prevent the gas evaporate to the surrounding. The sealed beaker with a small hole was placed on the hot plate and the hot plate was put into the fume hood because the diphenyl ether will disperse the gas when it evaporated. The solution was heated and stirrer until the solution become homogeneous (the solution looks like a clear solution).

3.4 Membrane Casting

Flat sheet polypropylene membranes were prepared according to the thermal induced phase separation process using casting technique. Before the casting process is work, the hot steel need to be heated on the hot plate to ensure that the solution will not froze immediately after pour the solution onto it. Usually, the casting process was conducted at room temperature. A small amount of homogeneous solution was poured onto the hot steel with the thickness of 500 μm. Then, the solution was close with the other side of the hot steel. After that, the cast membrane was immersed into water to initiate delamination and slightly peel the membrane from the steel plate. After that the produced membrane was extracted into the methanol according to the time taken of study. The membrane was selected to be extracted into the methanol within 1 hour, 2 hours, 3 hours, and 4 hours of immersion. Then, the membrane was wiped with the tissues and dried again into the oven about 12 hours with the temperature of 30°C to reduce the smell of the membrane because diphenyl ether have an odour of itself and dangerous when we inhaled it. After 12 hours, the smell of the membrane was not so strong likes before it dried. Then the sample was ready to be analyzed. Figure 3.4 shows the casting steel used.

Figure 3-4: casting steel

3.5 Gas Permeation Test

The performance of polypropylene membrane was test with pure gas permeation test. Carbon dioxide and nitrogen were used as test gas. Firstly, the membrane will be cut into a small circle with diameter 48cm. The size of circle membrane must to suite with the permeate core in order to avoid any leakage of pure gas. Then, the circle membrane will be pleated and folded around the permeate core. The separation of pure gas across fabricated membrane will be tested at range pressure between 1-3 bars. When the permeability unit already setup, the pure gas will be turn on and flowing through the membrane fitted in it. The volume different of the soap water by each membrane and the time of the first bubble formed was taken. Figure 3.5 illustrated the overall gas permeation system. The system was included the flexible hose, permeation cell, needle valve, pressure regulator and bubble flow meter. Gas permeation system was made up to measure pure gas permeation rate for asymmetric flat sheet membrane and approximately their selectivity.
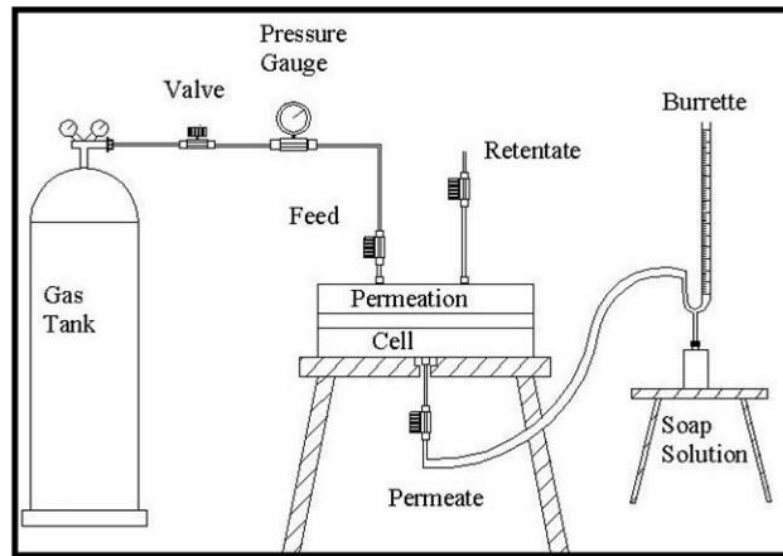
Figure 3-5: Gas Permeation System

The permeability is defined as the transport flux of material through the membrane per unit driving force per unit membrane thickness. The permeability investigated for CO2 and N2 gases. The gas permeability (P) was calculated using equation (3.1) and expressed in Barrer (1 Barrer = 1 x 10-10cm3 (STP) cm (cm2scmHg)).

$$P = \frac{Ql}{A\Delta p} \quad \text{(cm}^3\text{(STPcm))/(cm}^2\text{scmHg)} \tag{3.1}$$

The permence (P/l) for iPP membranes was calculated using equation equation (3.2) and Expressed in GPU (1 GPU = 1 x 10-6 cm3 (STP)/ (cm2scmHg)).

$$\frac{P}{l} = \frac{Q}{A\Delta p} \quad \text{(cm}^3\text{(STPcm))/(cm}^2\text{scmHg)} \tag{3.2}$$

Where l is the thickness of membrane in cm, A is the effective membrane area in cm2, V the volume (cm3) displaced in time t (s) and Δp is the transmembrane pressure expressed in cmHg. The selectivity (α) is expressed as the ratio of two pure gas permeance (in case of PP membranes or of permeabilities (in case of dense membranes.

3.6 Membrane Characterization

3.6.1 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy is an apparatus that is capable of magnifying a very discrete surface into a larger and clearer image. This methods is used for analyze the membrane surface to examine the morphology or specifically the membrane structure, pore distribution, defect and presence of impurities. A small piece of the tested membranes were cut and were dried then immersed to leave an undeformed structure and mounted on sample stubs. The samples were recovered with a thin platinum layer (Baltec) and placed on a support in the SEM. The morphologies (cross section and surface layer) were then view with scanning electron microscopic (Carl Zeiss EVO50) with potential of 20kV under magnifications ranging from 50X to 6000X.

3.6.2 Fourier Transform Infrared Radiation (FTIR)

FTIR were performed in order to study the chemical structure of organic molecules and potential structural changes that occur as a result of the membrane chemical treatment or degradation. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). FTIR spectra of thin films were recorded. It resulting spectrum represents the molecular absorption and transmission, creating a molecular finger prints of the sample. Like a finger print no two unique molecular structures produce the same infrared spectrum. These can be seen from the spectrum observed (n term of the wave number and the wave length) in order to know the different occurred in the membrane.

4 RESULT AND DISCUSSION

4.1 Effect of the diluents extraction of the membrane into the methanol on the membrane morphology

The SEM photographs of cross section for 1 hour, 2 hours, 3 hours, and 4 hours of time of immersion of membrane into the methanol specimens are taken under identical magnification. The comparisons of cross section image for different time of diluents extraction show the asymmetric nature of this membrane. The typical morphology of 4 hours of diluents extraction is shown in figure 4.1.
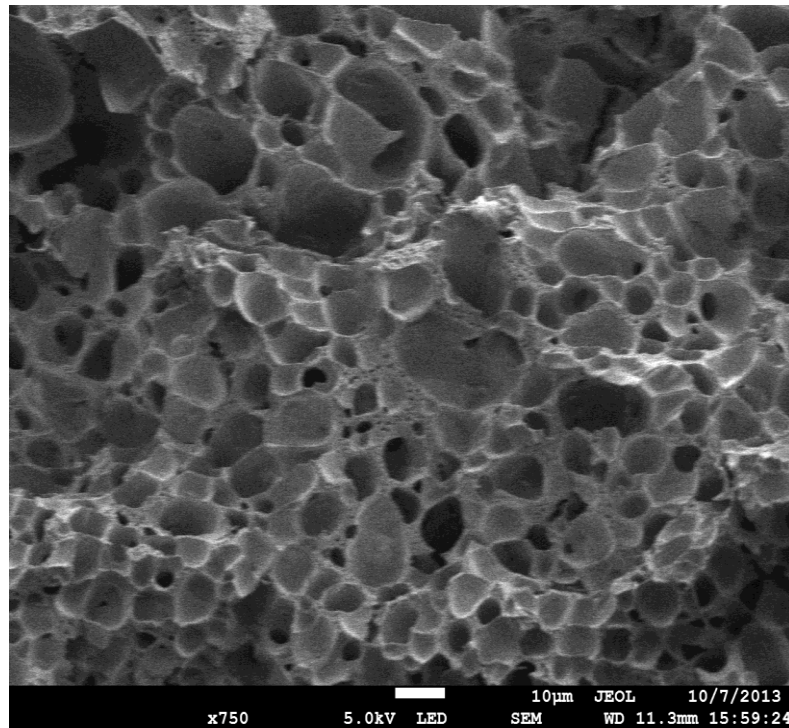

Figure 4-1: morphology cross section image of 4 hours diluents extraction

Based on the figure 4.1 we can see that the image form is clear and much pore was produced. The factor that affects the number of production of pore is the time of diluents extraction of the membrane into methanol. The longer the time of immersion into methanol makes the air inside the membrane is removed and the number of pore will increase since they are no disturbance by the air inside the membrane. However, if too long times taken when diluents extraction into methanol will cause the membrane lost the pore. To ensure the membrane release all the air inside the membrane without

effect the pore production, the optimum time for the diluents extraction need to be investigated. Figure 4.2, figure 4.3 and figure 4.4 show the morphology of 1 hour, 2 hours and 3 hours of diluents extraction image.
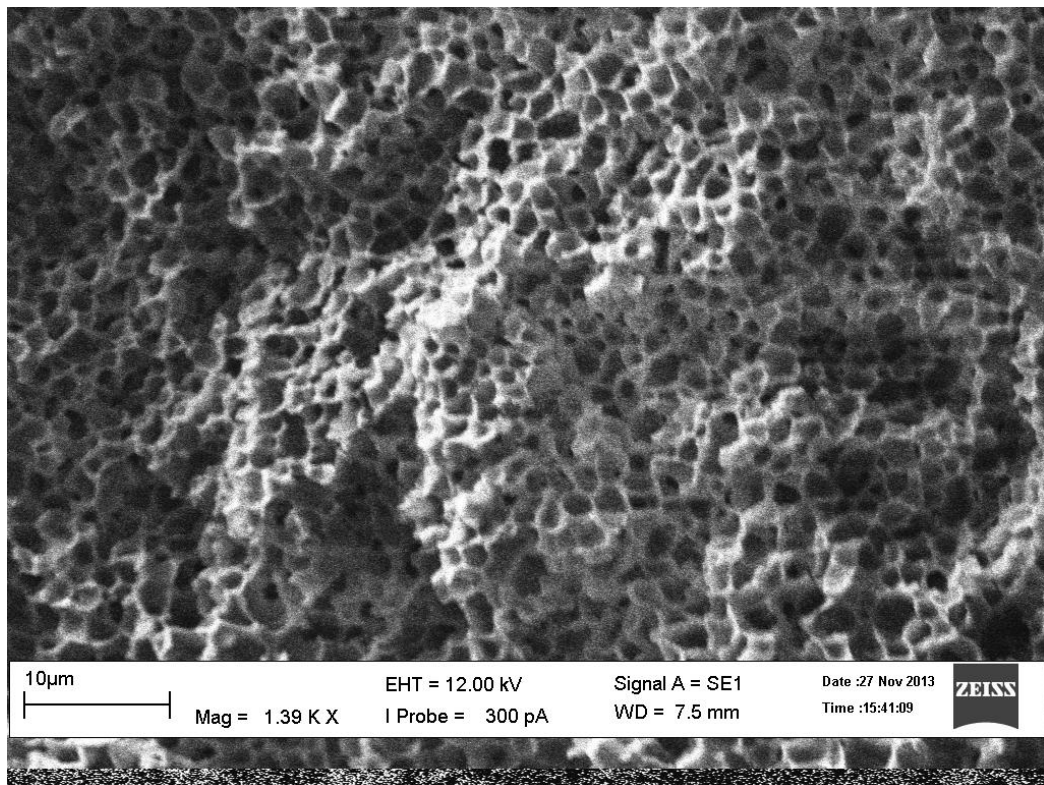

Figure 4-2: morphology cross section image of 3 hours diluents extraction
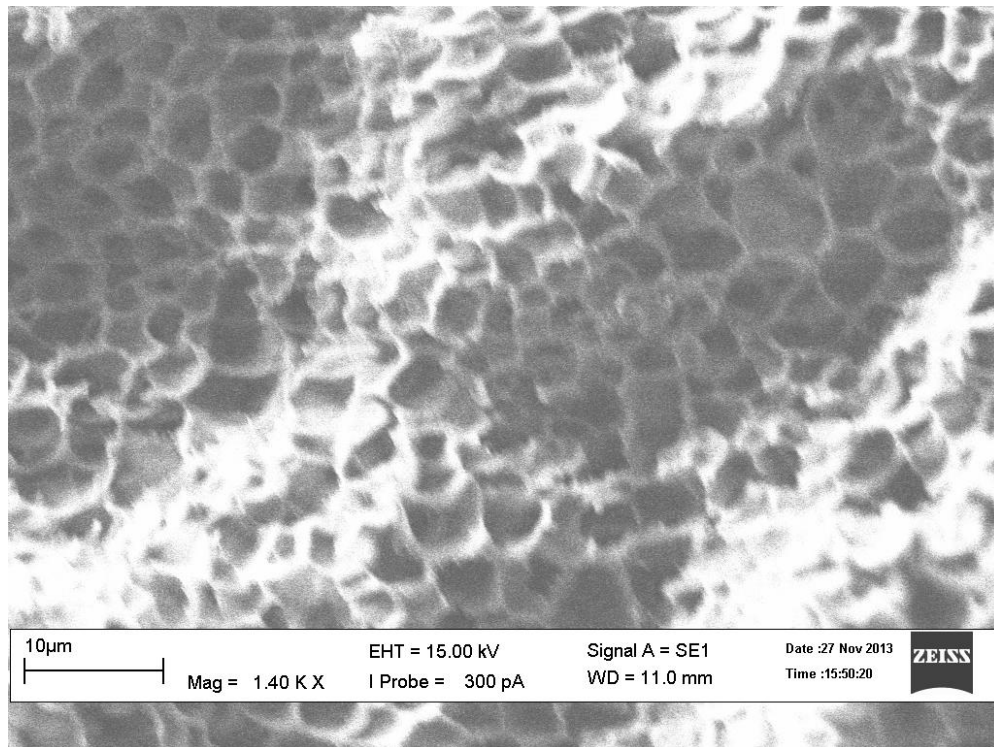
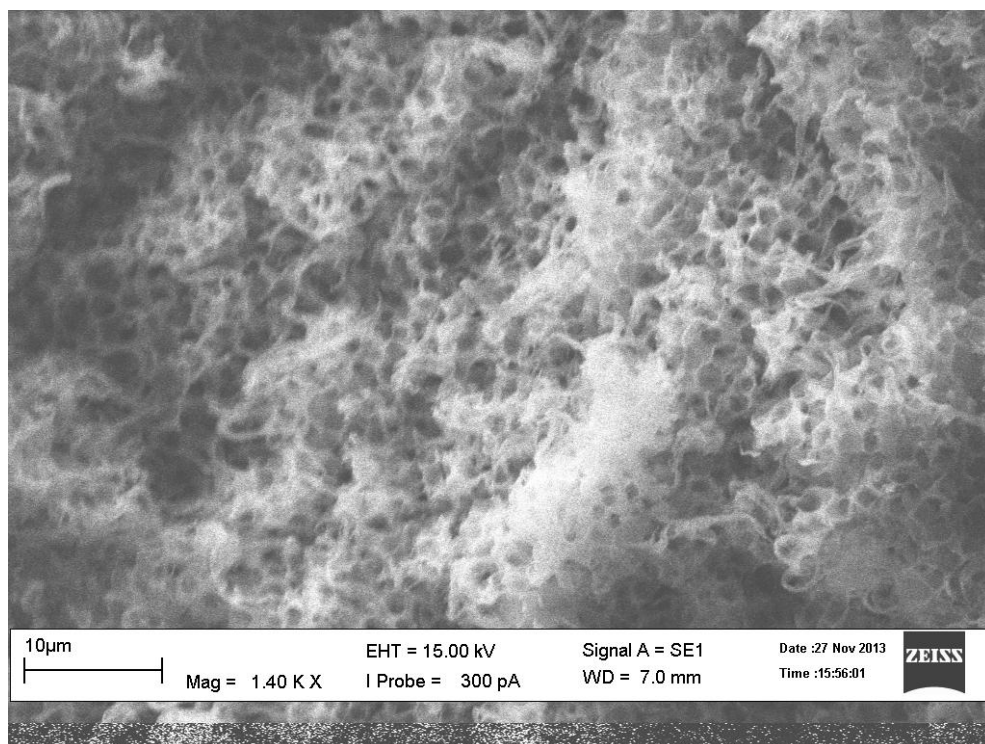Figure 4-3: morphology cross section image of 2 hours diluents extraction

Figure 4-4: morphology cross section image of 1 hour diluents extraction

4.2 Effect of the diluents extraction of the membrane into the methanol on the FTIR analysis

Fourier Transform Infrared Spectroscopy (FTIR) is an equipment to gain information about the membranes's chemical characteristics from unknown material in fabricate the membrane and determine the amount of components in the mixture. Therefore the relation of the chemical structure or molecular structure of materials can be determined after doing FTIR test. Usually, the bond or the group of bonds will be vibrating at characteristic frequency. Based on Ismail et al., (1997) In order to increase the selectivity of the membrane, the shear induced has been shown and directly measure using plane-polarized Fourier Transform Infrared Spectroscopy. Table 4.1 shows the wavelength numbers of the FTIR groups for PP-DPE membrane with different time of diluents extraction.

Table 4-1: the wavelength numbers of the FTIR groups for PP-DPE membrane with different time of diluents extraction.

Wavenumber(cm^{-1})	Groups	Class of compound
2400	OH stretch	Alcohol
2800-2950	C-H stretch	Alkanes
3650	O-H bond	Alcohol
1000-1300	C-O-C stretch	Ether

Based on the table, all the different time of diluents extraction for PP-DPE membranes have the same functional group but different in wavelength numbers. All these membranes contain alkanes, ethers and alcohol functional group. The ethers were determined as the peak range from 1000-1300 cm^{-1} for the (C-O-C) stretch dialkyl functional group. It was proved the presence of diphenyl ether as a solvent for the membrane. Furthermore, it also can be seen that there were a few absorption peaks detected at range of 2400 cm^{-1} wavenumber which were in (O-H) strech functional group. This kind of wavenumber proved the presence of water or alcohol that used as diluents extraction in membrane production. The most important absorption bond at range 2800-2950 cm^{-1} wavenumber representing C-H stretch bending was due to alkanes bonded to the polymer backboned. Other than that, absorption peak at range 3650 cm^{-1} wavenumber also for the (O-H) stretch functional group also for the presence of alcohol. Figure 4.5, figure 4.6, figure 4.7, figure 4.8 and figure 4.9 show the

absorbance peaks of extraction time of 1hour, 2hours, 3hours, 4 hours and comparison graph of PP-DPE membrane with different diluents extraction time respectively.
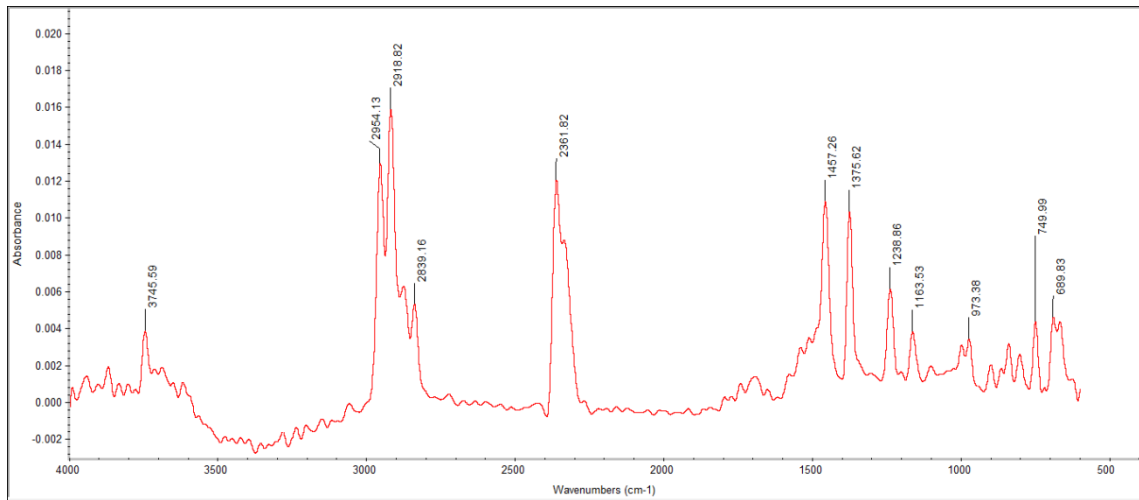

Figure 4-5: absorbance peak of extraction time of 1 hour
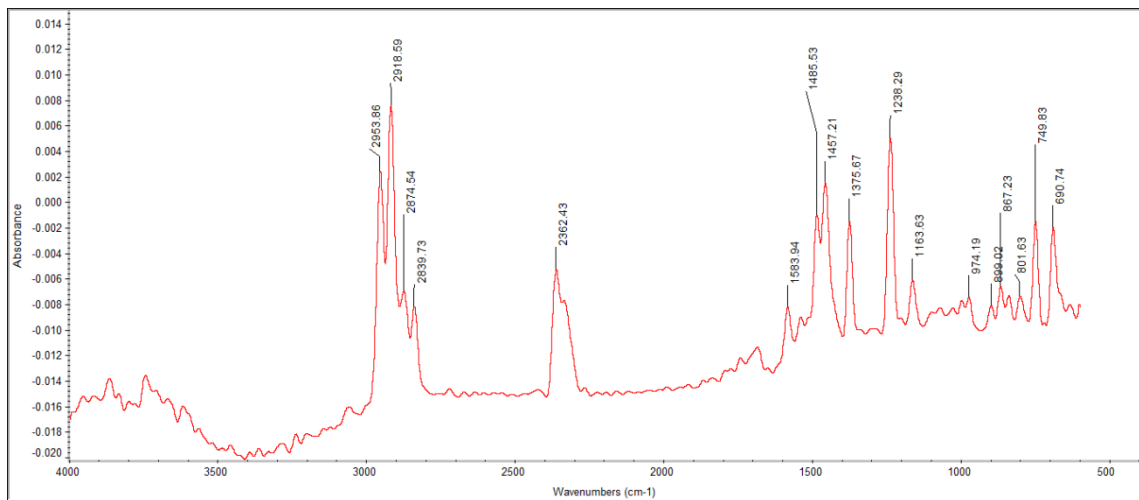
Figure 4-6: absorbance peak of extraction time of 2 hours
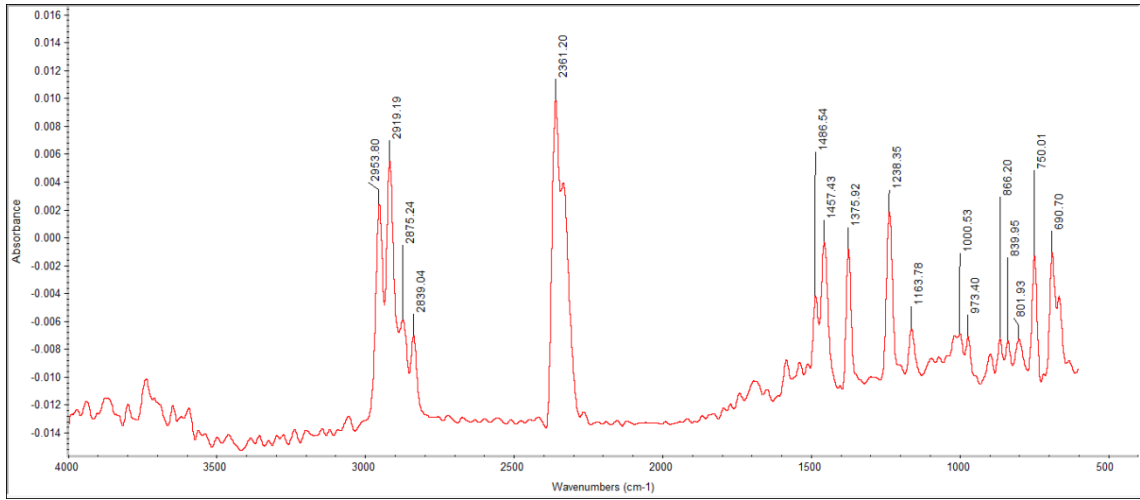
Figure 4-7: absorbance peak of extraction time of 3 hours

Figure 4-8: absorbance peak of extraction time of 4 hours

Figure 4-9: comparison graph of PP-DPE membrane with different diluents extraction time

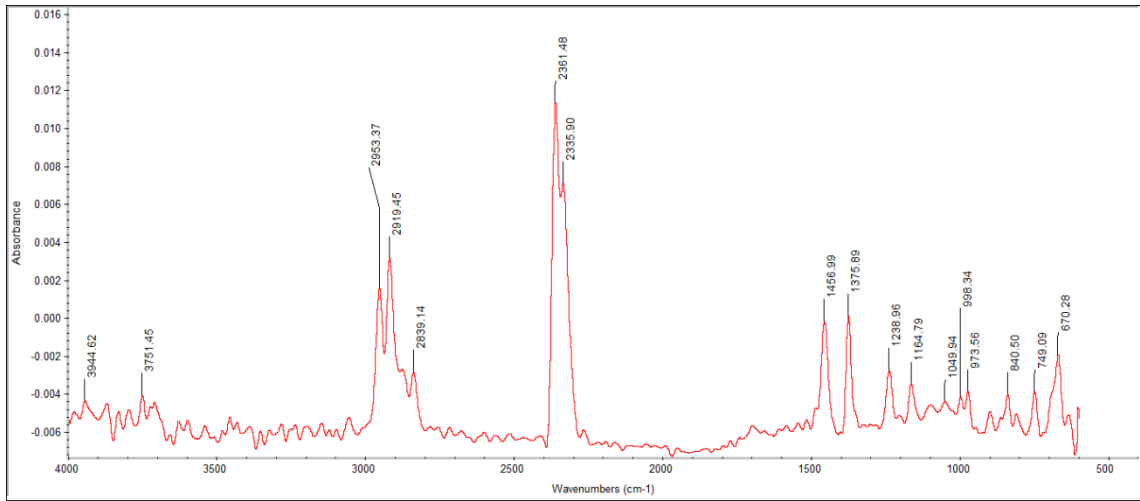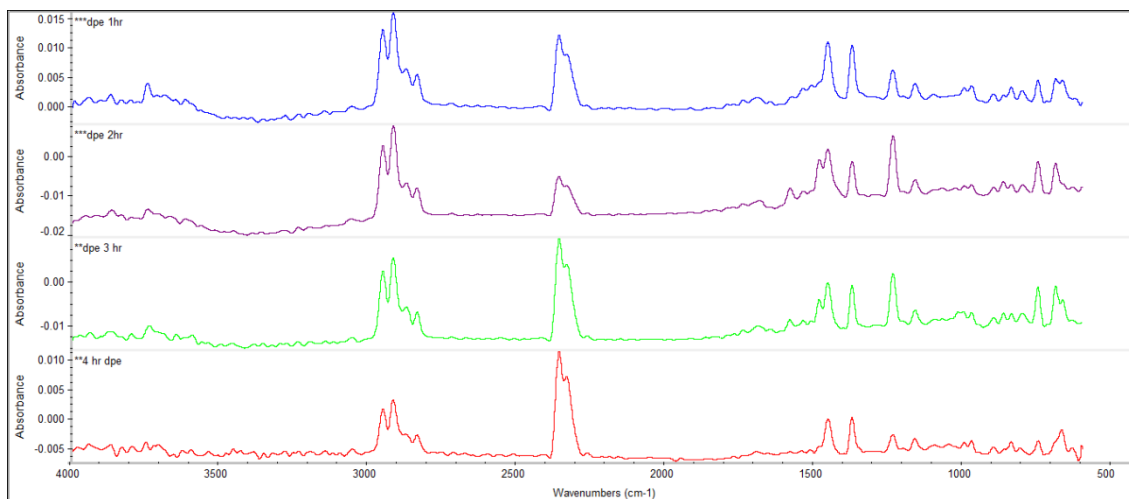As a result, we can see that all the diluents extraction time until 4 hours still contain the alcohol, ethers and alkanes group. Based on the graph above, we can conclude that the diluents extraction time does not affect the FTIR analysis on PP-DPE membrane.

4.3 Effect of the diluents extraction of the membrane into the methanol to the performance of PP-DPE membrane

4.3.1 Effect of pressure on CO_2 permeation for PP-DPE time of diluents extraction of the membrane

This research will discuss the effect of PP-DPE membrane on permeation gas test when the membrane has a different time of diluents extraction. Based on Fujioka (2009), gas separation in membrane is driven by a pressure difference across the membrane. Table 4.2 shows the effect of pressure on volumetric flowrate of CO_2 gas permeation for PP-DPE time of diluents extraction of the membrane.

Table 4-2: effect of pressure on volumetric flowrate of CO_2 gas permeation for PP-DPE time of diluents extraction of the membrane

Pressure, cmHg	Diluents extraction time, hr	Flowrate, ml/min	Time taken, min
0.076	1	0.54	1
	2	1.03	1
	3	1.56	1
	4	1.80	1
0.152	1	0.70	1
	2	1.05	1
	3	1.62	1
	4	1.93	1
0.228	1	0.88	1
	2	1.20	1
	3	1.75	1
	4	2.01	1

Based on the table we can see that the increase of pressure will increase the volumetric flowrate. For the diluents extraction of 1 hour, the flowrate increase from 0.54, 0.70, to 0.88 from 1 bar to 3 bars pressure supply. For the diluents extraction of 2 hours, the flowrate increase from 1.03, 1.05, to 1.20 from 1 bar to 3 bars pressure. 3 hours diluents extraction, the flowrate increase from 1.56, 1.62 to 1.75 and lastly for 4 hours diluents extraction, the flowrate increase from 1.80, 1.93 to 2.01 from 1 bar to 3 bars pressure supply. We can see that the longer time of diluents extraction will increased the volumetric flowrate. Figure 4.10 shows the volumetric flowrate versus time of diluents extraction graph for CO_2 gas.
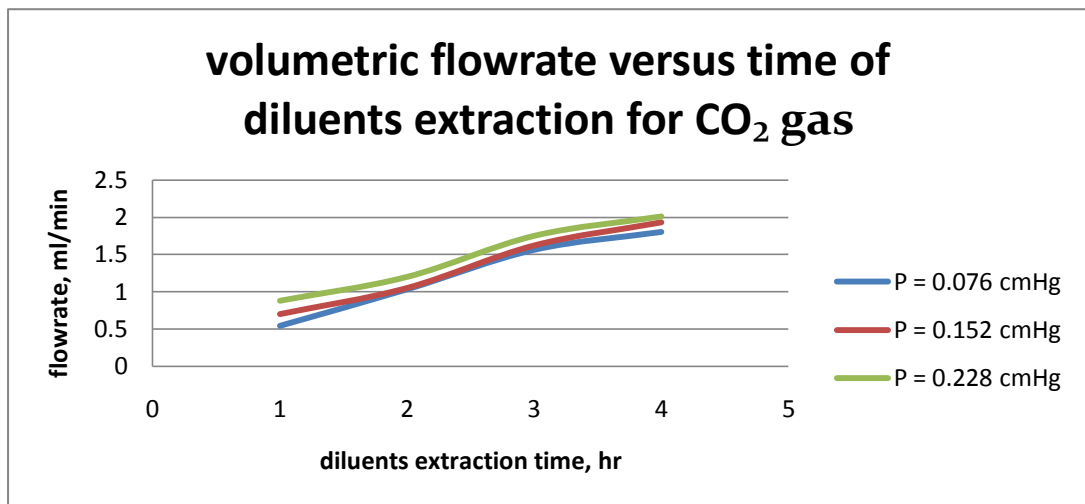
Figure 4-10: the volumetric flowrate versus time of diluents extraction graph for CO_2 gas

4.3.2 Effect of pressure on N_2 permeation for PP-DPE time of diluents extraction of the membrane

The effect of pressure on N_2 gas volumetric flowrate is actually lower than the CO_2 gas volumetric flowrate. Based on Ismail (2004) the size of the CO_2 gas 3.4 angstroms is smaller than N_2 gas 3.6 angstroms. From the theory, we can say that the volumetric flowrate of N_2 gas must be lower than CO_2 since the size of N_2 gas is bigger than CO_2 gas. Table 4.3 shows the effect of pressure on volumetric flowrate of N_2 gas permeation for PP-DPE time of diluents extraction of the membrane.

Table 4-3: the effect of pressure on volumetric flowrate of N_2 gas permeation for PP-DPE time of diluents extraction of the membrane.

Pressure, cmHg	Diluents extraction time, hr	Flowrate, ml/min	Time taken, min
0.076	1	0.11	1
	2	0.36	1
	3	0.72	1
	4	0.85	1
0.152	1	0.05	1
	2	0.28	1
	3	0.77	1
	4	1.49	1
0.228	1	0.04	1
	2	0.17	1
	3	0.66	1
	4	1.07	1

From the table, we can see that the volumetric flowrate of N_2 gas still lower compare to CO_2 gas volumetric flowrate. When the size of the gas is bigger, the gas only can permeate into the bigger pore produced by the membrane. This permeation is proved by looking at the volumetric flowrate of N_2 gas itself. As well, we also can see that the increase of pressure will increase the volumetric flowrate of N_2 gas. Furthermore, we can see that the longer time of diluents extraction affect the volumetric flowrate since the 4 hours of diluents extraction shows the highest volumetric flowrate at the same pressure supplied. Figure 4.11 shows the volumetric flowrate versus time of diluents extraction graph for N_2 gas.

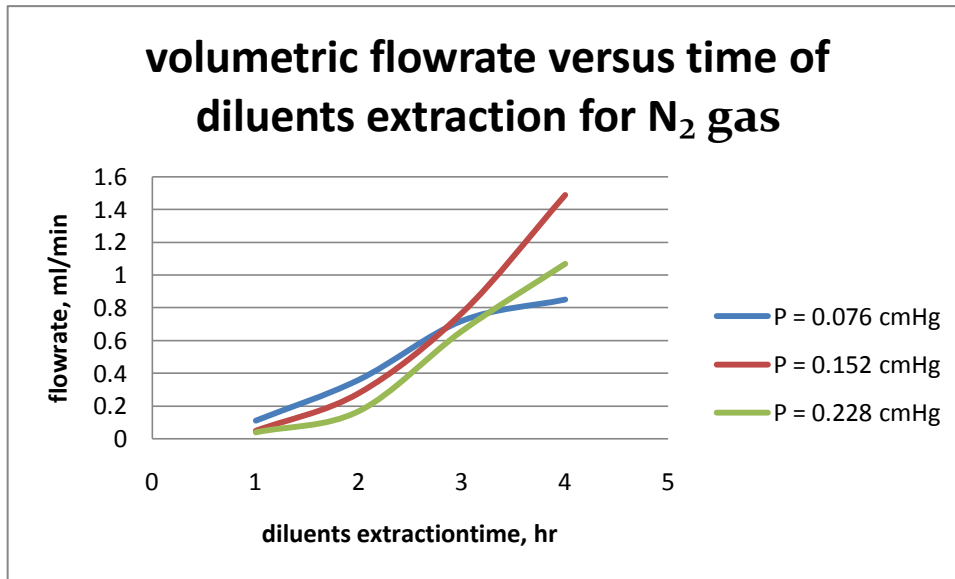Figure 4-11: the volumetric flowrate versus time of diluents extraction graph for N_2 gas

Based on the graph, the N_2 gas volumetric flowrate results were fluctuating. There were maybe had some error while collecting the data of volumetric flowrate but still the diluents extraction for 4 hours of immersion shows the highest flowrate for the same pressure supplied.

4.3.3 Effect of pressure on permeability of CO_2/N_2 separation for PP time of diluents extraction of the membrane

Pressure supplied is actually the factor how to increase the volumetric flowrate of the membrane. When the pressure increase the volumetric flowrate will increase and this will proved that the permeability of the membrane is increase. However, if we compare the volumetric flowrate at the same pressure we can see there are different reading volumetric flowrate from the different time of diluents extraction of the membrane. The permeability is increase when the pore produce inside the membrane is increase. While producing the membrane, many pores can be produced if the air inside the membrane can be removed. Diluents extraction of the membrane is one of the techniques how to remove the air inside the membrane. Methanol can be used as an agent to remove the air inside the membrane by using the extraction diluents technique when immersed the membrane inside the methanol solution. This research study is to identify whether the time of diluents extraction can affect the performance of the pore produced. Table 4.4

shows the result of permeability of CO_2 and N_2 gas for 1 hour, 2 hours, 3 hours and 4 hours of diluents extraction membrane sample**.**

Table 4-4: result of permeability of CO_2 and N_2 gas for 1 hour, 2 hours, 3 hours and 4 hours of diluents extraction membrane sample

Pressure, cmHg	Diluents extraction, hr	Permeability, GPU		Selectivity (α) (co_2/n_2)
		Co_2	N_2	
0.076	1	0.020	0.004	5.00
	2	0.037	0.013	2.85
	3	0.057	0.026	2.19
	4	0.065	0.031	2.10
0.152	1	0.013	0.0009	14.44
	2	0.019	0.005	3.80
	3	0.029	0.014	2.07
	4	0.035	0.027	1.30
0.228	1	0.011	0.0005	22.00
	2	0.015	0.002	7.50
	3	0.021	0.008	2.65
	4	0.024	0.013	1.85

Based on the table some of the permeability data of N_2 gas shows the result approached respecting to CO_2 gas since the size of the pore that produced is not constant per membrane. The bigger pore allowed the N_2 gas to permeate into it. It is difficult to get the membrane with the constant size of the pore produced. There are two factors contribute to the final pore size that is droplet growth rate and duration of the growth period where the growth rate is depends on the viscosity on the system while growth period is depends on the cooling rate (Yave W. et.al., 2006). The cooling rate when the step to produce the membrane gives the different size of the pore produced for different membrane. Any membrane produced have different cooling rate depend on the time we immersed immediately into the cold water. The longer the time taken to immerse into the cold water, the bigger the size of the pore produced. As a result, the performances of the membrane depend on the pore produced will not accurate since the size produced is not constant. Therefore, the cooling rate must be taken as a constant parameter while we want to study about the effect of diluents extraction of membrane into methanol. As usual, the permeability of the both gas increase when the pressure is increase and when the pressure constant we can see that the permeability for the 4 hours time of diluents extraction shows the highest. Compared to the CO_2 gas with N_2 gas for the same

diluents extraction time, CO_2 gas still gives the higher permeability. Figure 4.12 shows the permeability of CO_2 gas against diluents extraction time.

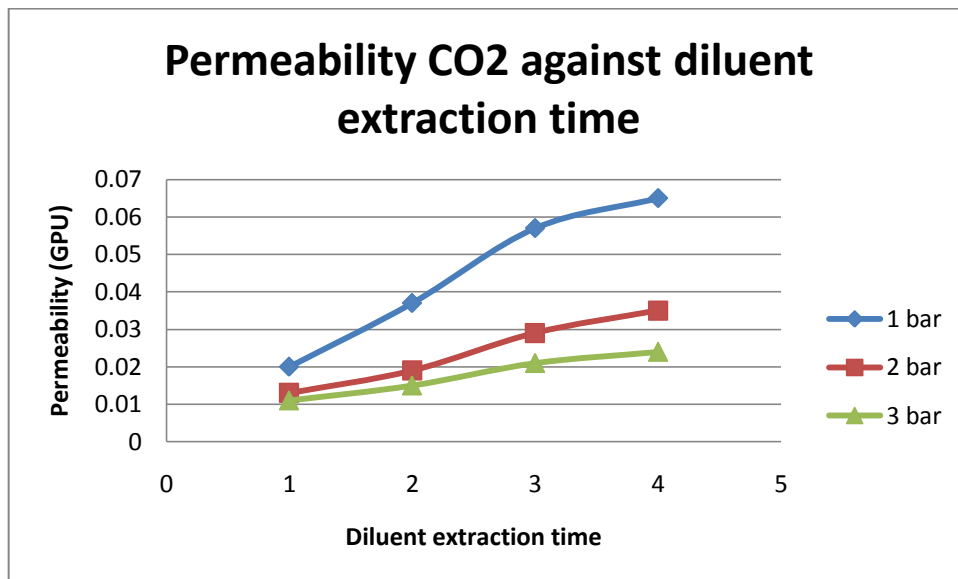**Permeability CO2 against diluent extraction time**

Figure 4-12: the permeability of CO_2 gas against diluents extraction time

From the graph we can see that the permeability of CO_2 gas increase from 1 hour diluents extraction time to 4 hours diluents extraction time. For the pressure of 1 bar, the permeability increase from 0.020, 0.037, 0.057, to 0.065. For the pressure of 2 bars, the permeability increase from 0.013, 0.019, 0.029, to 0.035. Lastly, for pressure of 3 bars, the permeability increase from 0.011, 0.015, 0.021, to 0.024. The pressure of 1 bar gives the highest permeability among 2 bars and 3 bars. Figure 4.13 shows the permeability of N_2 gas against diluents extraction time.
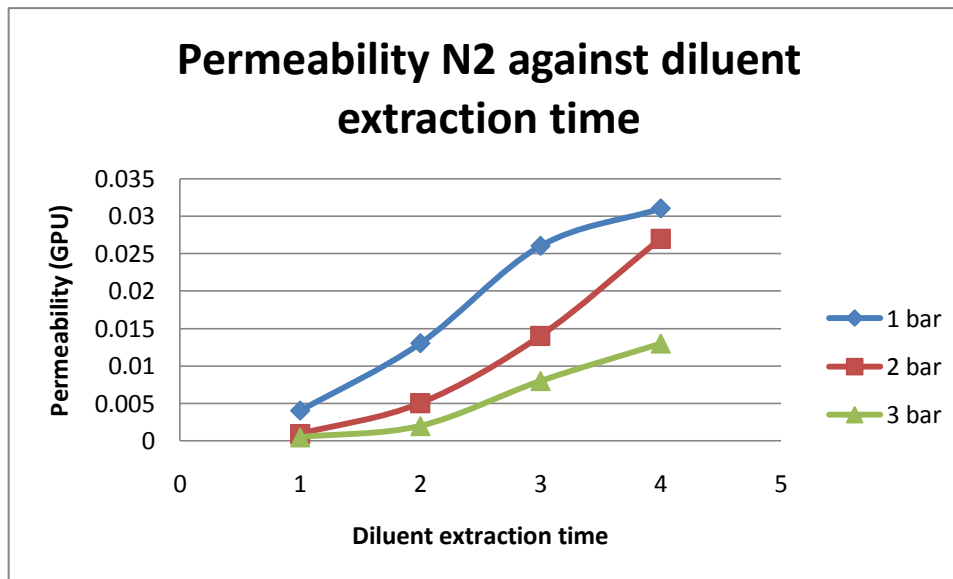
Figure 4-13: the permeability of N_2 gas against diluents extraction time

From the graph we can see that the permeability of N_2 gas increase from 1 hour diluents extraction time to 4 hours diluents extraction time. For the pressure of 1 bar, the permeability increase from 0.004, 0.013, 0.026, to 0.031. For the pressure of 2 bars, the permeability increase from 0.0009, 0.005, 0.014, to 0.027. Lastly, for pressure of 3 bars, the permeability increase from 0.0005, 0.002, 0.008, to 0.013. The pressure of 1 bar gives the highest permeability among 2 bars and 3 bars.

4.3.4 Effect of pressure on selectivity of CO_2/N_2 separation for PP time of diluents extraction of the membrane

The selectivity is the ratio of the permeability of CO_2 gas with N_2 gas. The selectivity is different with the permeability because selectivity is inversely proportional with permeability. Figure 4.14, 4.15, and 4.16 shows the graph of selectivity against pressure for 1 hour, 2 hours, 3 hours and 4 hours of diluents extraction, the graph of selectivity against permeability of CO_2 and the graph of selectivity against permeability of N_2.
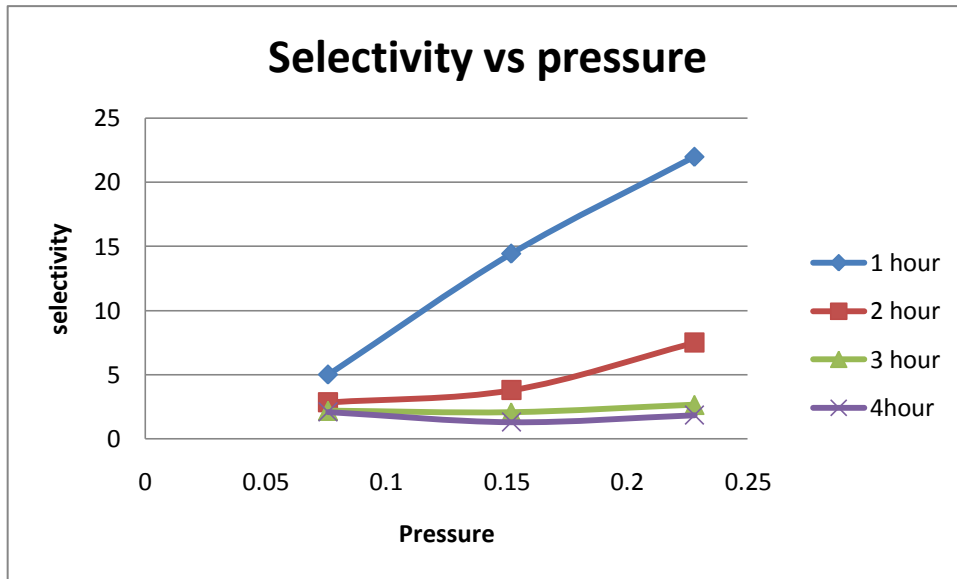
Figure 4-14: the graph of selectivity against pressure for 1 hour, 2 hours, 3 hours and 4 hours of diluents extraction

From the graph we can see that selectivity is directly proportional to pressure supplied. When the pressure increased, the selectivity will increase for all those 4 different time of diluents extraction.
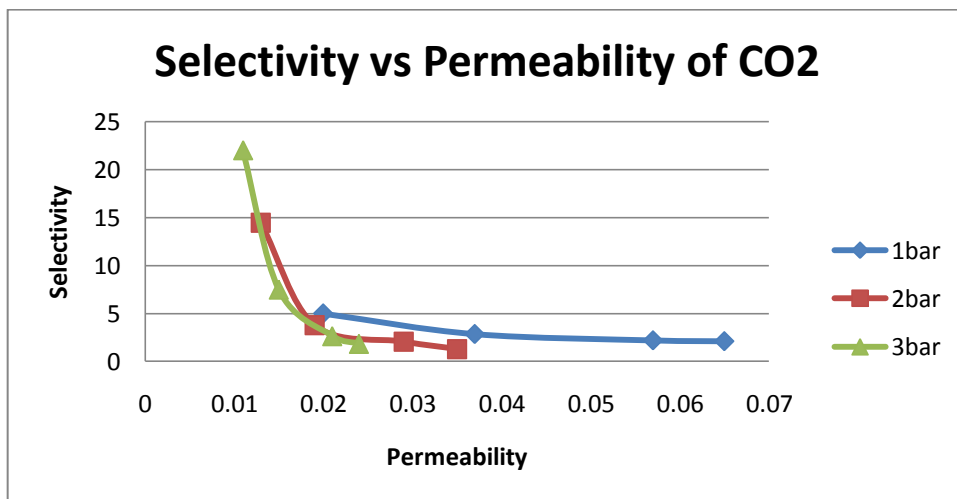

Figure 4-15: the graph of selectivity against permeability of CO_2

From the graph we can see that when the permeability of CO_2 increased then the selectivity will decrease. For the pressure of 1 bar the selectivity will decrease from the 1 hour diluents extraction to 4 hours diluents extraction from 5.00, 2.85, 2.19 and to 2.10. For the pressure of 2 bars the selectivity also decrease from 1 hour diluents

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extraction to 4 hours diluents extraction from 14.44, 3.80, 2.07, to 1.30 and for the pressure of 3 bars the selectivity also decrease from 1 hour diluents extraction to 4 hours diluents extraction from 22.00, 7.50, 2.65, to 1.85.
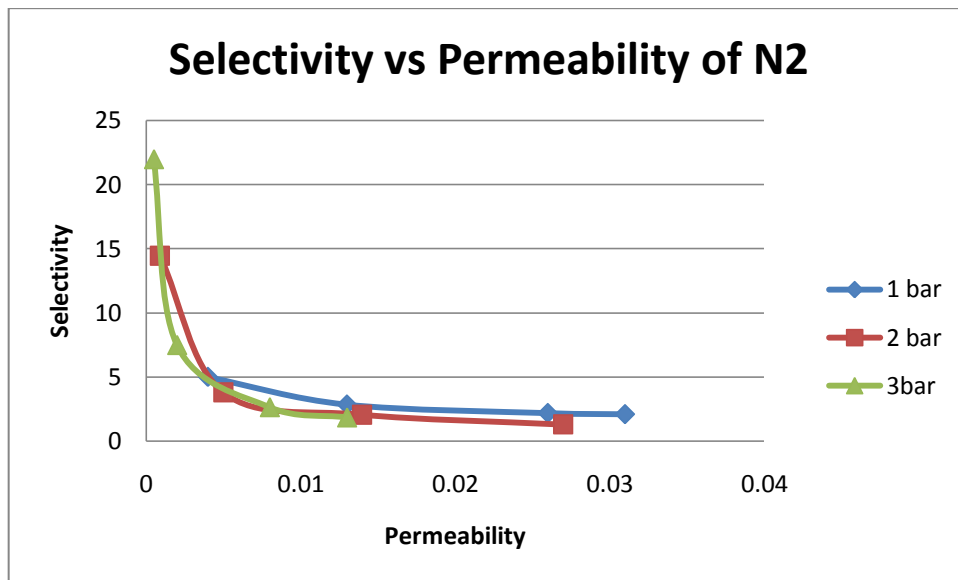
Figure 4-16: the graph of selectivity against permeability of N_2

From the graph we can see that when the permeability of N_2 increased then the selectivity will decrease. For the pressure of 1 bar the selectivity will decrease from the 1 hour diluents extraction to 4 hours diluents extraction from 5.00, 2.85, 2.19 and to 2.10. For the pressure of 2 bars the selectivity also decrease from 1 hour diluents extraction to 4 hours diluents extraction from 14.44, 3.80, 2.07, to 1.30 and for the pressure of 3 bars the selectivity also decrease from 1 hour diluents extraction to 4 hours diluents extraction from 22.00, 7.50, 2.65, to 1.85.

5 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

From this research, the effect of the time of diluents extraction on the PP-DPE flat sheet asymmetric membrane was studied. Based on the analyses for PP-DPE, the way of diluents extraction time affected membrane preparation was investigated in terms of membrane morphology structure, FTIR analysis and gas permeation test. Refer to the SEM analyze, it was found that the diluents extraction for 4 hours time shows the better pore produces since the methanol completely remove the air inside the membrane for a better pore produced compared to others time of diluents extraction. As the air is completely removed, the image also can be seen clearly. However, if too long time takes for the diluents extraction of membrane maybe the methanol will removes along the pore together. Thus, an optimum time of diluents extraction is an important part for the best pore produced. Based on FTIR analysis the diluents extraction time of membrane into methanol does not influence the FTIR test. Related to the gas permeation test, the time of diluents extraction also affect the performance of the membrane. Based on the permeability, the 4 hours of diluents extraction gives the highest permeability compared to others time of diluents extraction. Therefore, the diluents extraction can be one of the parameter consideration to increase the performance of the membrane since the performance of the membrane is depends on the pore production of the membrane itself. However it is difficult to get the higher selectivity when the permeability is higher since they are inversely proportional to each other.

5.2 Recommendations

Based on this research, some recommendation can be used for the future usage:

- Invent the suitable casting equipment for the flat sheet membrane because the steel casting does not give the constant thickness of the membrane produces.
- Time to immerse all the membrane produce into the cold water need to do immediately in constant of time so that the pore size produce will gives the constant size.

- Increase the performance of the membrane by calibrating the digital flow meter to have a better reading of gas flow rate during gas test permeation.
- The diphenyl ether is not suitable solvent to be used in industries since this solvent evaporates at a room temperature. This solvent is odour and will affect the human health while handling it.
- Characterization technique of flat sheet membrane should be expanded by using other test such as Atomic Force Microscopy (AFM) which can measure the details of the membrane characteristic such as pore size, pore distribution of membranes, surface roughness and also the size of macro molar nodules.

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APPENDICES
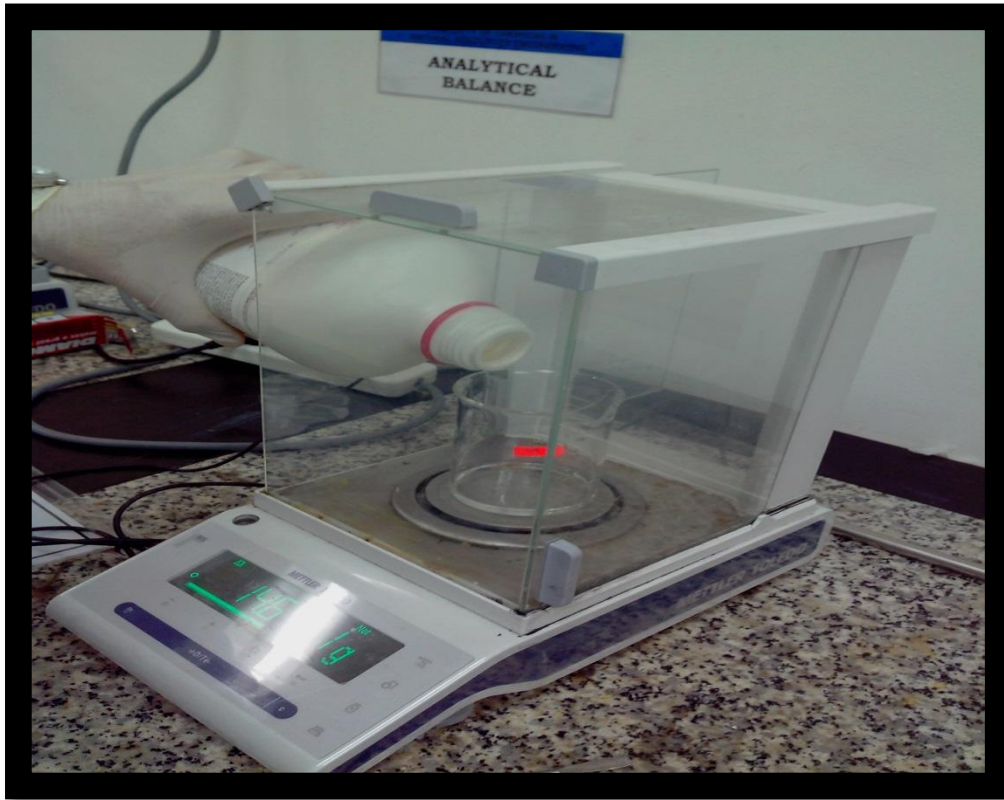
Weighing PP solid using analytical balance
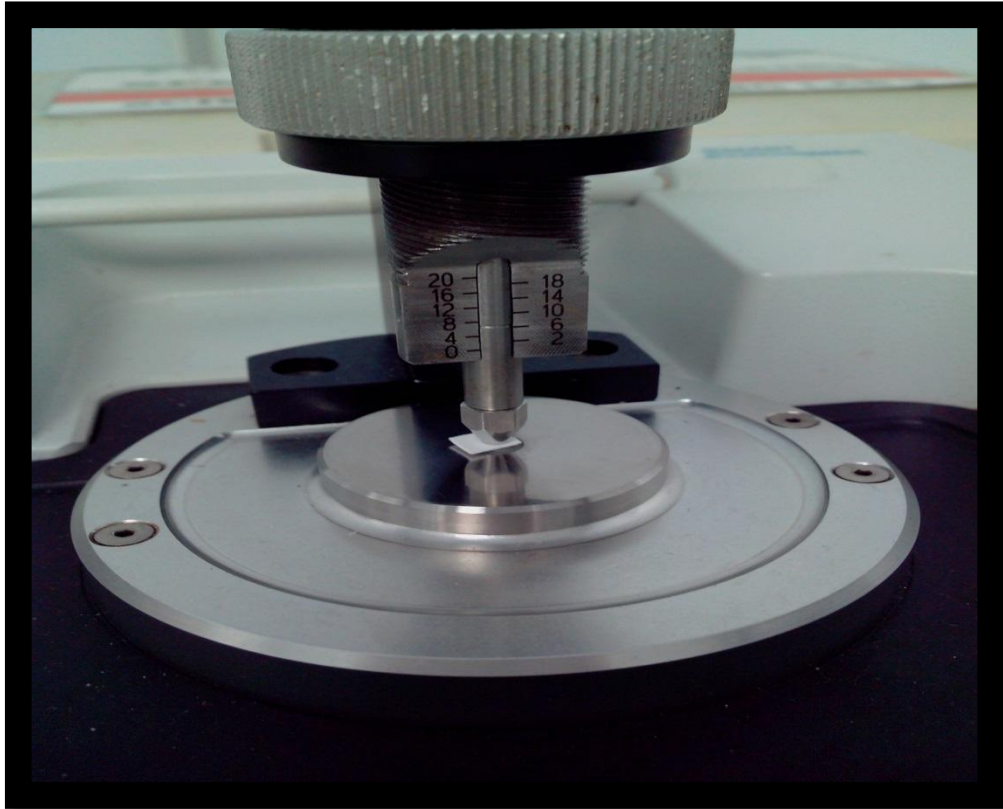
Covered the beaker with aluminium foil

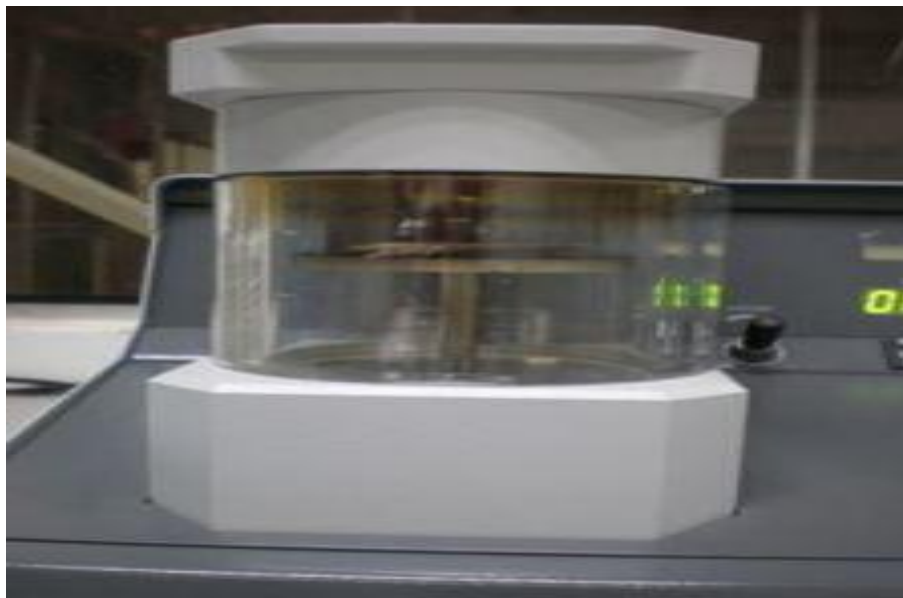Heating of solution using hot plate

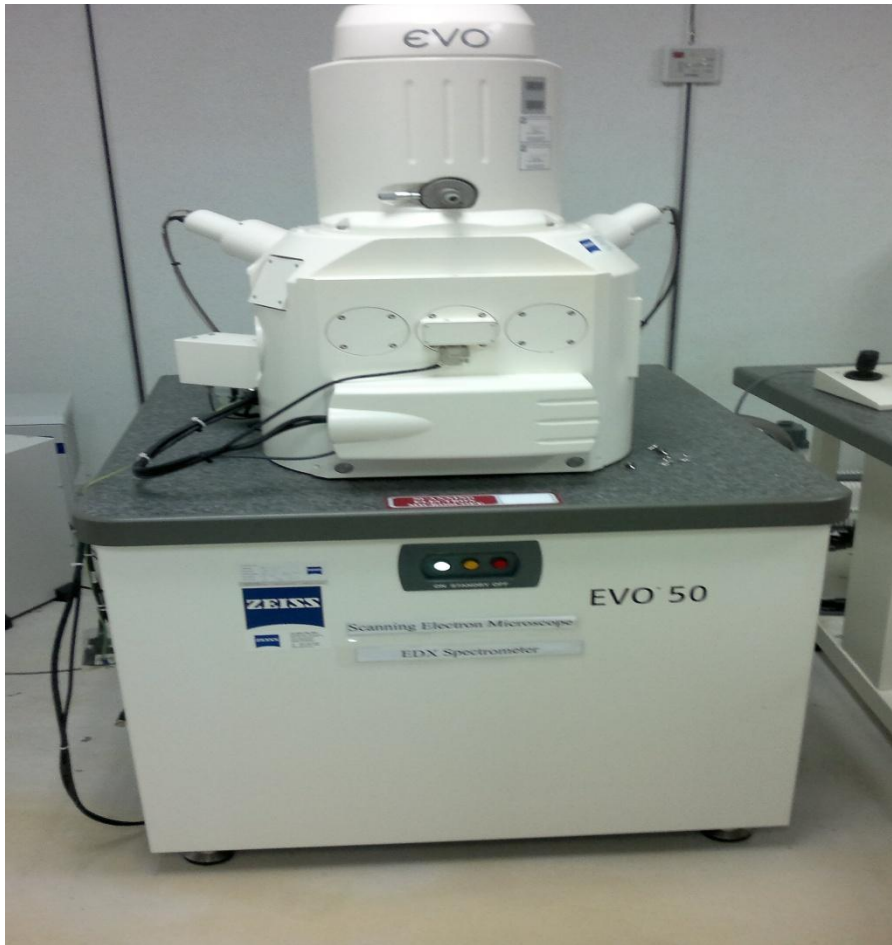Extraction of diluent in methanol

Drying process of membrane

Sample was placed on FTIR plate

Coating of membrane for SEM analyze

Scanning Electron Microscopy