FABRICATION OF NF MEMBRANE USING PHASE INVERSION METHOD FOR DYE REMOVAL

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Report submitted in partial fulfilment of the requirements for the award of the degree of Bachelor of Chemical Engineering

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this project and in my opinion, this project is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering.

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STUDENT'S DECLARATION

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

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Date : 07/02/2012

Special dedication to my father, mother, and my family, who love me,

My supervisor, my beloved friends, my fellow colleague,

and all faculty members.

For all your love, care, support and believe in me

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"In the name of God, the most gracious, the most compassionate"

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ABSTRACT

Nowadays membranes play an important role as a separation tool in many industrial processes such as in pharmaceutical and biotechnological industries, in pure water production and in water and waste water treatment. Rejection of dye concentration one of the factors that can reduce the performance of membrane as well as flux decline and indirectly will affect the quality of the water produced and consequently increases in cost and energy replacement. The rejection can define as the percentage of solids concentration removed from system feed water. Therefore this research was conducted to produce high performance of NF membrane by manipulating the concentration of PES in order to produce the high rejection of colour removal. The manipulated variable is the percentage concentration of PES, meanwhile the dependent variable is percentage PVP and also NMP. In this study, the membranes were synthesized by using wet phase inversion method. In this method, the cast polymer solution is immersed in water bath and absorption of water will cause the film to rapidly precipitate from the top surface of membrane. Based from the 3 membrane that studied which is 18% PES membrane, 20% PES membrane, 23% PES membrane, 23% PES membrane gives greatest rejection 64.64% at 3 bar pressures applied. In the meantime, the value of flux at high percentage concentration of PES decrease 16.0162 L/m^2.h compare with low percentage PES concentration 871.0801 L/m^2.h. This is due to the thickness and tightens the porosity at high percentage concentration of PES. Increased feed water pressure also results in increased the dye rejection.

ABSTRAK

Kini membran memainkan peranan penting sebagai alat pemisahan dalam proses industri seperti industri farmaseutikal dan bioteknologi, dalam pengeluaran air tulen dan air dan rawatan air sisa. Penolakan kepekatan warna salah satu factor-faktor yang boleh mengurangkan prestasi membran serta penurunan flux dan secara tidak langsung akan menjejaskan kualiti air yang dihasilkan dan seterusnya meningkatkan kos dan penggantian tenaga. Penolakan itu boleh ditakrifkan sebagai peratusan kepekatan pepejal yang dikeluarkan dari sistem air aliran masuk. Oleh itu, kajian ini dijalankan untuk menghasilkan prestasi yang tinggi membran NF dengan memanipulasi kepekatan PES untuk menghasilakn penolakan penyingkiran warna yang tinggi. Pemboleh ubah yang dimanipulasikan adalah kepekatan PES untuk menghasilkan penolakan penyingkiran warna yang tinggi. Pemboleh ubah bersandar pula adalah peratusan PVP dan NMP. Dalam kajian ini, larutan cast polimer direndam di dalam air rendaman dan penyerapan air akan menyebabkan lapisan nipis (filem) dengan pantas termendak dari permukaan atas membran. Berdasarkan dari tiga membrane yang dikaji yang membrane PES 18%, 20% membrane PES, 23 % membrane PES, 23% membrane PES memberikan penolakan terbesar 64.64% pada tekanan 3 bar yang digunakan. Pada masa yang sama, nilai flux pada peratus kepekatan PES yang paling tiggi berkurang 16.0162 $L/m^2.h$ berbanding dengan peratusan rendah peratusan kepekatan 871.0801 $L/m^2.h$. Ini adalah kerana ketebalan dan ketetatan liang pada kepekatan peratusan tinggi PES. Tekanan aliran air masuk yang meningkat juga menyebabkan peningkatan penolakan warna yang tiggi.

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LIST OF ABBREVIATION

MB	Methylene blue
NF	Nanofiltration
NMP	1-methyl-2-pyrrolidone
PES	Polyetehersulfone
PVP	Polyvinylpyrrolidone
RO	Reverse osmosis
UF	Ultrafiltration

CHAPTER 1

INTRODUCTION

This chapter will introduce about the topic of research method and also goal of this study. Besides that, this chapter also will describe about process of clarifying the scopes of the study and identifying the problem statement. It also will cover about the significant of this study to the environment and to the human being as well.

1.1 Research background

In chemical process industry, separation processes are crucial as chemical reaction and high purity water is necessary. Azhar and Liew (2005) observed that recently it was guesstimate that over 7 $x10^5$ tonnes of 10 000 commercial dyes and pigments exist and produces annually world wide. These dyes are hardly fading on exposure water, light and many chemical due to their complex chemical structure and synthetic origin. The industry such as printing, textile, paper, plastic, cosmetic and other else are needed to recovery the dyes for their manufacturing and treatment process. Azhar and Liew (2005) also confirmed that textile industry is the first rank in usage of dyes for coloration of fibre among these industries.

This dyes effluent should be treated as it may exert great impact to our mother earth as well as to our health. In order to reduce or prevent the effluent dyes, many researches have been conducted to find the most effective and economical ways to treat this effluent. There are several methods of dyes removal from industrial effluent which is can be characterized into two parts which is physical and chemical method. Gonder et. al., (2010) have reported that among the advanced treatment processes, membrane technology offers an attractive alternative method to treat dye effluent for that purposed as well as its consume less energy, low space requirement and also simplicity of operation (Celik et al.,2010).

Nowadays, more and more field is planning to use membrane technology to separate the fluid and get high quality result. The application of membrane separation process are expending much larger with the principal of the membrane technology as it is also bring significant economy benefits. Recently many countries in the world have noticed the crucial of membrane technology especially deficient in resources, short of energy and also declining environment is all in existence in our life. Thus, the industries and the technology regard the membrane separation technology is very important in our daily life.

Membrane act as a semi permeable barrier and separation occur by the membrane controlling the rate of movement of the various molecules between two liquid phases. There basically have three type of membrane which is ultrafiltration (UF) membrane, reverse osmosis (RO) membrane and also nanofiltration (NF) membrane. Petrinic et. al., (2007) studies' show that, the UF membrane could not completely decolourise wastewater as it did not remove low molecular weight dyes.

Meanwhile to assure the decolourised and desalinated of wastewater the combination of RO and NF is required. This is due to the behaviour of RO itself can desalinate (NaCl retention) wastewater effectively up to 93% beside produce a colourless permeate. But this research focusing more on decolourised of divalent ion and dye molecules only. Petrinic et. al., (2007) also observed that the NF show it can remove up to 99% of dyes and 84% electrolyte even sodium chloride can pass trough it. So, that direct NF of dye water is the most realistic method for the dye removal treatment.

1.2 Problem statement

The most important goals in membrane technology are to control the membrane structure and thus the membrane flux and rejection. Even NF show that the dye can remove up to 99%, but not all type of dye can exactly remove until 99%. Therefore this research was conducted to know how much the dye of MB will reject via phase inversion method by manipulation of PES concentration

1.3 Objective

The main objective of this research is to produce high performance of NF membrane by using phase inversion method and also to produce high rejection of the NF membrane by manipulating the concentration of PVP and PES.

1.4 Scope of study

In order to meet the objective, there are some scopes that need to be focused:

i. To fabricate NF membrane by using phase inversion method

ii. To study the effect of PES concentration on NF performance (flux, dye removal and percentage rejection)

1.5 Significance of study

There have so many significance of this research in term of knowledge generation and positive social change that would be beneficial not only to the human community and culture but also to the environment. Firstly, the priority doing this research is to remove the colour from the effluent of textile industry since Azhar and Liew (2005) have reported that textile industry is the first rank in usage of dyes for coloration of fibre among these industries. Because the huge consumption of dye in this industry, dye removal is so important in order to be friendly to our mother nature.

Beside, Gonder et. al., (2010) have stated that the high consumption of fresh water is the most crucial environmental concern in the industry. Thus, in order to reduce the consumption of freshwater and lower the wastewater treatment plant capacity, there is a need to recycle the treated wastewater. The cost of treatment this water effluent also reduced since the membrane filtration are the more cost effectively compare to others (Ali et. al., 2009)

Other than that, the significant of this study is to follow the standard quality of Environmental Quality Act 1974 [Act 127]. In this Act 127, there have stated the limit of dye of effluent can be discharge for Standard A is 100 ADMI (American Dye Manufacturer Institute) and Standard B is 200 ADMI. For the Standard A, the catchment areas referred to the areas upstream or surface above subsurface including water intakes which are the water for the human consumption including drinking. Meanwhile for the Standard B is for the any other inland waters not including human consumption.

CHAPTER 2

LITERATURE REVIEW

This chapter will point out about the definition and the basic concept of membrane separation technology, membrane structure that have been used nowadays and also the membrane module in separation industry.

2.1 Membrane definition

Membrane can be defined basically as thin layer of semi-permeable barrier, which separate two phases and restrict transport of various chemical when a driving force is applied across the membrane (G. Srikath, 2011). It can control the rate of movement of various chemical between two liquid phases, two gas phase, or a gas and liquid phases. The chemical component that allowed passage by the membrane into the permeate stream is called permeate, whereas the others that retain and accumulate is known as retentate. The two fluid phases are usually miscible and the membrane barrier prevents actual, ordinary hydrodynamic flow. A membrane can be homogeneous or heterogeneous, symmetric or asymmetric in structure, solid or liquid. This membrane process are increasingly used nowadays for removal of bacteria, microorganism, particulates, and natural organic material, which can impart colour, tastes, and odours to water and react with disinfectants to form disinfection by product.

2.2 Principle of membrane based separation process

There are two ways for membrane separation process which is dead end filtration and cross flow filtration as shown in Figure 2.1.

2.2.1 Dead End Filtration

In the dead end filtration, the pressure will apply to the feed solution as a force through the membrane. The surface of the filtration membrane is vertical with the feed flow direction. Basically the filtrate direction which passes the membrane is the same direction with the feed flow. The particles that retained in the feed solution will adhere to the surface of membrane which causes clogged and the consequently cartridge filters have to be replacing frequently and can not reused to maintain the performance of membrane.

2.2.2 Tangential (Cross) Flow Filtration

In the cross flow filtration, the fluid to be separated is pumped across the membrane parallel to its surface. Clear permeate and retentate solution that containing most of the retain particle in the solution will produce from the cross flow. The retained particle can be swept off the membrane surface by maintaining a high velocity. This will make the cross flow filtration is more efficient in operation compare to dead end filtration that easily built up the filter cake

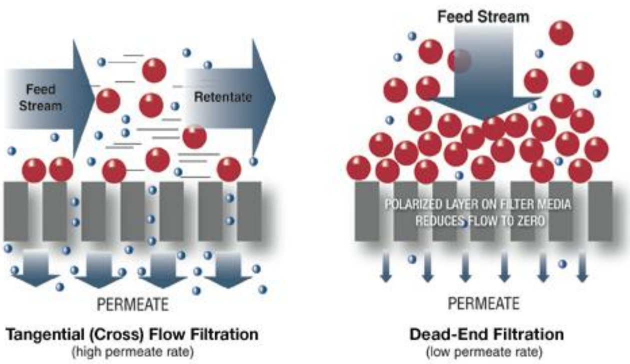

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Figure 2.1: Membrane separation process (Schmeling et.al, 2010)

2.3 Membrane structure

The proper choice of membrane should be determined by their specific application. There are two type of membrane structure that commonly used which is asymmetric and isotropic membrane. The different between these two structures are the physical and chemical properties.

2.3.1 Asymmetric Membranes

Asymmetric or as known as anisotropic are non-uniform over the membrane cross section and they consist of a number of layers each with different structure permeability and chemical composition. The skin layer is very thin (0.1- 1.0 micron) and highly porous (100-200 microns) thick substructure (G. Srikath, 2011). Chakrabarty et.al, (2008) stated this membrane are characterized by the existence of a dense top layer and a porous sublayer. Because the nature of this membrane itself have a thin top layer that acts as a selective barrier film, and a porous sublayer that offer good mechanical strange makes this membrane have been widely used for gas and liquid separation process.

2.3.2 Isotropic membrane

2.3.2.1 Nonporous membrane.

The transmembrane of dense nonporous isotropic membrane fluxes through this membrane relatively thick make it to low for practical separation process and rarely used in membrane separation process. On the other hand, this nonporous isotropic membrane is commonly used in laboratory work to characterize the membrane properties.

2.3.2.2 Microporous membrane.

This isotropic microporous membrane almost behave like fibre filter and separate by sieving mechanism determined by the pore diameter and particle size. The pores in the membrane may vary between 1nm- 20 micron (G. Srikath, 2011). By comparing with the isotropic dense membrane, the isotropic microporous membranes have higher fluxes and more widely used as microfiltration membrane. Besides, it is also used as a inert spacers in a battery and fuel cell applications and as the rate controlling element in controlled drug delivery device.

2.4 Type of membrane separation process

There are various types of membrane separation that have been developed for specific application. Each of those have different characteristic and some are widely used in industry. This membrane separation process to be considered here is a membrane liquid process such as reverse osmosis, ultrafiltration, microfiltration and nanofiltration. The difference between these four membranes has shown in Table 2.1 below.

Table 2.1: Comparing Four Membrane Process (Wagner. J, 2001)

	Reverse Osmosis	Nanofiltration	Ultrafiltration	Micro filtration
Membrane	Asymmetrical	Asymmetrical	Asymmetrical	Symmetrical Asymmetrical
Thickness Thin film	150 μm 1 μm	150 μm 1 μm	150 - 250 μm 1 μm	10-150 μm
Pore size	<0.002 μm	<0.002 μm	0.2 - 0.02 μm	4 - 0.02 μm
Rejection of	HMWC, LMWC sodium chloride glucose amino acids	HMWC mono-, di- and oligosaccharides polyvalent neg. ions,	Macro molecules, proteins, polysaccharides vira	Particles, clay bacteria
Membrane material(s)	CA Thin film	CA Thin film	Ceramic PSO, PVDF, CA Thin film	Ceramic PP, PSO, PVDF
Membrane Module	Tubular, spiral wound, plate-and-frame	Tubular, spiral wound, plate-and-frame	Tubular, hollow fiber, spiral wound, plate-and-frame	Tubular, hollow fiber
Operating pressure	15-150 bar	5-35 bar	1-10 bar	<2 bar

2.4.1 Reverse osmosis membrane

Reverse osmosis (RO) is the tightest possible membrane process in liquid-liquid separation. The process RO membrane just not only can remove some suspended solid but also it does eliminate bacteria, viruses and other germ that contain in water (G. Srikath, 2011). RO is essentially a pressure driven membrane diffusion process for separating dissolve solute. According to Geankoplis (2003) the important commercial used of RO is in the desalination of seawater or brackish water because of the effectiveness and characteristic of RO itself. Reverse osmosis membranes have the smallest pore structure, with pore diameter ranging from approximately 5-15 A^{o} (0.5 nm - 1.5 nm). The operating pressures in RO are generally between 10 and 100 bar (J. Timer, 2001). Extremely small size of RO pores allows only the smallest organic molecules and unchanged solutes to pass through the semi-permeable membrane along with the water.

2.4.2 Ultrafiltration membrane

UF is a membrane process that is quite comparable to reverse osmosis. It is a pressure driven process where the small solute molecule pass thought the membrane and are collected as a permeated. Basically, the solute or molecules to be separate have a higher molecular weight which is greater than 500 and up to 1000 000 or more such as polymers, starch and etc (Geankoplis, 2003). The UF membrane has small pore diameters size, between 10Å to 2000Å.

2.4.3 Microfiltration membrane

The separation of micron and submicron level can be efficiently be operate by using microfiltration membrane filter. The pore sizes of microfiltration membranes are usually larger than RO, UF and NF. Microfiltration membrane are used to filter the size particle that have range from 0.02 µm to 10 µm such as suspended particulate, bacteria or large colloids from solutions. This membrane usually used the pressure from 100kPa to 500kPa.

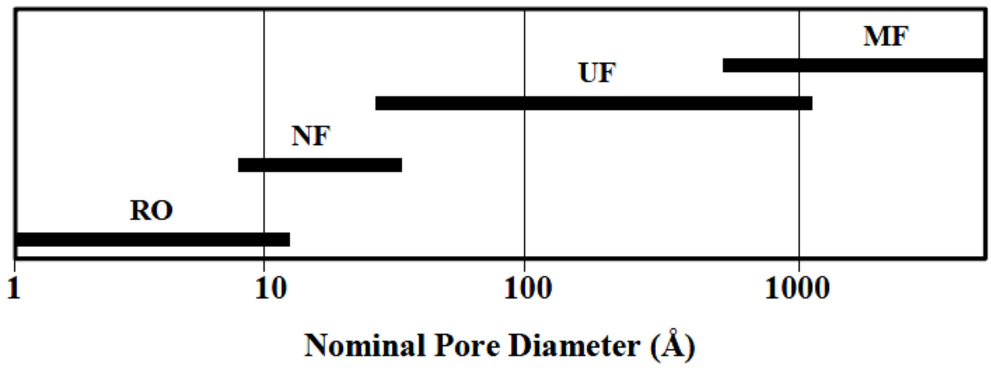

Figure 2.2: Range of membrane nominal pore size (Sagle, A & Freeman, B)

2.4.4 Nanofiltration membrane

Nanofiltration membrane is the most recent membrane that is very thin and has a small pore size which is between 10nm to 200nm. The pressure that used in nanofiltration process is from 0.3 MPa to 3 MPa. This membrane is known as different from others because it consist charge and can reject ion with more than one negative charge, such as sulphate or phosphate, while passing single charge ion. This charge is basically used to retain selective molecule to avoid fouling (Cheng et.al, 2010). Wagner. J (2001) comment that NF also rejects uncharged, dissolved materials and positively charged ions according to the size and shape of molecule in the solution and also feed concentration.

2.5 Membrane module

A membrane module is a pack of the membrane area into the least volume, to decline the capital and operating cost with providing acceptable flow hydrodynamics in the vessel. The practicability of the membrane separation process usually depends on the module configuration as the active separation membrane area can affect the membrane module configuration. There are four type of membrane which is tubular, spiral wound, plate and frame and hollow fibres. The comparison between these four membranes has been discussed in Table 2.2.

2.5.1 Tubular

The tubular module are now generally restricted to ultra-filtration, for which the benefit of resistance to membrane fouling outweighs the high cost. These modules enclose as many as 5 to 7 smaller tubes, each 0.5 to 1.0 cm in diameter (Cheah S M., 2000). The membrane is often on the inside of a tube and the feed solution is pumped through the tube and permeate is removed from each tube from each tube and sent to permeate collection header.

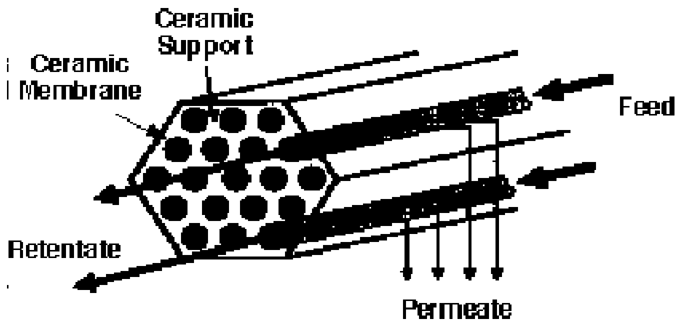

Figure 2.3:A schematic drawing of a tubular membrane module(Sagle,A& Freeman, B)

2.5.2 Spiral wound

The spiral wound module very popular in industry for nanofiltration or reverse osmosis membrane. This module has a flat sheet membrane wrapped around a perforated permeate collection tube. The feed flows on one side of the membrane and permeate is collected on the other side of the membrane meanwhile spirals in towards the centre collection tube. The low price and very compact design of spiral wound module was originally made exclusively for water desalination caught the attention to other industry (Wagner.J, 2001). But nowadays after redesign have made, this module can be used for a variety of industrial application such as in the dairy industry, the pulp and paper industry and other else.

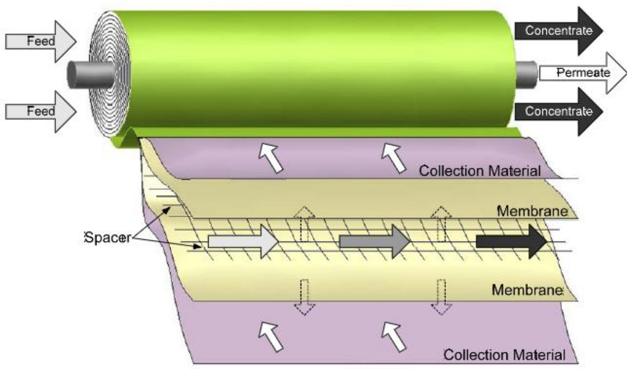

Figure 2.4: A schematic drawing of a spiral wound membrane module (Li et al., 2006).

2.5.3 Plate and Frame

Plat and frame membrane module were one of the earliest type of membrane modules and were widely used in separation process. But, because of their relatively high cost they have replace in most application by spiral wound modules and also hollow fiber modules. Nowadays, the plate frame module used only in electrodialysis and pervaporation system in a limited number of reverse osmosis and ultrafiltration applications with highly fouling condition (Cheah S M., 2000).

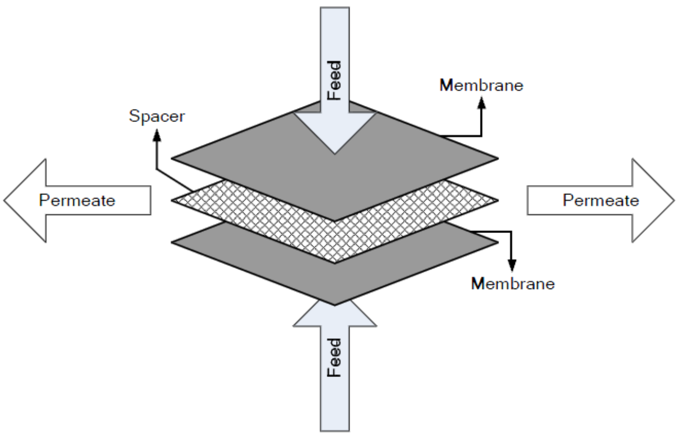

Figure 2.5: A schematic drawing of a plate and frame membrane module (Li et al., 2011).

2.5.4 Hollow Fibres

The hollow fiber module also has been widely used for desalination that usually consists of bundle of hollow fibers in a pressure vessel. The figure 2.6 below depicted a schematic drawing two kind of a tubular membrane module based on different operation condition. This module has been characterized in 4-8 inch (10-20 cm) in diameter and 3-5 feet (1.0-1.6 m) long (Cheah S M., 2000). The system of hollow fiber module will pressurised from the shell side, and the filtrate passes pass along the fibre wall and exits through the open fibre ends. Bore-side of hollow fiber modules can also be used where the feed is circulated through the fiber. The most advantages of hollow fiber modules are the ability to pack a very large membrane to single module. For instance, in an 8-inch diameter, 40-inch long spiral-wound module would contain about 20 - 40 m^2 of membrane area. The correspondent hollow-fibre module filled with fibres of 100-mm diameter will contain approximately 600 m^2 of membrane area.

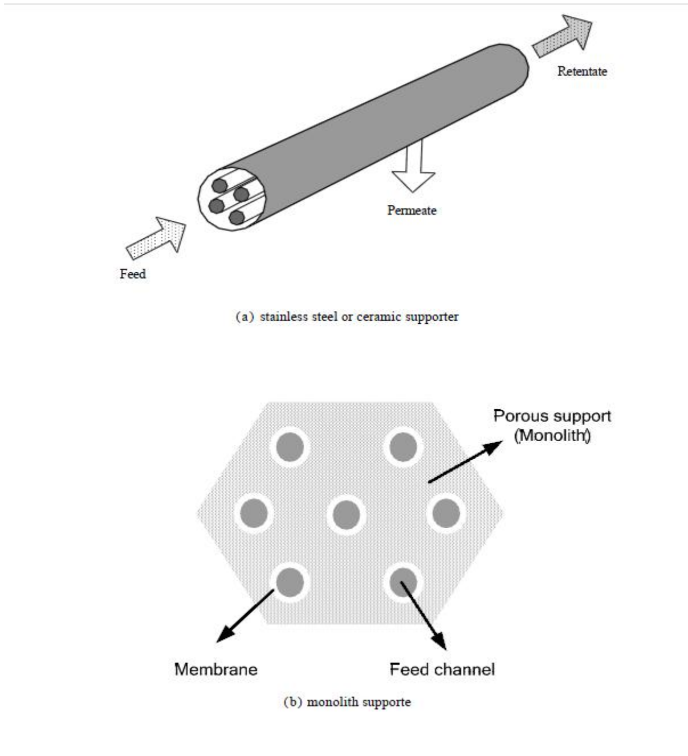

Figure 2.6: A schematic drawing of a tubular membrane module (Li et al., 2011).

Table 2.2: Comparison between several membrane modules (Wagner. J, 2001).

	Spiral wound element	Tubular high price	Tubular low price	Plate and frame system	Hollow wide fiber system	Hollow fine fiber	Ceramic
Membrane density [m2/m3]	high	low		average	average	very high	low
Plant investment	low	high	low	high	very high	medium	very high
Tendency to fouling	average	low		average	low	very high	medium
Cleanability	good	good		good	low	none	good
Variable costs	low	high	low	average	average	low	high
Change of membrane only, see note 1	no	yes	no	yes	no	no	yes
Flow demand	medium	high	medium	medium	high	low	very high
Prefilter other demands (see also table 26)	≤ 50 µm. no fibers	sieve		≤ 100 µm. few fibers	≤ 100 µm. few fibers	≤ 5 µm extreme pretreatment	sieve

CHAPTER 3

CHEMICALS, EQUIPMENTS AND METHODOLOGY OF RESEARCH

In this chapter, the characteristic of chemical that used and the detail procedure in this study have been described that in order to make sure the experiment can be performed smoothly so this would achieve the objective of this study.

3.1 CHEMICAL

3.1.1 Polyethersulfone

Polyethersulfone (PES) has many advantages compare to other polymers. PES has high thermoplastic performance, excellent thermal stability and lower hydrophobility. The PES also can also withstand with the wide range of pH which is 0-14 (Gonder et al., 2010).PES was purchased from Merck Sdn Bhd Malaysia.

Figure 3.1: Polymer PES

3.1.2 1-methyl-2-pyrrolidone

The solvent chosen in this process is 1-methyl-2-pyrrolidone (NMP) provided by company Merck Sdn Bhd Malaysia because of its strong interaction with polymer and miscibility with water (Ahmad et al, 2004). It is also high solvency and low volatility (Ali et al., 2009). The typical pH value of NMP is 8.5 – 10.0, the boiling point is 202^{o}C and the melting point is -24^{0}C.

3.1.3 Polyvinylpyrrolidone

Polyvinylpyrrolidone (PVP) obtained from company Merck Sdn Bhd Malaysia is a hydrophilic polymer (Qin et al., 2002) that acts as an additive to a polymer to reduce hydrophobicity and increase hydrophilicity. Beside that, it also increases the quantity of pores in membranes. This monomer is carcinogenic and extremely toxic to aquatic life. Therefore, must extremely care while handling this chemical.

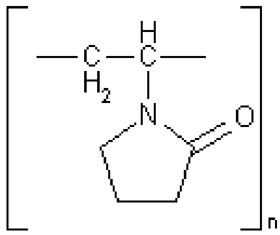

Figure 3.2: Chemical structure for PVP (retrieved: www.chemistrydaily.com)

3.1.4 Methylene blue

The dye used as a model for colour removal in this study is methylene blue (MB). MB was purchased from Merck Sdn Bhd Malaysia. It has many applications in a range of different fields such as biology or chemistry. The form of MB in room temperature it appears as a solid, odourless, dark green solid and it is really harmful to human body. The methyl/methylene group of methylene blue is a hydrophobic (Oliva et.al., 1995)

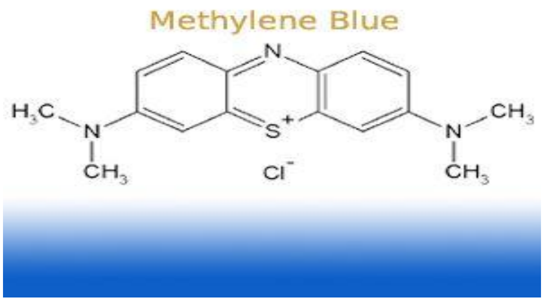

Figure 3.3: Chemical structure for MB (retrieve: www.nilesbio.com)

3.1.5 Glycerol

During the storage process the membrane was kept in a glycerol solution (Merck Sdn Bhd Malaysia) to prevent the pore membrane to be shrinking. Besides, that also works as a humidifier and plasticizer to maintain the quality of membrane.

3.1.6 Sodium metabisulfite

Sodium metabisulfate as known as sodium pyrosulfate or disodium is an inorganic compound was obtained from Merck Sdn Bhd Malaysia. The chemical properties of this compound make it is very useful for a variety of industrial purposed especially as a bacteriostatic agent. The purposed of adding sodium metabisulfate during the storages process is to prevent the bacteria growth. These compounds not exactly kill the bacteria but simply prevent from replicating. Extremely care should been taken during handle this compound is really harmful.

3.2 EQUIPMENT

3.2.1 Hot plate with magnetic stirrer

The hot plate as shown below with magnetic stirrer is to help the solution of PES and PVP with the solvent NMP well mixed. Besides that, this equipment also makes this solution more miscible when the heat is supply.

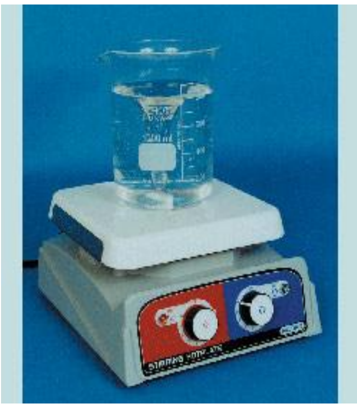

Figure 3.4: hot plate (source:www.favorit-sci.com)

3.2.2 Water bath

Water bath is used in the final stage of casting process to allow NMP solvent in membrane matrix diffused in water medium. The PES membranes that have been prepared before will be immersed into the water bath.

Figure 3.5: Water bath

3.2.3 Stirred cell Amicon

Stirred cell Amicon model 8200 was used to study the performance of the fabricated membrane in term of flux, permeability and colour removal.

Figure 3.6: Stirred cell Amicon (Source: www.fisher.co.uk)

3.2.4 Glass rod and a flat glass plate

To fabricate the PES membrane, a glass rod and a flat glass plate are used as a casting knife. A cellophane tape is coated at the end of a both side of the rod until the thickness of the tape is around 0.05 cm. This is to ensure the membrane is flat and thin.

3.2.5 Pressure supply

Nitrogen gas was used as a pressure supply to make sure the flow of the solution through the membrane. The applied pressures are between $100kNm^{-2}$ until $500kNm^{-2}$.

3.3 METHODOLOGY

In this methodology part the membrane fabrication procedure and the testing process of membrane were described in detail. Basically there are five processes that have been carried out in this research which are mixing process, casting process, bath process, storage process and testing process. The Figure 3.6 shows the flow of this experiment.

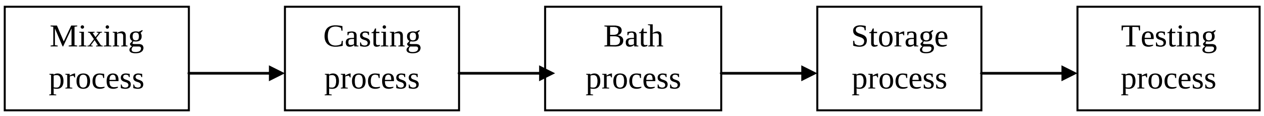

Figure 3.7: Process in fabricating PES membrane

3.3.1 Membrane preparation

PES (18%wt) in a powder form and PVP (2%wt) were dissolved into NMP as a solvent. Before that, the PES and PVP were mixed well in the different beaker before dissolved into NMP. Then, the solution was infused carefully into NMP and was covered with the aluminium foil and then was stirred on the hot plate with magnetic stirred until it mixed well. A yellowish dope solution with a lot of bubble inside like honey will form and this solution was left a few hours in room temperature until no bubble inside the solution. After that the solutions was poured onto a glass plate and flatten it with the glass rod. Then, the glass plate with the liquid film (membrane) was immersed into the water bath. The colour of PES membrane will transform from transparent to white because the precipitation of PES. After a few minutes the liquid film automatically takes off from the glass plate. Carefully, take the membrane and wash it several times with the distillation water to remove the excess solvent. At the final stage, the membrane were stored in a water bath that consist of distillation water, glycerol (20%wt) and sodium metabisulfite (1%wt) to avoid shrinking of pores and bacteria growth in room temperature. The step was repeated using different percentage concentration of PES and PVP. The PES should be kept in a refrigerator when not used.

Figure 3.8: Mixing process

Figure 3.9: Casting process

Figure 3.10: Immersion in water bath process

3.3.2 Process of solution preparation

Before starting the experiment, the polymer dope solutions were prepared correctly as indicated in Table 4.1. The NF membrane was prepared by phase inversion by using PVP as an additive, NMP as a solvent and PES as a polymer.

Table 3.1: Percentage and mass of chemical to fabricate the membrane

chemical	18% PES membrane		20% PES membrane		23% PES membrane	
	Mass (g)	%	Mass (g)	%	Mass (g)	%
PES	11.61	18	13.23	20	15.82	23
PVP	1.29	2	1.32	2	1.38	2
NMP	51.60	80	51.60	78	51.60	75

3.3.3 Membrane Performance Test

For the membrane performance test, firstly the PES membrane was cut in circular shape with the same size of Amicon stirred cell. The time taken for the dye penetrates the membrane will record. This process will be repeated by using other membrane with different concentration of PVP and PES. The data time taken for the dye to penetrate the membrane and the percentage of concentration of PES and PVP will be tabulate. The flux will be calculated by using the formula. The graph of flux versus pressure will be plot.

Figure 3.11: Testing process

CHAPTER 4

RESULT AND DISCUSSSION

In this chapter the performance of membrane fabricated from three different concentration of PES have been investigated and discussed. This including membrane flux, permeability and colour removal using MB as a model solution.

4.1 Flux and Permeability of membrane.

Performance of membranes was measured by calculating its flux and permeability of distilled water. Flux for difference percentage of PES membrane is determined by testing each membrane using Stirred Cell Amicon. The percentage PES used in this study was 18 % wt, 20%wt and also 23%wt. The volume of distilled water was fixed at 20ml and the effective area of each membrane was 28.7 cm^2. The operating pressureregulated using Nitrogen gas was varied from 1 bar to 4 bars. The time taken for each membrane to collect permeate of 20ml was recorded as shown in Appendix C.

Flux can be defined as solvent flow per unit area .Flux of difference percentage of PES membrane were shown in Table 4.1. The equation of flux is given by:

$$J_w = \frac{V}{(Ax\,\Delta t)}$$

Permeability can be described as the ability of water to flow through a membrane per unit time at certain pressure and area. The membrane permeance, *Pm* can be determined from the slope obtained by plotting the permeate flux *Jw* against ΔP. The equation of permeance as shown below:

$$Pm = \frac{Jw}{(\Delta P)}$$

Table 4.1: Flux for difference percentage of PES

Pressure (bar)	Flux (L/ m^2.h)		
	18% PES membrane	20% PES membrane	23% PES membrane
1	327.166	12.3887	6.7323
2	467.6940	20.0191	9.9170
3	882.1065	39.7527	11.2072
4	1222.5687	103.8546	32.8554

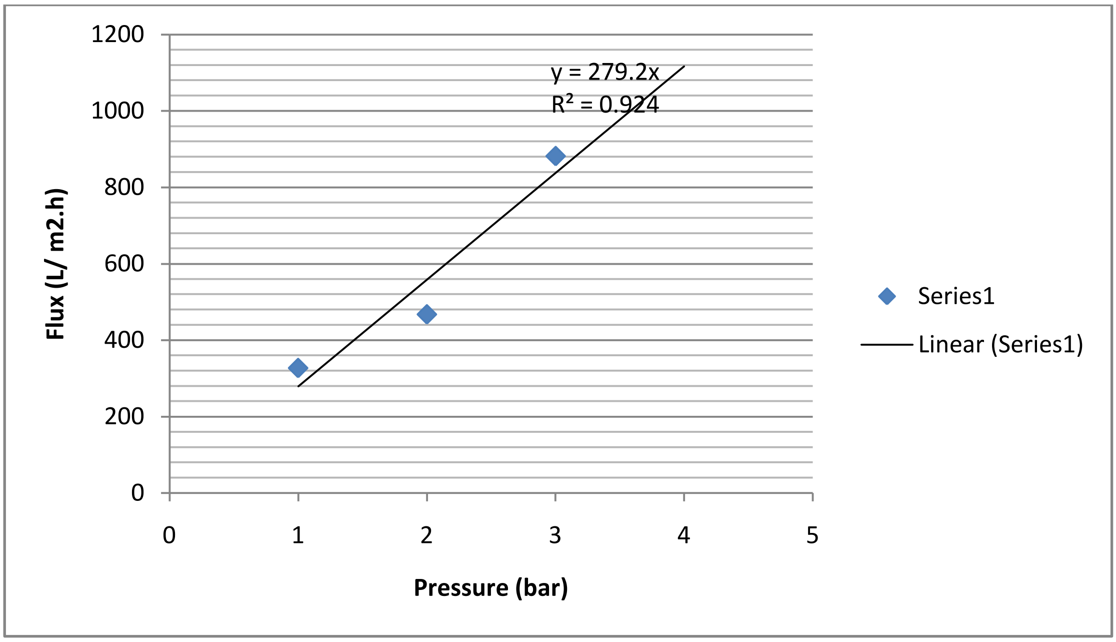

Figure 4.1: Water Flux of 18% PES Membrane vs. Pressure.

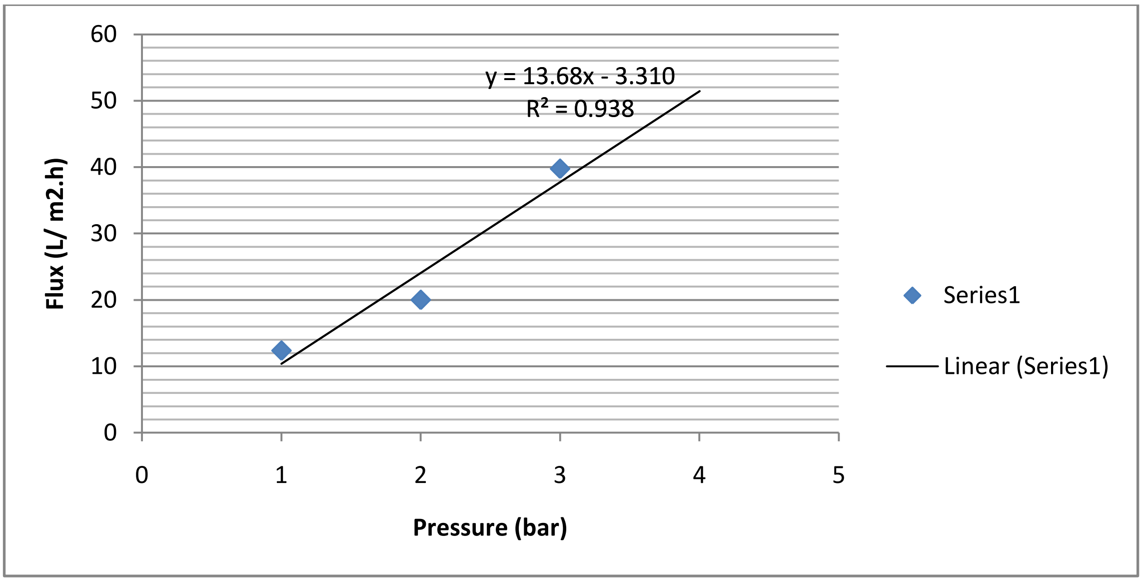

Figure 4.2: Water Flux of 20% PES Membrane vs. Pressure.

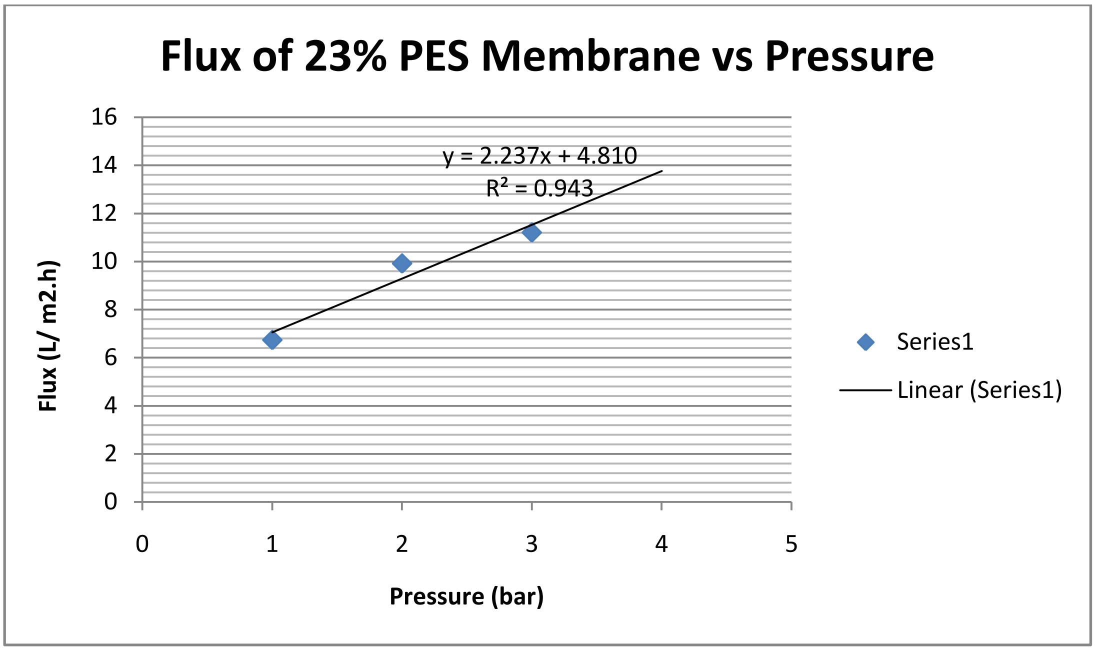

Figure 4.3: Water Flux of 23% PES Membrane vs. Pressure

The three linear graphs above represent the water flux vs. pressure. The slope of each graph is correspond to the permeability of each membrane which is 279.2 L/m^2h bar for 18% PES membrane, 13.68 L/m^2h bar for 23% PES membrane and 2.237 L/m^2h bar for 23% PES membrane. The higher the content of PES polymer, the tighten membrane produced leading to lower permeability. Khayet et al, (2005) observed that the permeate flux through membrane decreased clearly with increasing the membrane thickness while the separation factor remained nearly constant.

4.2 Standard Curve of MB

Standard curve of MB must be performed first to identify the concentration thus the rejection for the unknown concentration of dye solution could be determined. The standard curve is a general method for determining the concentration of a substance in an unknown sample by comparing the unknown to a set of standard samples of known concentration. The standard curve can be determined by using UV- Spectrophotometer using light in the visible and adjacent ranges. The absorption in the visible range affects the perceived of the chemical involved. During preparing the standard curve, the wavelength of MB is 665nm because of at this wavelength MB absorbs most of the light. Absorbance for different concentration is shown in Table 4.2 below.

Table 4.2: Absorbance for different concentration

Concentration of MB (mg/L)	ABS
2	0.048
5	0.401
10	1.096
15	2.009
20	2.620
25	2.969

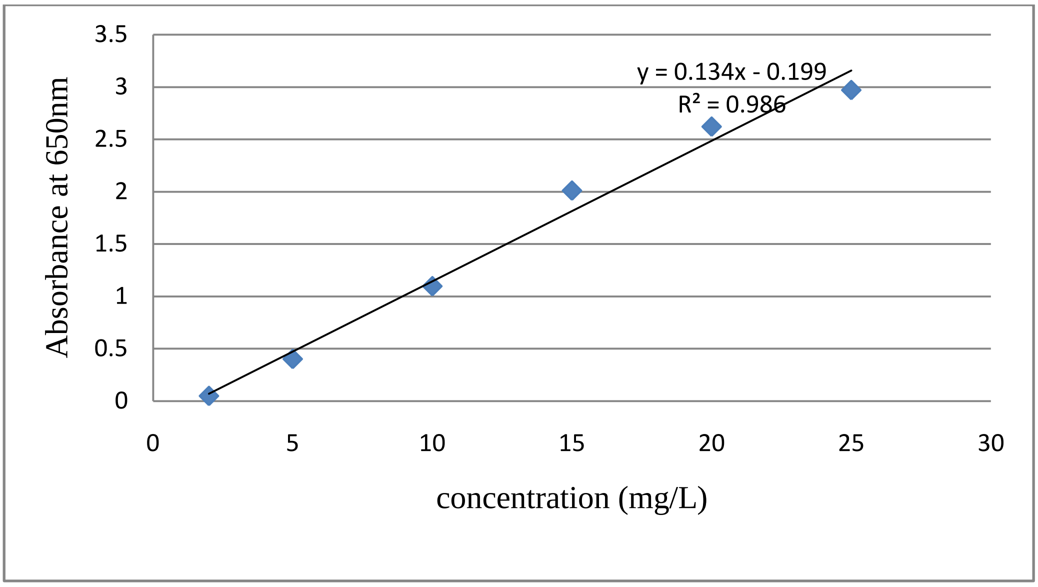

Figure 4.4: Standard Curve for Absorbance vs. Concentration.

The graph show that the concentration is directly proportional to the absorbance From the linear graph above, the concentration of MB solution after filtration can be determined by correlation between Absorbance and Concentration.

4.3 MB Removal

One of the membrane performances is measured by the membrane ability to remove any solute. In this study, the membrane ability to remove colour was represented by rejection of MB dye. Rejection of membrane is define as one minus the ratio of the permeate concentration to the permeate concentration to the concentration initial.

$$R = \left(1 - \frac{Cp}{Cf}\right) x\ 100$$

Table 4.3: The time taken for membrane to filtrate the MB.

Pressure (bar)	18% PES membrane		20% PES membrane		23% PES membrane	
	V(L)	t (h)	V(L)	t (h)	V(L)	t (h)
1	0.02	0.0247	0.02	0.2967	0.02	1.4978
2	0.02	0.015	0.02	0.13	0.02	0.6915
3	0.02	0.0080	0.02	0.0822	0.02	0.4351

Table 4.4: Absorbance and concentration of difference percentage of PES

Pressure(bar)	18% PES		20% PES		23% PES	
	abs	Conc(mg/L)	abs	conc(mg/L)	abs	Conc(mg/L)
1	2.786	22.27	2.531	20.37	1.299	11.18
2	2.5532	20.54	2.443	19.72	1.204	10.48
3	2.444	19.72	1.833	15.16	0.992	8.89

Table 4.5: Percentage rejection of difference percentage of PES

Pressure (bar)	18% PES membrane		20% PES membrane		23% PES membrane	
	% rejection	flux	% rejection	flux	% rejection	flux
1	11.00	282.1312	18.52	23.4872	55.28	4.6526
2	17.84	464.5760	21.12	53.6049	58.08	10.0776
3	21.12	871.0801	39.36	84.7767	64.64	16.0162

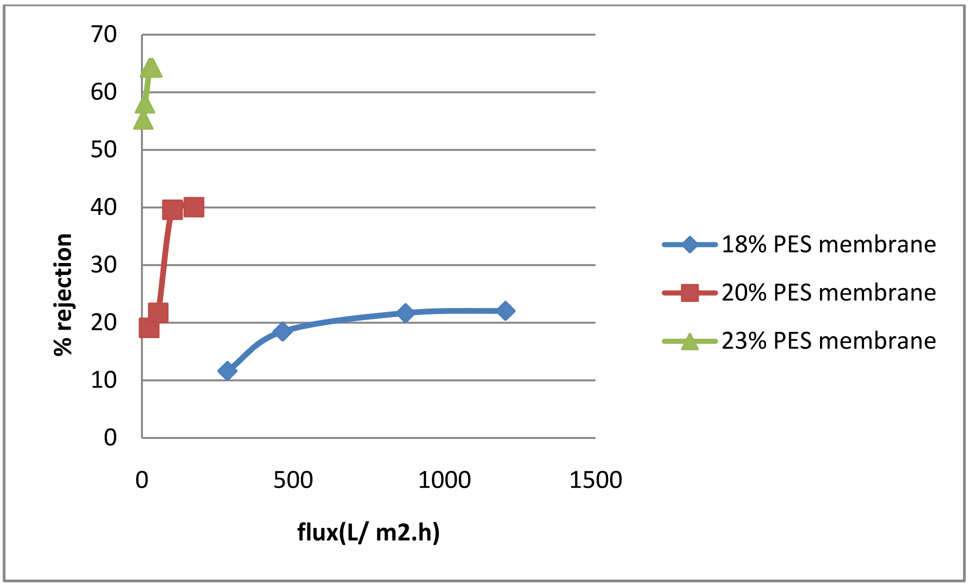

Figure 4.5: Graph of MB rejection vs. flux.

Figure 4.5 shows the percentage of rejection proportional with the flux. The 23% PES membrane showed the lowest value of flux with highest percentage rejection of dye. Meanwhile for the 18% PES membrane possess high value of flux but low percentage of rejection.

In a membrane separation technology, membrane performance is important to produce high selectivity and productivity of membrane. Microstructure and performance of membrane can be influenced by the properties of the casting solution such as choice of polymer, solvent non solvent and the polymer concentration (Zheng et al., 2006). Modify one of these variables, will turn out to different kinds of membrane structures that eventually affect the membrane. Polymer concentration is one of the variables that usually can manipulate the general formation of membrane thickness, membrane porosity.

By looking at Figure 4.1, Figure 4.2 and Figure 4.3 it is obviously revealed that the permeate flux decrease gradually with the increasing of percentage concentration of PES polymer. The permeability also decreases because the permeability is directly proportional with the flux. This is due to the tight porosity and thickness of the membrane itself. Adding the additive PVP in this study will increasing the membrane porosity. But since the PVP is act as dependent variable, the porosity will diminish as the amount of concentration PES increases. Beside, it is proven by Khayet et al, (2005) that the permeated flux through membrane decreases clearly with increasing the membrane thickness even as the separation factor remained constant.

The graph result of Figure 4.5 show that the percentage of rejection proportional with the flux. The graph illustrate 23% PES membrane gives lowest value of flux but high rejection compared with 18% PES membrane and 20% PES membrane. At 23% PES membrane the particle of dye is hardly to penetrate through the tight of the membrane. Furthermore, the porosity of membrane structure become narrower as the high percentage concentration of PES increase and the microvoid structure suppressed, which enhance the rejection ability. A thinner skin like 18% PES membrane resulted in higher flux rate. Therefore, to produce the high performance of membrane, amount of concentration of PES have to take count.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In a nutshell the phase inversion method is one of the method in fabricate the membrane for dye removal. By doing this research, we can conclude that effect of the concentration polymer PES while fabricating the membrane will influent the microstructure and also performance of membrane. It was shown as the percentage concentration of PES increase will consequent high percentage rejection of dye and low value of flux. This is due to the tight membrane porosity and thickness of the membrane. In this study, a percentage concentration of 23% PES is the most recommended because the separation performance was the most favourable. The objective of the research had been achieved

5.2 Recommendation

This study have own limitation therefore some recommendation have to do to make this membrane separation technology performance more efficient than before. The membrane separation can combine with the other separation technology such as chemical method in order to make the rejection of solutes could achieve 100% removal. Besides that, in the future, some modifications have to be done to make the higher value of flux as well as high performance of membrane can produced. .

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APPENDIX A

Calculation of percentage PES used.

Percentage of PES needed, 18%, 20%, 23%.

For 18% PES, 2% PVP

% NMP = 100- (18+2)

= 80%

Density of NMP, ρ = 1.032 kg/L volume NMP = 50 mL

Density $\rho = \frac{mass\ \ (m)}{volume\ \ (v)}$

Mass of NMP, *m* = (1.032 g/mL) (50 mL)

= 51.60 g

Mass of PES, $m = \frac{18}{80}$ X 51.6 g

= 11.61 g

Mass of PVP, $m = \frac{2}{80}$ X 51.6 g

= 1.29 g

PES		PVP	
Percentage (%)	Mass, m (g)	Percentage (%)	Mass, m (g)
18	11.61 g	2	1.29
20	13.23 g	2	1.32
23	15.82 g	2	1.38

APPENDIX B

Calculation for concentration of Methylene Blue (Standard curve)

Stock solution

1000 mg/L

Standard solution

$M_1V_I = M_2V_2$

500 mg/L (0.05 L) = 1000 mg/L (V_2)

V_2 = 25 mL

$M_1V_I = M_2V_2$

25 mg/L (0.01 L) = 500 mg/L (V_2)

V_2 = 0.5 mL

$M_1V_I = M_2V_2$

20 mg/L (0.01 L) = 25 mg/L (V_2)

V_2 = 8 mL

$M_1V_I = M_2V_2$

15 mg/L (0.01 L) = 20 mg/L (V_2)

V_2 = 7.5 mL

$M_1V_I = M_2V_2$

10 mg/L (0.01 L) = 15 mg/L (V_2)

V_2 = 6.67 mL

$M_1V_I = M_2V_2$

5 mg/L (0.01 L) = 10 mg/L (V_2)

V_2 = 5.0 mL

$M_1V_I = M_2V_2$

2 mg/L (0.01 L) = 5 mg/L (V_2)

V_2 = 4.0 mL

APPENDIX C

Time taken for distilled water to permeate.

18% PES Membrane

Pressure (bar)	1st reading		2nd reading		3rd reading		Average	
	V(L)	t (h)	V(L)	t (h)	V(L)	t (h)	V (L)	t (h)
1	0.02	0.0200	0.02	0.0245	0.02	0.0193	0.02	0.0213
2	0.02	0.0170	0.02	0.0190	0.02	0.0088	0.02	0.0149
3	0.02	0.0077	0.02	0.0093	0.02	0.0067	0.02	0.0079
4	0.02	0.0060	0.02	0.0067	0.02	0.0045	0.02	0.0057

20% PES Membrane

Pressure (bar)	1st reading		2nd reading		3rd reading		Average	
	V(L)	t (h)	V(L)	t (h)	V(L)	t (h)	V(L)	t (h)
1	0.02	0.1872	0.02	0.3885	0.02	1.1118	0.02	0.5625
2	0.02	0.1005	0.02	0.1187	0.02	0.8250	0.02	0.3481
3	0.02	0.0550	0.02	0.0525	0.02	0.4185	0.02	0.1753
4	0.02	0.0410	0.02	0.0375	0.02	0.1227	0.02	0.0671

23% PES Membrane

Pressure (bar)	1st reading		2nd reading		3rd reading		Average	
	V(L)	t (h)	V(L)	t (h)	V(L)	t (h)	V(L)	t (h)
1	0.02	0.2263	0.02	0.9202	0.02	1.9588	0.02	1.0351
2	0.02	0.1173	0.02	0.5028	0.02	1.4880	0.02	0.7027
3	0.02	0.0705	0.02	0.2890	0.02	0.8840	0.02	0.6218
4	0.02	0.0060	0.02	0.2075	0.02	0.4228	0.02	0.2121