

DYE REMOVAL BY THIN FILM COMPOSITE
(TFC) MEMBRANE PRODUCED THROUGH
INTERFACIAL POLYMERIZATION
TECHNIQUE

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BACHELOR OF CHEMICAL ENGINEERING
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Report submitted in partial fulfilment of the requirements for the award of the degree of
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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.

Signature

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Position:

Date:

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.

Signature

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ABSTRACT

There are many types of dye removal technique such as biological method, chemical method, and physical method. Membrane – filtration is one of the physical methods that widely use for dye removal. Thin film composite membranes were synthesized through interfacial polymerization at fix monomer concentration of 2% w/v at different reaction time. The reaction between triethanolamine (TEOA) aqueous and trimesoylchloride (TMC) in hexane produce new layer polyester on top of polyethersulfone (PES) miroporous support. The fabricated thin film composite membranes were characterized in term of flux and permeability. In addition, the membrane performances were tested for dye removal using methylene blue as dye model. Increasing the reaction time resulted in decreasing the water permeabilities and improving removal efficiency.

ABSTRAK

Terdapat banyak jenis teknik penyingkiran pewarna seperti kaedah biologi, kaedah kimia, dan kaedah fizikal. Membran – penapisan adalah salah satu kaedah fizikal yang meluas digunakan untuk penyingkiran pewarna. Membran komposit filem nipis disintesis melalui pempolimeran antaramuka pada kepekatan monomer 2% dan reaksi masa yang berbeza. Tindak balas antara larutan triethanolamine (TEOA) dan trimesoylchloride (TMC) dalam heksana menghasilkan lapisan baru poliester di atas miroporous sokongan polyethersulfone (PES). Membran filem nipis komposit yang dihasilkan dicirikan dari segi fluks dan ketelapan. Di samping itu, prestasi membrane telah diuji untuk penyingkiran pewarna menggunakan metilena biru sebagai model pewarna. Meningkatkan masa tindak balas menyebabkan mengurangkan kebolehtelapan air dan meningkatkan kecekapan penyingkiran.

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LIST OF SYMBOLS

C_p	Concentration of permeate
C_f	Concentration of feed
h	Hour
P	Permeability
b	bar
J	Permeate flux
L	Liter
M	Meter
ΔP	Filtration pressure
R	Rejection
Δt	Filtration time
V	Volume

LIST OF ABBREVIATIONS

TEOA	Triethanolamine
TMC	Trimesoylchloride
PES	Polyethersulfone
PVP	Polyvinylpyrrolidone
NMP	N-metyl-pyrrolidone
MB	Methylene Blue
UF	Ultrafiltration
RO	Reverse Osmosis
NF	Nanofiltration
IF	Interfacial Polymerization

CHAPTER 1

INTRODUCTION

1.1 RESEARCH BACKGROUND

Textile industries generally produced dye wastewater which generates from dyeing, printing, and other process of coloring in its daily operation. Facts shows, the dye wastewater contains many dangerous chemical such as dyes, detergents sulphide compound, solvents, heavy metals and other dangerous substances. Therefore, dye wastewater cannot directly discharged from the factory because it has striking effect on receiving water body which sooner or later can cause harm to the environment, aquatic life and especially human. Many textile industries out there use a huge amount of water to decrease the pollution load from discharged wastewater. That practice increase the textile industries operation cost just for water consumption. So, it is better if textile industries use one type of treatment system which profitable operation through recycling the dye wastewater and at the same time protecting the environment (Lau and Ismail, 2009). That treatment system is called Nanofiltration. This treatment system allow for recovery of water and valuable chemical compound from dye wastewater.

Nanofiltration membrane separation are widely use in various industrial fields. Wastewater treatment is one of the field that use this kind of technology to treat the water until the water reach the quality that it can be discharged to the environment or reusable for other process. Nanofiltration has been knew having the properties in between reverse osmosis and ultrafiltration (Mansourpanah *et. al.* 2010). Because of that, it has significant advantages including low operation pressure, higher permeation flux, high retention of multivalent salt, molecular weight compound more than 300, and low investments, operation and maintenance costs (Tang *et al.* 2008). Nanofiltration

membrane are produced by using two preparation steps which is polymer phase inversion resulting a microporous support membrane and interfacial polymerization of a thin film composite (TFC) layer on top of a microporous support membrane or other porous substrate (Mohammad *et al.* 2003).

The thin film composite layer can be prepared using interfacial polymerization. Thin-film composite membranes are usually studied in reverse osmosis and nanofiltration. The interfacial polymerization has significant advantages which involve rapid reaction rates under ambient conditions, no requirement for reactant stoichiometric balance and a low requirement for reactant purity (Li *et al.* 2008). The interfacial polymerization technique is an adequate method for the preparation of composite membranes with an ultra thin polyester active layer. In this study, thin-film polyester composite membranes were prepared using interfacial polymerization for dye removal.

1.2 PROBLEM STATEMENT

Dyes is one of the substance contain in textile wastewater. Dyes are considered as problematic because the families of chemical compounds that make good dyes are also toxic to humans. Each new synthetic dye developed is a brand new compound, and because it's new, no-one knows its risks to humans and the environment. Therefore, because of these reasons, dye from textile industrial wastewater need to be removed.

In this research, nanofiltration membrane process is used because of its advantages. The major advantages of nanofiltration are

- No chemical additive
- Removal for health-related contaminants
- Removal of suspended solids, some dissolve ions, and other non-health related contaminant.
- Cost effective and low maintenances costs.

Retrieved from (Nanofiltration article by Peter S. Cartwright)

`The chosen thin film composite membrane is polyester membrane which is considered new in the field of composite thin-film membrane development compared to polyamide for example. In addition, the future nanofiltration in textile industries is also provided in view of developing a more competitive nanofiltration membrane, especially for textile wastewater treatment.

1.3 RESEARCH OBJECTIVES

The objective of this experiment is to produce Nanofiltration Polyester membrane for dye removal.

1.4 SCOPES OF STUDY

In order to achieve the objective of this research, the scope of study has been determined based on two parameters to produce polyester. These are including effect of monomer concentration and reaction time on membrane performance.

1.5 SIGNIFICANCE OF STUDY

This research purpose is to prepare the NF membrane which suitable for dyes removing from textile industries. If the dyes completely can be removed by this filtration method, the wastewater discharged volume could be minimized and thus reducing impact on environment.

CHAPTER 2

LITERATURE REVIEW

This chapter will describe detail about the basic concepts of membrane separation technology, background of the membrane, type of membrane which will be use in the experiment (nanofiltration membrane) and also information about dyes and the technologies of dye removal. It will cover it characteristic, and the filtration mechanism.

2.1 MEMBRANE REVIEW

Membrane has been used in various fields such as waste treatment, medical purpose and many more. The selective permeability characteristic of membrane made it very useful especially in filtration and separation process.

2.1.1 Membrane Definition

The word membrane originally comes from the Latin word “*membrane*” which means skin. The other definition of membrane are a thin barrier that permits selective mass transport, selective barrier between two phases, and a phase that acts as a barrier to prevent mass movement, but allows restricted and / or regulated passage of one or more species (Wang *et. al.* 2011).

2.1.2 Membrane Separation Process

Membrane is a thin layer which allows smaller molecule liquid or gas than its pore size to pass through it. These pores size normally measured in Armstrong scale or micron. The thickness of the membrane usually is between 100 nm until a few centimeters over. The membrane layer is supported by a supported layer which is strong and thick. These limited routes of membrane only allow selected liquid or gas which means the other particle could not get into this membrane. The separation through membrane is effected by absorption, convection, concentration, pressure, the charge value of the solution, operation time and the temperature (Hesampour, 2009).

Membrane uses separation process as it operation. The basic membrane separation is shown in Figure 2.1

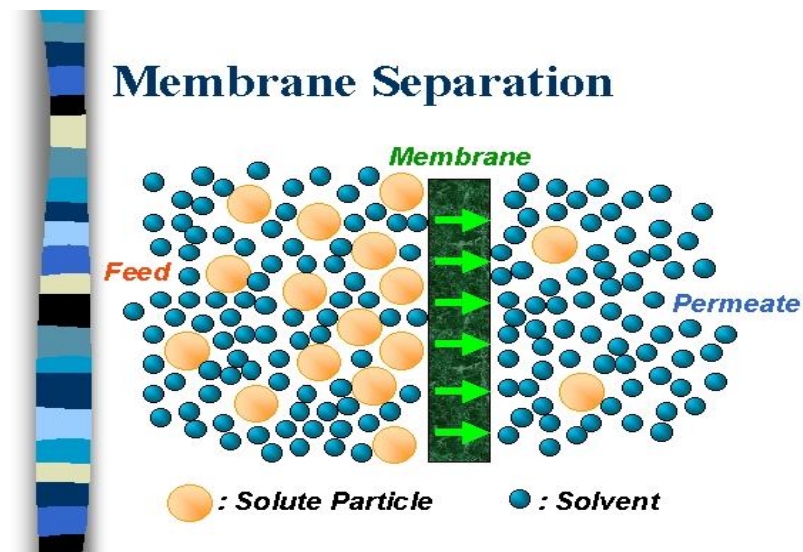


Figure 2.1: Basic Membrane Separation Process

Retrieved from (<http://www.yale.edu/env/elimelech/Conc-Polarization/sld002.htm>)

2.2 DRIVING FORCE OF MEMBRANE PROCESS

Based on the main driving force, which is applied to accomplish the separation, many membrane processes can be distinguished. An overview of the driving forces and the related membrane separation processes is given in Table 1.1

Table 2.1: Driving forces and their related membrane separation processes

Driving Force	Membrane Process
Pressure Differences	Microfiltration, ultrafiltration, nanofiltration, reverse osmosis or hyperfiltration
Chemical potential differences	Pervaporation, pertraction, dialysis, gas separation, vapor permeation, liquid membranes
Electrical potential differences	Electrodialysis, membrane electrophoresis, membrane electrolysis
Temperature differences	Membrane distillation

Source adapted from (Timmer, 2001)

Each membrane processes have their own mechanism and applications. For example, pressure driven membrane separation processes, electrodialysis and gas separation are industrially implemented and are generally considered as proven technology (Timmer, 2001).

2.2.1 Pressure Driven Membrane

Four pressures driven membranes are distinguished in practice (Timmer, 2001)

1. Microfiltration (MF), which is characterized by a membrane pore size between 0.05 and 2 μm and operating pressures below 2 bars. MF is primarily used to separate particles and bacteria from other smaller solutes.
2. Ultrafiltration (UF), this is characterized by a membrane pore size between 2 nm and 0.05 μm and operating pressures between 1 and 10 bars. UF is used to separate colloids like proteins from small molecules like sugars and salts.
3. Nanofiltration (NF), this is characterized by a membrane pore size between 0.5 and 2 nm and operating pressures between 5 and 40 bars. NF is used to achieve separation between sugars, other organic molecules and multivalent salt on one hand and monovalent salts, ions and water on the other.
4. Reverse osmosis (RO) or hyper filtration, which is characterized by a membrane pore size in the range of 0.0005 microns. Some researchers consider the RO membrane as without have pores. Transport of the solvent is accomplished through the free volume between the segments of the polymer of which the membrane is constituted. The operating pressures in RO are generally between 7 and 100 bar and this technique is mainly used to remove water.

Membrane Application can be seen in Table 1.2

Table 2.2: Membrane Applications

Process	Advantages
Microfiltration	Sterile solution, water purification, baverage filtrationeffluents
Ultrafiltration	Protein concentration(enzyme), oily wastewaters effluent, blood fractionation, antibiotic separation
Nanofiltration	Potable water, desalination of brackish water, polyvalent ions stream cleaning, whey fractionation
Reverse Osmosis	Food concentration, water purification, desalination (monovalent ions stream), biomedical application
Electrodialysis	Desalination, water purification, deacidification of citrus juice.
Gas permeation	Separation of He from natural gas, He recovery, CO ₂ removal, NG dehydration
Pervaporation	Dehydration of organic solvents

Source adapted from (Muhsen, 2011)

2.2.2 Advantages of Membrane Processes

Compared with the conventional methods, membrane systems offer more economical benefits over conventional systems. The followings are specific criteria of the membrane system (Mustaffar, 2004):

1. Flexibility and versatility; as this technology can be applied at wide spectrum of separation ranges comparable to conventional methods. The system can be operated in combination with the conventional methods and /or on its own.
2. Simplicity of operation; that is the system is relatively less complex and less sophisticated.
3. Low energy consumption; membrane systems consume less energy since separation does not involve phase change. Thus the system driving force is mainly pressure provided by using pump.
4. Low capital and maintenance cost; the overall membrane system is (30-40) % cheaper than the corresponding conventional systems.
5. Reliability; the membrane system is very reliable and requires minimum human intervention during operation.
6. Physical separation; separation is done physically thereby undesirable by products and no side reactions, no waste generation and is environmentally green.

2.3 NANOFILTRATION MEMBRANE

Nanofiltration membrane is classified as anisotropic membranes. Anisotropic membrane are non-uniform over cross-section and they typically consist of layers which vary in structure and/or chemical composition. There are two main types of anisotropic membrane which are phase separation membranes and thin film composite membranes. Phase separation membranes are homogeneous in chemical composition but not in structure and often consist of a rather dense layer of polymer on the surface of an increasingly porous layer. This membrane is produced via phase inversion. While thin film composite membranes are both chemically and structurally heterogeneous. Thin film composites usually consist of a highly porous substrate coated with a thin dense film of a different polymer. This membrane can be made via several methods including interfacial polymerization, solution coating, plasma polymerization or surface treatment.

2.3.1 The Asymmetry

The Nanofiltration membrane is an asymmetry membrane. It will not allow the larger molecule to pass through the membrane and will stop at the surface. While the smaller molecule will pass through the membrane and known as permeate.

2.3.2 Pore Size

There are many types of membrane used in industry and one of them is nanofiltration. The nominal pore size for this type of membrane is between 0.5 and 2 nanometer.

2.3.3 Mass Transfer in Nanofiltration

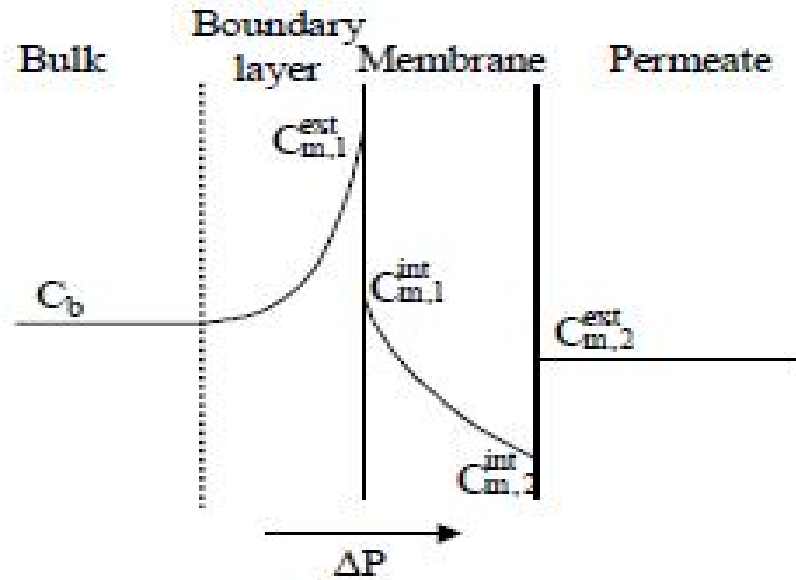


Figure 2.2: Mass transfer in nanofiltration

When an external pressure ΔP is imposed on a liquid which is adjacent to a semi-permeable membrane, solvent will flow through the membrane. The general terms that are used in the description of membrane separation processes are the solvent flux (J) and the rejection (R). The solvent flux is given by Eq. 2.1

$$J = \frac{W}{A\Delta t} \quad (2.1)$$

Where W is the weight of the obtained permeate during a predetermined Nanofiltration operation time Δt and A is the membrane area.

A neutral solute dissolved in the solvent at a concentration level C_f will also flow towards the membrane. If the membrane exhibits rejection for the solute, partial permeation will occur and non-permeated solute accumulates in the boundary layer, and hence a concentration profile develops. This phenomenon is called concentration polarisation. The solute distributes at the membrane/solution interface and will be transported through the membrane by convection and diffusion. At the permeate side, a

second distribution process will occur and a final concentration of solute in the permeate, C_p will be reached. Rejection as Eq. 2.2

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100 \quad (2.2)$$

The membrane permeance, P_m can be determined from the slope obtained by plotting the permeate flux J against ΔP :

$$P_m = \frac{J}{\Delta P} \quad (2.3)$$

2.4 THIN FILM COMPOSITE MEMBRANE

Thin film composite the top layer is formed by a variety of techniques ranging from simple solution casting such as spin coating to intricate polymerizations like interfacial polymerization, in-situ polymerization, plasma polymerization and grafting (Dalwani, 2011).

2.4.1 Spin Coating

Spin coating is quick and easy laboratory method to produce thin film composite. This technique use centrifugal force mechanism. An excess of solution is pour on the ultraporous substrate that is then rotated at high speed. A uniform layer will form on substrate when liquid spreads due to the centrifugal forces. Evaporation of solvent results a uniform solid polymer coating. The schematic of spin coater is shown as Figure 2.3

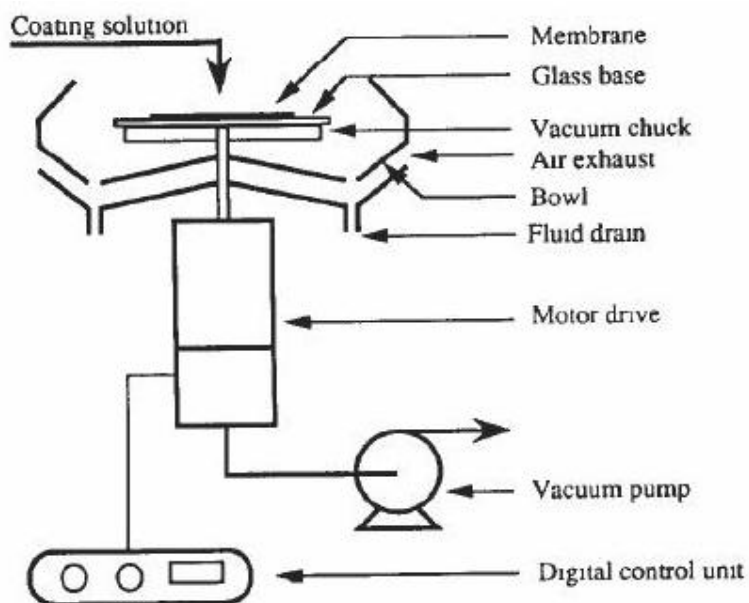


Figure 2.3: Schematic of spin coater

Source adapted from (Dalwani, 2011)

The quality of the thin coating layer depends mainly on the spinning speed, solvent evaporation rate, viscosity or concentration of the polymer solution and the pore and surface characteristics of the support. Spin coating has limited application in the field of membrane technology on a commercial scale, due to restricted product size, but benefits from the minimal amount of required polymer solution.

2.4.2 Interfacial Polymerization

The thin film composite was produced via polymerization which takes place at the interface of the two liquids which are insoluble to each other (Lau *et.al* 2011). It is carried out between two highly reactive monomers dissolved in two immiscible solvents (Dalwani, 2011). Even though the interfacial polymerization (IP) involves many complex parameters compared to spin coating, it is the major technique to produce commercial TFC membranes because composite membranes with surprisingly high flux could be made by interfacial crosslinking. The IP process consists of a sequence of steps, as shown in Figure 2.4. In the first step an ultraporous support is pretreated to make it suitable for the IP process. In the second step, the substrate is immersed in aqueous solution containing one of the monomer. Then, continue immersed in second solution containing the second monomer. Since the two solutions are immiscible to each other, the interface is created between them. One of the monomer travels through the interface and react with the other polymer to produce polymerized product. IP layer usually dense and very thin due to high reactivity of two monomers and limited barrier created at interface.

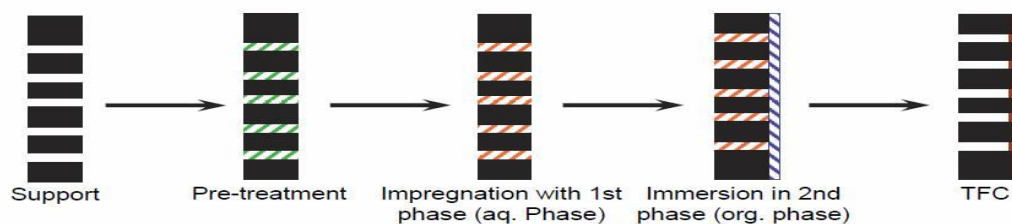


Figure 2.4: Interfacial Polymerization Process

Source adapted from (Dalwani, 2011)

In order to produce a variety type with high performance of membrane, there are several parameters which can be varied during IP process which is

1. Different monomer types – Many types of monomer can be used in both aqueous and organic phase. Piperazine is an example of monomer used to make NF membrane as aqueous phase reactant and types of acyl chloride such as trimes
2. Monomer concentration and reaction time – In general high monomer concentration, high reaction rates, longer polymerization time results in thicker layers with high rejection but lower flux.

2.4.3 Triethanolamine (TEOA) as Monomer

There is only few research that use TEOA as monomer as well as to produce polyester composite membrane which considered new because most of the research for the composite membranes are more to the production of polyamide. Compare to other monomer like Bisphenol A which also used to produce polyester membrane, TEOA is less dangerous to human health. Then, it is natural to seek an inexpensive monomer to manufacture a new membrane. It is found that triethanolamine (TEOA) is an active monomer which is environment-friendly, economical and easy to be obtained (Tang *et. al*, 2008).

2.5 DYES

Dye is the substance that has characteristic like colored, ionizing, and aromatic compounds which shows affinity towards solution that is aqueous. Generally, dyes more used in industries such as printing, cosmetic, leather and textile industries. The dye being used widely is synthetic dyes which are easier and low cost in synthesis, firmness, more stable in light, detergent and microbial attack. Besides that, synthetic dyes are made in varieties of color compared to natural color.

Dyes can be classified as problematic compounds because it can pollute the environment. Dye is soluble in water and difficult to be decolorized and decomposed biologically. Dye usually withstand to biological attack, light, heat and oxidation and it also has very low concentration and need to remove before water can be discharged (Ahmad *et al.* 2002).

Dye molecule contains two groups which is called chromophores and auxochromes. Chromophores are part which responsible for its color and auxochromes helps a dye to bind to the object that is to be colored.

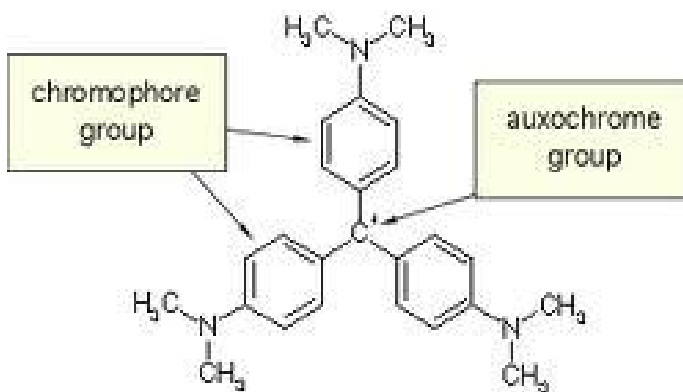
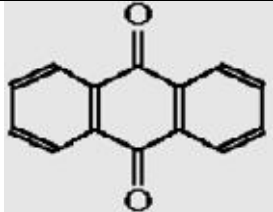
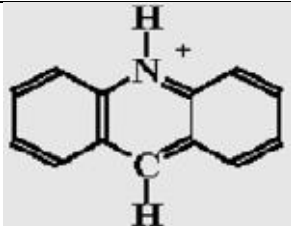
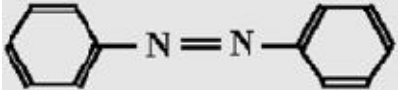
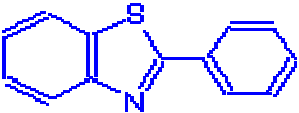
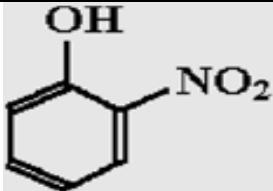
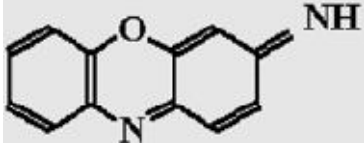


Figure 2.5: Chromophores and auxochromes in dye.

Retrived from (<http://eglobalmed.com>)

Many types of dyes are poisonous and have carcinogenic and mutagenic effects that can affect aquatic lives and also humans. The carcinogenic was comes from known carcinogens such as benzene or other aromatic compound that might be formed as a result of microbial metabolism (Susana, 2009). Different dyes will have different chromophoric group. For examples:

Table 2.3: Types of Chromophores

Class	General Formula
Anthraquinone	
Acridine	
Azo	
Thiazole	
Nitro	
Oxazin	

Source adapted from (<http://stainsfile.info/StainsFile/dyes/class/dye/class>)

There is more than one type of dyes in the industries. Some properties of dyes can be classified based on the usage of the dye (Gupta, 2009). Each type of dyes has different usage or purpose.

Table 2.4: Properties of dye and usage

Type of dye	Usage	Principle chemical classes
Acid dyes	Nylon, wool, silk, modified acrylics, and also to some extent for paper, leather, ink-jet printing, food, and cosmetics.	Azo (including premetallized), anthraquinone, triphenylmethane, azine, xanthene, nitro and nitroso.
Direct dyes	Dyeing of cotton and rayon, paper, leather, and, to some extent to nylon.	Polyazo compounds, along with some stilbenes,.
Reactive dyes	Cotton and other cellulose, but are also used to a small extent on wool and nylon.	Azo, anthraquinone, triaryl methane, phthalocyanine, formazan, oxazine, etc
Sulphur dyes	Cotton and rayon and have limited use with polyamide fibers, silk, leather, paper, and wood.	Have intermediate structures
Vat dyes	Cotton mainly to cellulosic fibers as soluble leuco salts and for rayon and wool too.	Anthraquinone (including polycyclic quinones) and indigoids.
Azoic dyes	Cotton and other cellulosic materials, fluorescent brighteners having stilbene, pyrazoles, coumarin and naphthalimides used for soaps and detergents, fibers, oils, paints, and plastics.	Azo group

Source adapted from (Gupta, 2009)

2.5.1 Methylene Blue Dye

The dye model used for this research is called Methylene Blue. Other name for this dye is Swiss Blue. It is in Thiazin class or which call Thiazole. Thiazole dyes contain Thiazole ring which is heterocyclic compound that contains both sulfur and nitrogen.

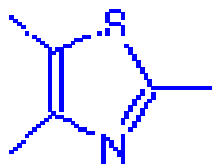


Figure 2.6: Thiazole ring

The empirical formulae of this Methylene Blue are $C_{16}H_{18}N_3SCl$. Methylene blue is a very commonly used dye. It stains nuclei blue, and is often used for that purpose.

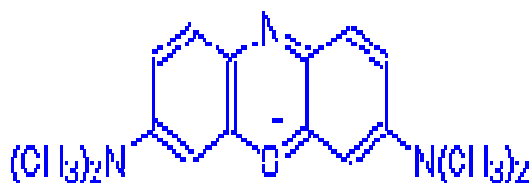


Figure 2.7: Methylene Blue General Formulae

Methylene blue (MB) has wide applications, which include paper coloring, temporary hair colorant, dying cottons, and wools. Although not strongly hazardous, it can cause some harmful effects in humans such as heartbeat increase, vomiting, shock, cyanosis, jaundice, quadriplegia, and tissue necrosis (Zendehdel *et. al.* 2010).

2.6 TECHNOLOGIES FOR DYE REMOVAL

There are three categories for dye removal technique which is biological, chemical and physical. All of them have advantages and disadvantages. Source adapt from (<http://publicweb.unimap.edu.my/>)

2.6.1 Biological Method

Compared with other physical and chemical methods, biological is the most economical alternative. Example of biological treatment is microbial degradation, adsorption by microbial biomass and fungal decolorization. But their application is limited because of technical constraint. Biological treatment requires a large land, less flexibility in design and operation, and incapable of obtaining satisfactory color dismissal due to dye's complex chemical structure and its synthetic organic origin.

2.6.2 Chemical Method

Coagulation, flocculation, electroflotation, electrokinetic coagulation, conventional oxidation techniques, irradiation and electrochemical processes are some example of chemical method for color elimination. All these techniques are often expensive, even dyes are removed, accumulation of concentrated sludge may create a disposal problem and also the probability that other pollution problem will arise because of excessive chemical usage.

2.6.3 Physical Method

There are two major process of physical method that widely used for dye removal, which is membrane – filtration process and adsorption technique. Membrane has advantages such as no chemical use, can use both for liquid and gas filtration and many more. Adsorption has advantages of flexibility and simplicity of design, ease of operation and insensitivity to toxic pollutants.

Table 2.5: Advantages of physical and chemical methods of dye removal from industrial effluent

Physical/chemical method	Advantages	Disadvantages
Fentons reagent	Effective decolourisation of both soluble and insoluble dyes	Sludge generation
Ozonation	Applied in gaseous state: no alteration of volume	Short half-life (20 min)
Photochemical	No sludge production	Formation of by-product
NaOCl	Initiates and accelerates azo-bond cleavage	Release of aromatic amine
Cucurbituril	Good sorption capacity for various dyes	High cost
Electrochemical destruction	Breakdown compounds are non-hazardous	High cost of electricity
Activated carbon	Good removal of wide variety of dyes	Very expensive
Peat	Good adsorbent due to cellular structure	Specific surface area for adsorption are lower than activated carbon
Wood chips	Good sorption capacity for acid dyes	Requires long retention times
Silica gel	Effective for basic dye removal	Side reactions prevent commercial application
Membrane filtration	Remove all dye types	Concentrated sludge production
Ion exchange	Regeneration: no adsorbent loss	Not effective for all dyes
Irradiation	Effective oxidation at lab scale	Requires a lot of dissolved O ₂
Elektrokinetic coagulation	Economically feasible	High sludge production

Source adapted from (Ahmad *et. al.* 2002)

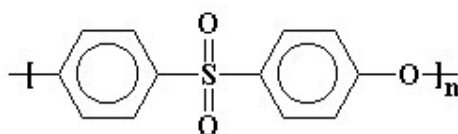
CHAPTER 3

METHODOLOGY

In this chapter, there will be an explanation about the material used and detailed procedure that be going through in the experiment to achieve the objective of this research.

3.1 MATERIAL

In this research, material used for membrane preparation is polyethersulfone palette (PES), polyvinylpyrrolidone (PVP), N-methyl-2-pyrrolidone (NMP), triethanolamine (TEOA), trimesoylchloride (TMC), sodium hydroxide (NaOH), and n-hexane. Methylene Blue (MB) is used as dye model in the filtration process.



Polyethersulfone Structure

Figure 3.1: Structural formulae of PES

3.2 DYE SOLUTION PREPARATION

For preparing the dye solution, MB stock solution was prepared by dissolving the accurate dye weight in distilled water to the concentration of 1 g/L. Then, the stock solution will dilute to become 0.025g/L for dye removal initial concentration.

3.3 MEMBRANE PREPARATION

3.3.1 PES Base Membrane

PES base ultrafiltration membrane was prepared through phase-inversion method. Firstly, a solution composed of 18wt% PES and 15wt% PVP dissolved in the solvent N-methyl-2-pyrrolidone. Before PES and PVP dissolved, ensure both well mixed in other flask. This solution is prepared by stirring the powder of PES and PVP in the NMP solvent at certain speed by using a magnetic stirrer hotplate. A yellow sticky solution like honey will be produced which have a lot of bubble inside. This solution should bubble free by leaving for several hours at room temperature until no more bubbles appear in that solution. Then, pour the solution to the glass plate covered with drawing paper at room temperature and spread it using doctor bar with opening of 0.25mm. Quickly but gently, immerse the liquid film in water bath. That liquid film turned into dark white due to precipitation of PES. About 1 to 2 minutes, the film will automatically come off from the glass plate. After get the PES membrane, rinse it with deionized water and stored at 20% glycerol solution to avoid pore collapse or shrinkage.

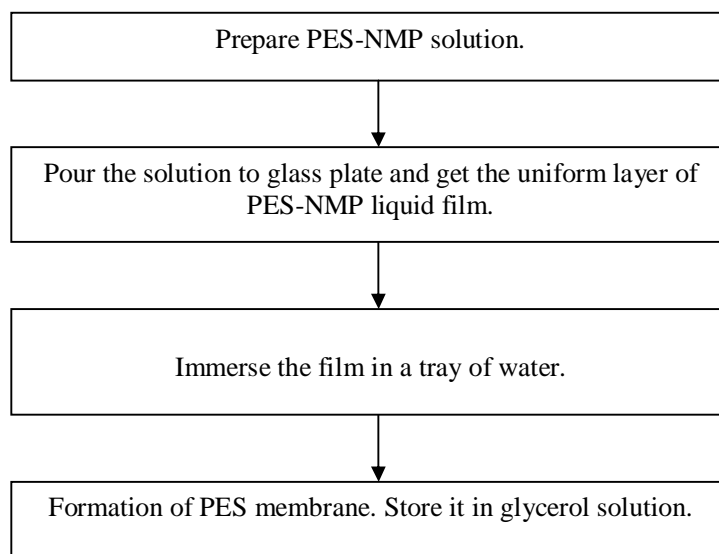


Figure 3.2: Preparation of PES membrane

3.3.2 Preparation of Polyester Membrane

Polyester NF membrane was prepared through interfacial polymerization method by coating the PES membrane. Firstly, sodium hydroxide solution with concentration of 1 wt% prepared by dissolving 10 g sodium hydroxide in 1000 ml of distilled water. It is used as a solvent for TEOA. TEOA aqueous solution with the concentration of 2wt% prepared by dissolving 2g TEOA in 100 ml aqueous solution of sodium hydroxide (NaOH 1 wt %). Then, TMC organic solution (TMC-hexane) with 0.15% weight concentration was prepared in the form of organic phase by dissolving 0.15g TMC in 100 ml n-hexane. Immerse the PES coupon membrane into TEOA aqueous solution for 30 minutes. After that, take out the coupon and drain the excess TEOA solution on its surface by using rubber rollers. Next, that coupon dipped in TMC-hexane organic solution and left it for predetermined time for interfacial polymerization to occur. The polyester composite membrane that produced is dried in air for overnight. About 3 membrane sample were produced for each different concentration and immersion time. The preparation of these NF composite membranes is repeated by changing or varying the concentration of TEOA aqueous solution and the immersion (reaction) time in organic solution.

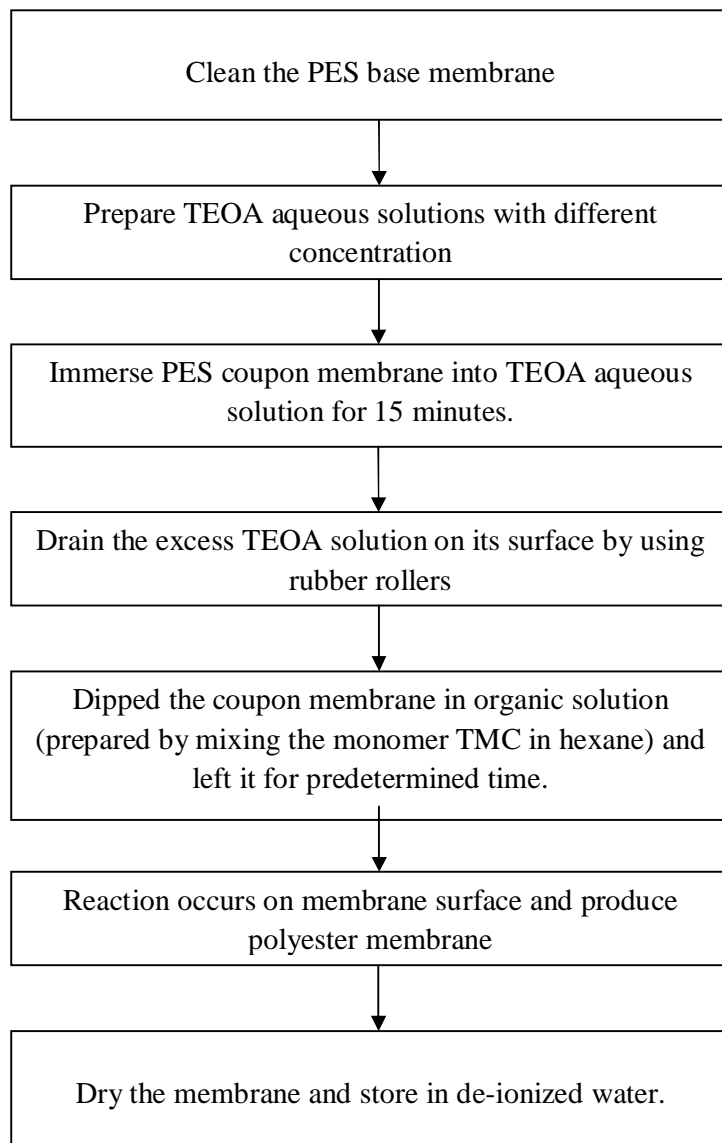


Figure 3.3: Preparation Polyester NF membrane through interfacial polymerization

3.4 MEMBRANE SEPARATION PROCESS

For membrane separation process, this research used equipment called Amicon stirred cell. The process is operates under pressure range between 100kPa to 500kPa.



Figure 3.4: Amicon Model 8200

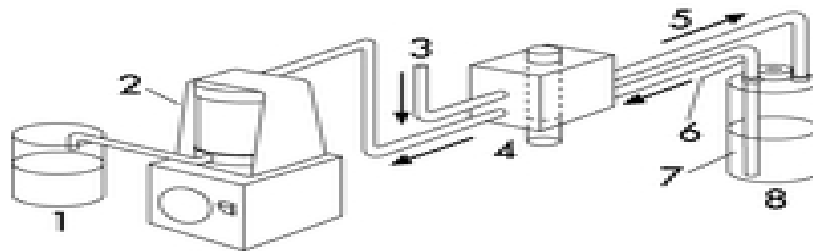


Figure 3.5: Schematic diagram of Amicon stirred cell

1. Permeate will collected here
2. Filtration Amicon – this is where membrane separation occur
3. Pressure supply
4. Selector valve
5. Inlet
6. Outlet – this is where the feed goes to filtration equipment
7. Feed liquid
8. Reservoir

3.5 OVERALL PROCESS FOR DYE REMOVAL

The overall process of dye removal is simplified as shown in Figure 3.6

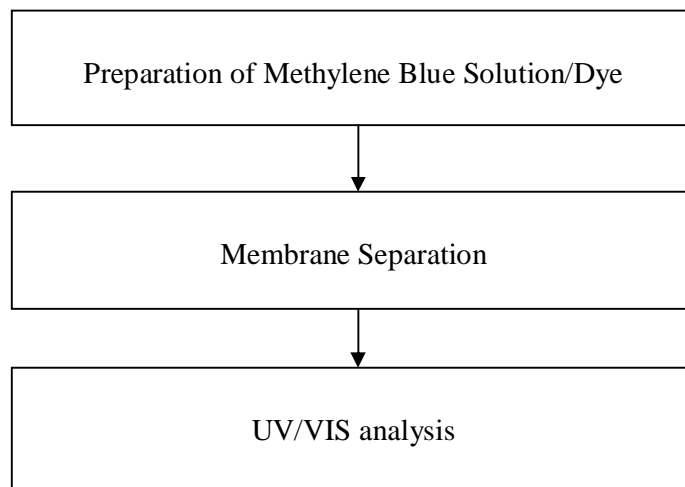


Figure 3.6: Overall Methodology

3.6 EQUIPMENT

3.6.1 Glass Rod and Glass Plate

Glass rod is used to replace the doctor bar due to lack of skill in its use which cause the membrane defects. Both ends of the glass rod is wrapped with a thin transparent tape as much as 3 to 5 turns to determine the required thickness of the membrane with a glass plate.

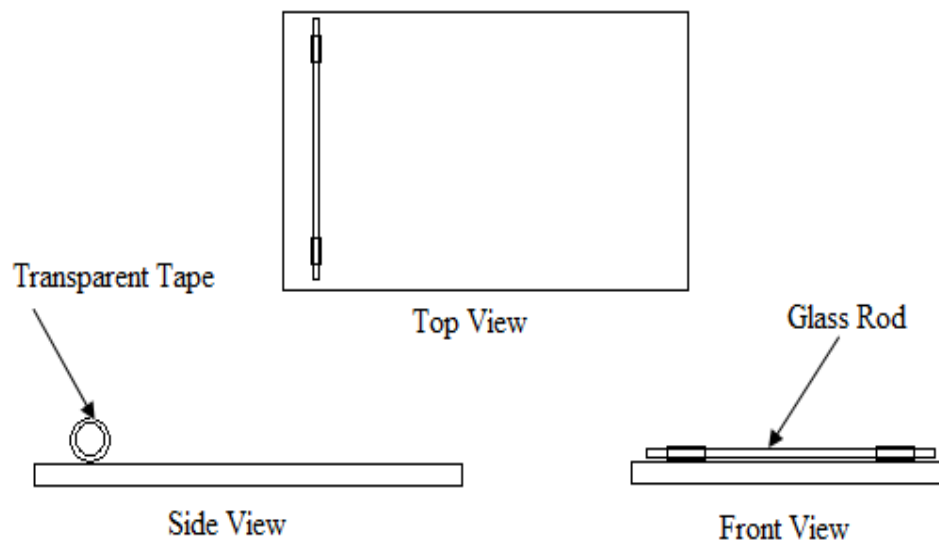


Figure 3.7: Arrangement of glass rod with glass plate.

3.6.2 Hot Plate and Blending Motor

Hot plate was used in the blending process of polyethersulfone (PES) in the solvent N-methyl-2-pyrrolidone (NMP). It supply the heat to the solution to dissolve quickly with the help of blending motor as Figure 3.8

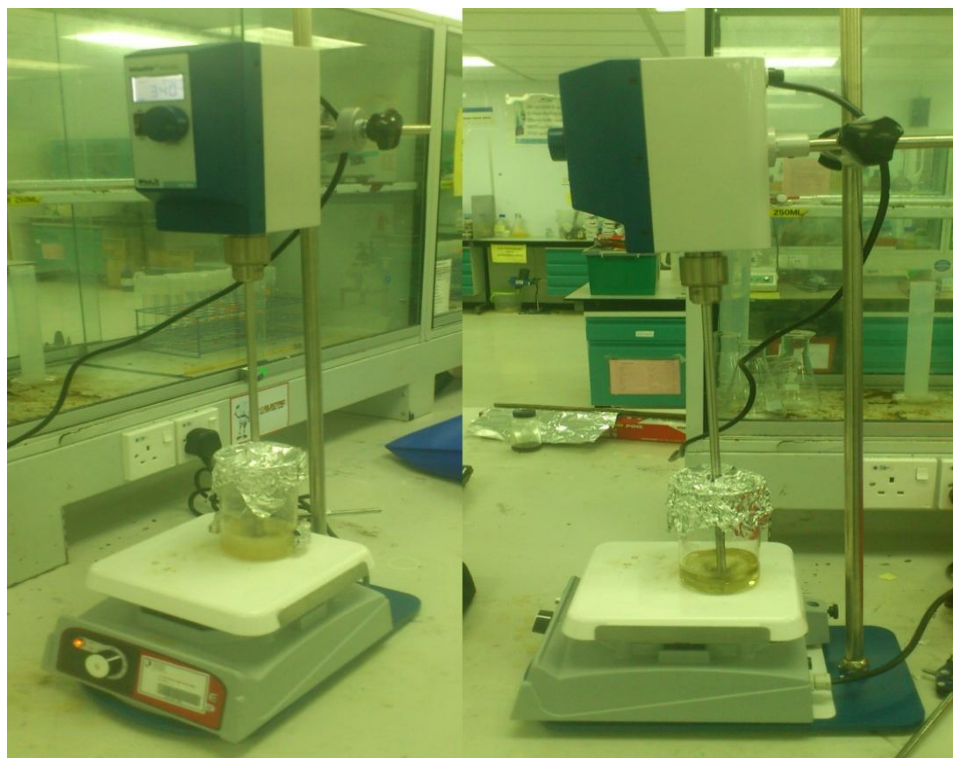


Figure 3.8: Blending Process

3.6.3 Nitrogen Gas

Nitrogen gas used to give the pressure to Amicon stirred cell. Operation pressure is set between 100kPa (1 bar) to 500kPa (bar) by valve regulator. Safety precautions when handling nitrogen gas should be noted that nitrogen gas is very dangerous to the human body. Make sure there are no leaks in the hose connecting the nitrogen tank with Amicon stirred cell.

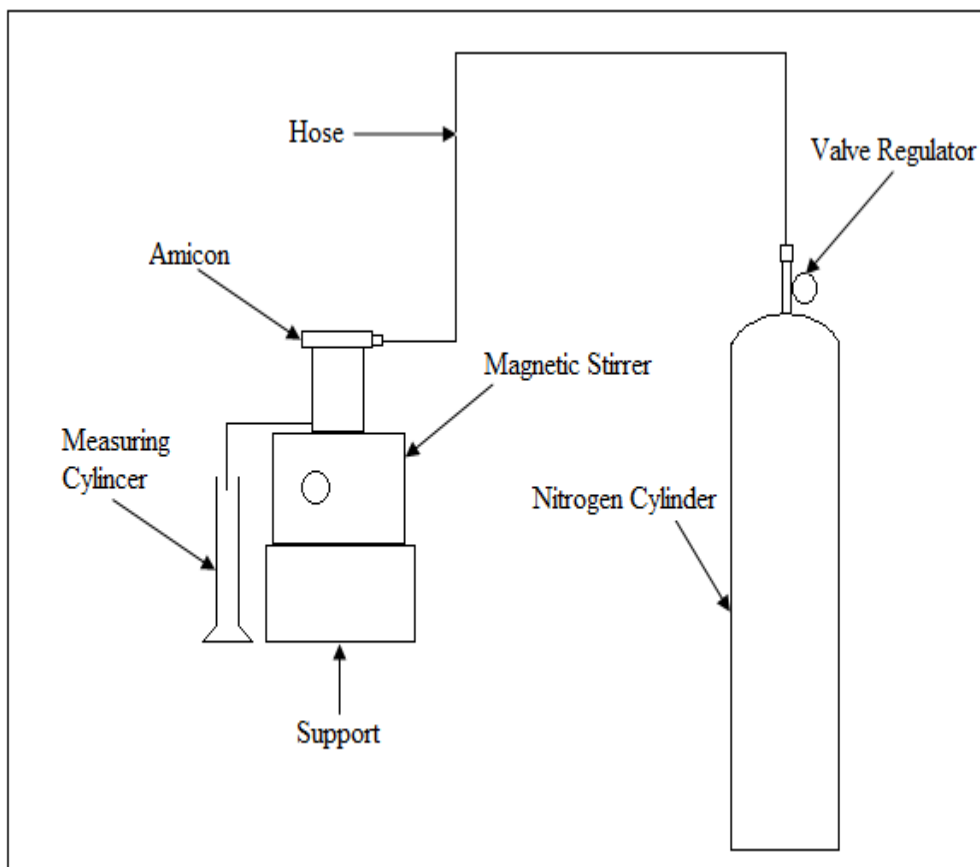


Figure 3.9: Nitrogen Cylinder attached to Amicon Stirred Cell

CHAPTER 4

RESULT AND DISCUSSION

4.1 STANDARD CURVE OF METHYLENE BLUE

Before start the dye removal process, the standard curve of Methylene Blue must be prepared at wavelength of 660 nm. The initial concentration before dye removal process is fixed at 25mg/L for each filtration. So, the ABS value for below the initial concentration must be known and plotted as graph. Using UV-Visible spectrophotometer, the ABS values at concentration at 25mg/L and below were taken and show as Table 4.1.

Table 4.1: ABS values of MB.

Concentration, mg/L	ABS
2	0.038
5	0.445
10	1.096
15	1.989
20	2.58
25	2.971

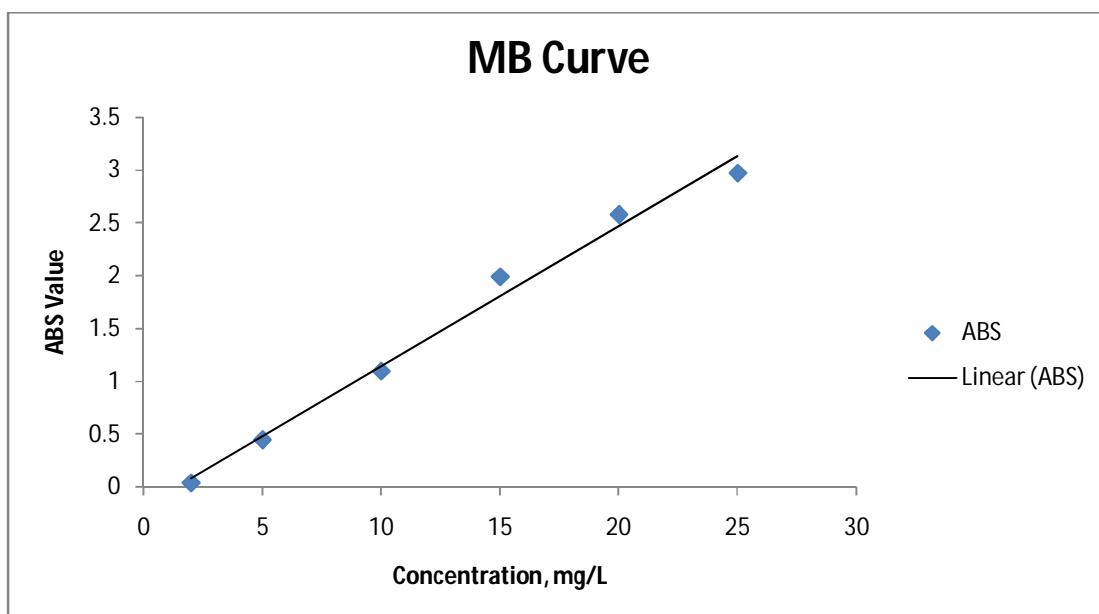


Figure 4.1: Standard Curve of Methylene Blue

From the straight line at graph above, the final concentration of MB dye after the filtration process can be determine by taking the ABS value of final filtrate using UV-Vis.

4.2 PURE WATER FLUX AND PERMEABILITIES

There are total of three membrane studied which is all of them are membrane coated with 2% concentration of TEOA monomer aqueous solution at three different immerse time in TMC organic solution. Each membrane was labelled such as M2-30S refers to 2% TEOA membrane at 30 second immersion time.

Flux for each membrane is determined by testing using distilled water. From flux equation 2.1, whether volume of permeate or time can be fixed at some value to determine the value of the other. The effective surface area of the membrane is 0.00287 m^2 . For this research, the volume for permeate is being set at 20ml. So, the time for each membrane to get 20ml permeate was recorded.

4.2.1 Performance of Uncoated PES Membrane

Table 4.2 below shows water fluxes for the uncoated PES membrane which prepared by phase inversion method. Volume of permeate is fixed at 20ml. Time for collected 20ml of permeate was recorded at different operating pressure.

Table 4.2: Flux of uncoated PES Membrane

Pressure	Time, h	Flux, L/ $\text{m}^2 \cdot \text{h}$
5	0.1221	57.073
4	0.1267	55.001
3	0.1389	50.170
2	0.1802	38.672
1	0.2085	33.423

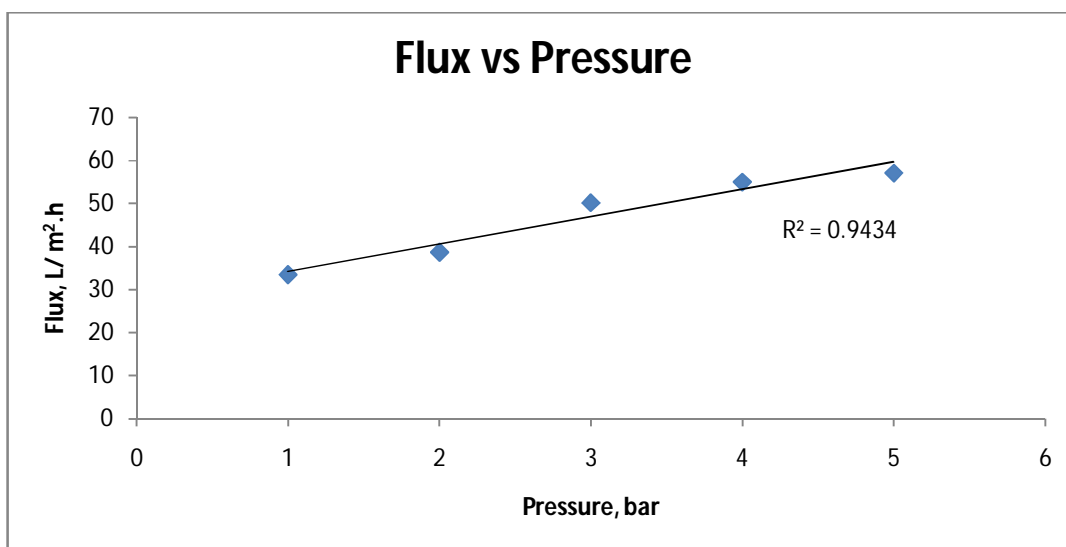


Figure 4.2: Graph Flux vs. Pressure of Uncoated Membrane

Figure 4.2 above show the gradient of the graph or water permeability of the uncoated membrane is $6.3629 \text{ Lm}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$. This value will be use as reference to prove the presence of thin layer on coated membranes.

4.2.2 Performance of 2% TEOA – Polyester Membrane

As shown in Table 4.3, the membrane performance improved after being coated with the monomer at aqueous and organic solutions. The flux of coated membranes was decrease but showing better performance in filtration process compare to uncoated membrane. The time for collect the filtrate also increase as immersion time increase as shown in time column. From data collected, it proves the formation of dense thin layer on the surface of PES support membranes cause the water to take a longer time to cross the membrane layer.

Table 4.3: Flux for 2% TEOA Membranes at different reaction time

Membrane	Pressure (bar)	Time (h)	Flux (L/ m ² .h)
M2-30S	5	0.1357	51.3533
	4	0.1383	50.3879
	3	0.1545	45.1045
	2	0.2132	32.6859
	1	0.2633	26.4665
M2-45S	5	0.1605	43.4183
	4	0.1702	40.9438
	3	0.2247	31.0131
	2	0.2627	26.5270
	1	0.2935	23.7432
M2-60S	5	0.1920	36.2950
	4	0.1988	35.0535
	3	0.2475	28.1561
	2	0.2715	25.6672
	1	0.3022	23.0597

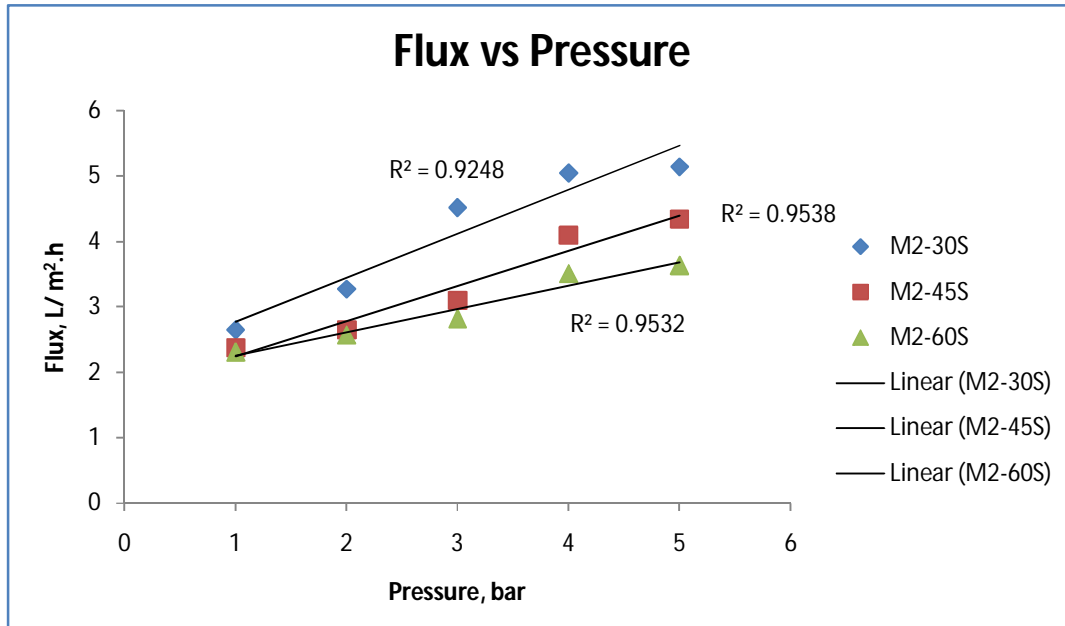


Figure 4.3: Graph Flux vs. Pressure of three different Polyester membranes

Figure 4.3 above graphically show that the gradient or water permeability decrease following the increasing immersion time during the interfacial polymerization process. M2-30S membrane has slightly increased of permeability compared with uncoated membrane maybe due to the error of time measuring for uncoated membrane. From the graph gradient, permeability for M2-30S membrane is $6.7475 \text{ Lm}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$ which is higher compare to the M2-45S and M2-60S which is $5.3767 \text{ Lm}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$ and $3.5857 \text{ Lm}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$. The range of values obtained were well within the range of values reported previously for NF membranes available commercially which is between $1.33 \text{ Lm}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$ and $50.50 \text{ Lm}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$ (A. W. Mohammad *et. al*, 2003). The different value of water permeability for each membrane may be due to the layer thickness on the membrane are different because according to Baker (2000), flux is inversely proportional to thickness of the membrane where the thickness of the membrane will give a lower flux. So, it can say that the layer of the M2-60S is denser than M2-30S and M2-45S.

4.3 REJECTION

Dye removal processes were conducted at pressure between 100kPa (1bar) to 500kPa (5bar). Each final concentration of MB dye solution at different pressure was taken to determine the percentage of dye rejection. With the help of Figure 4.1 the standard curve of MB, the concentration of filtrate can be determined from ABS value obtain from the spectrophotometer. This process determined whether the pressure will affect the concentration of the filtrate. Rejection is calculated using Eq. 2.2

$$R = \left(1 - \frac{C_f}{C_p}\right) \times 100 \quad (2.2)$$

Table 4.4: Methylene Blue Rejection

Membrane	Pressure, bar	Final Concentration, mg/L	% Rejection
M2-30S	5	12.13	51.48
	4	12.52	49.92
	3	13.13	47.48
	2	13.65	45.40
	1	14.57	41.72
M2-45S	5	11.04	55.84
	4	11.53	53.88
	3	11.57	53.72
	2	13.32	46.72
	1	14.17	43.32
M2-60S	5	10.97	56.12
	4	12.03	51.88
	3	12.54	49.84
	2	13.30	46.80
	1	14.06	43.76

Table 4.5 show the summary of the rejection carried out with Methylene Blue dye solution at initial concentration of 25mg/L using all three membranes prepared. Each final concentration of filtrate MB dye solution at different operating pressure was measured and plot as Figure 4.4.

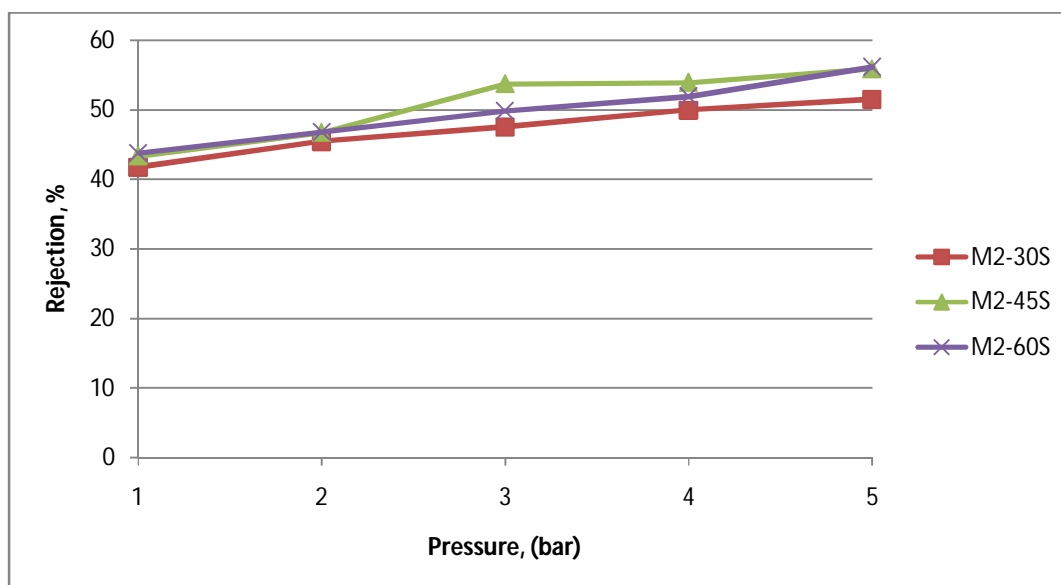


Figure 4.4: Effect of Operating Pressure on Rejection

As shown in the Figure 4.4, the rejection increased as the operating pressure increased. While at the same pressure, for example at 5 bars, the rejection of dyes is higher for M2-60s membrane compared to the other two membranes. So, it can conclude that when the immersion time of TEOA monomer increased the rejection also increase because the rejection of an uncharged solute such as dye is affected mainly by pore size as well as flux of the membrane (A.W. Muhammad et. Al 2003). The longer of reaction or immersion time taken, the smaller pore size as well as denser layer produced on surface of support PES membrane which result the lower flux and increased of filtration performance.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Based on the findings from this study, by using Polyester NF membrane which prepared through interfacial polymerization, the dyes concentration can be reduced. Besides that, the variation of reaction time in interfacial polymerization technique improved the filtration performance of the membrane. In terms of flux of the membrane, increasing the reaction time of in interfacial polymerization technique resulted in decreasing the flux and water permeabilities.

5.2 RECOMMENDATION

To improve filtration process or rejection of dyes, it is recommended to increase the TEOA concentration and reaction time in interfacial polymerization technique in order to improve the filtration performance of the membrane. For future research, upgrade the reaction time to minutes and increase the concentration of TEOA up to 4% or 6%.

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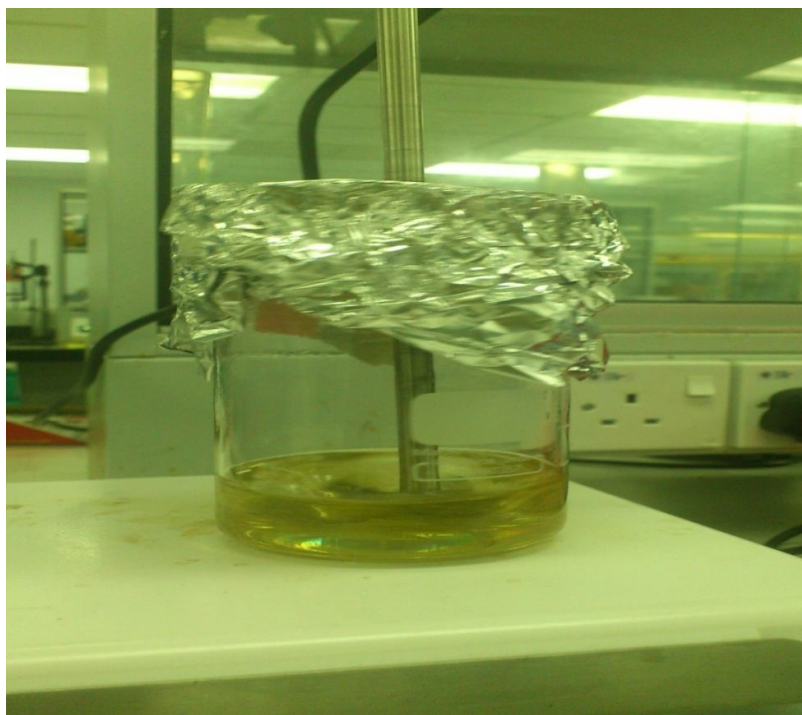
APPENDIX A**PICTURE OF BLENDING PROCESS**

Figure A-1: Yellow sticky solution

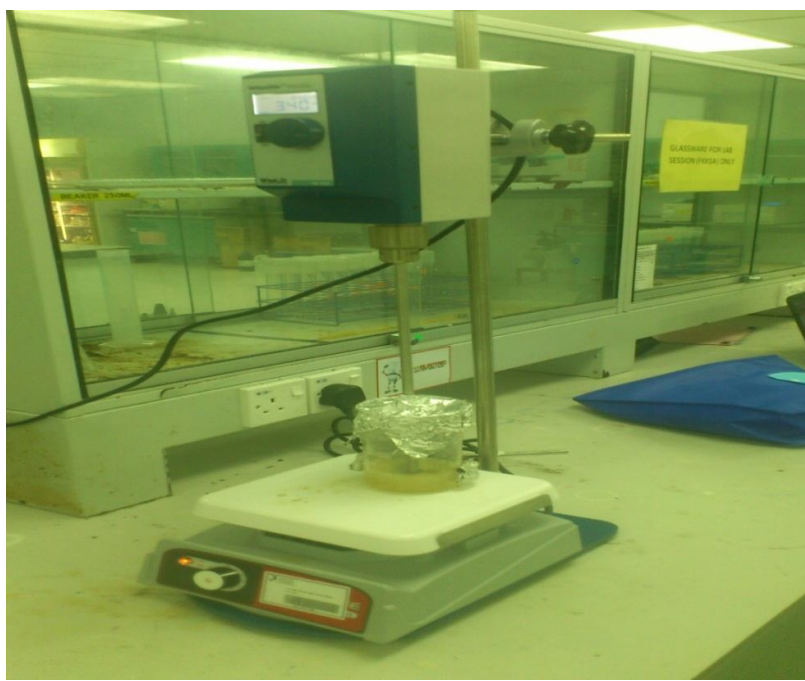
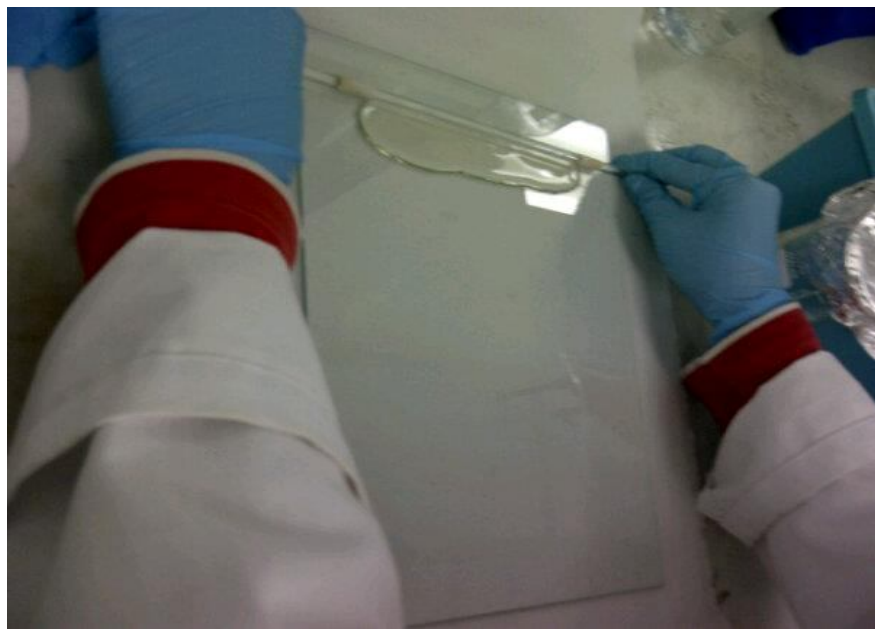
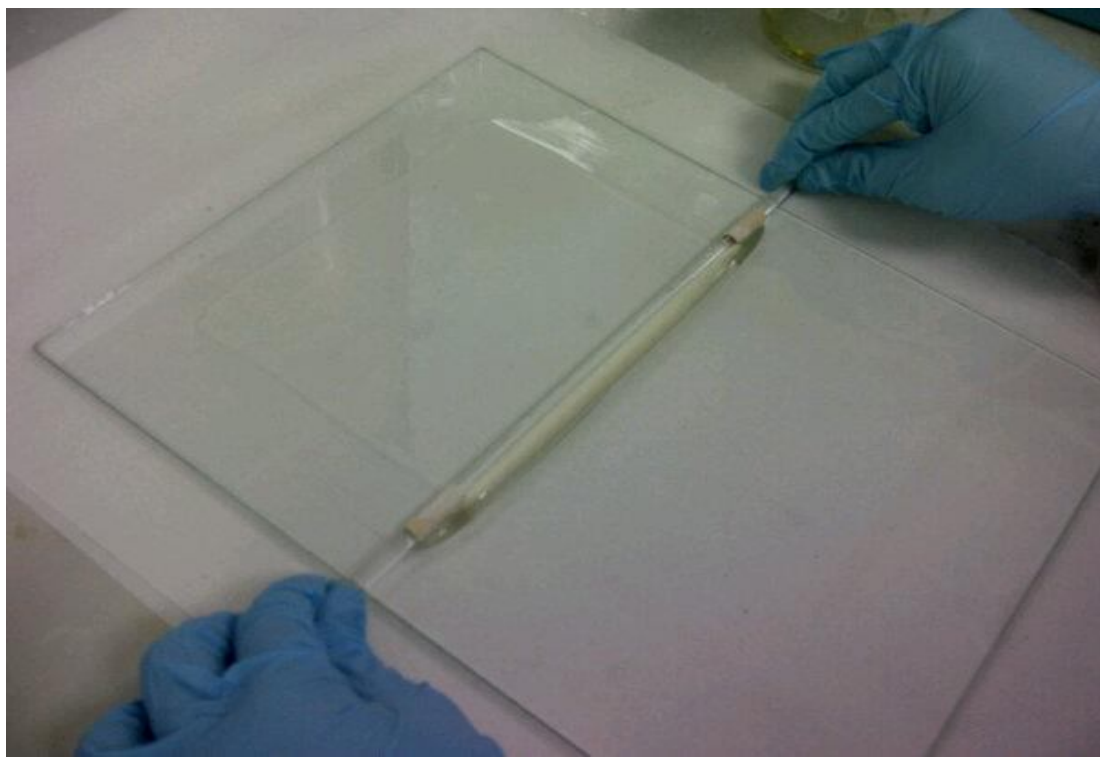


Figure A-2: Blending process

PICTURE OF CASTING PROCESS**Figure A-3: Casting process****Figure A-4: Sticky solution spread in glass plate**

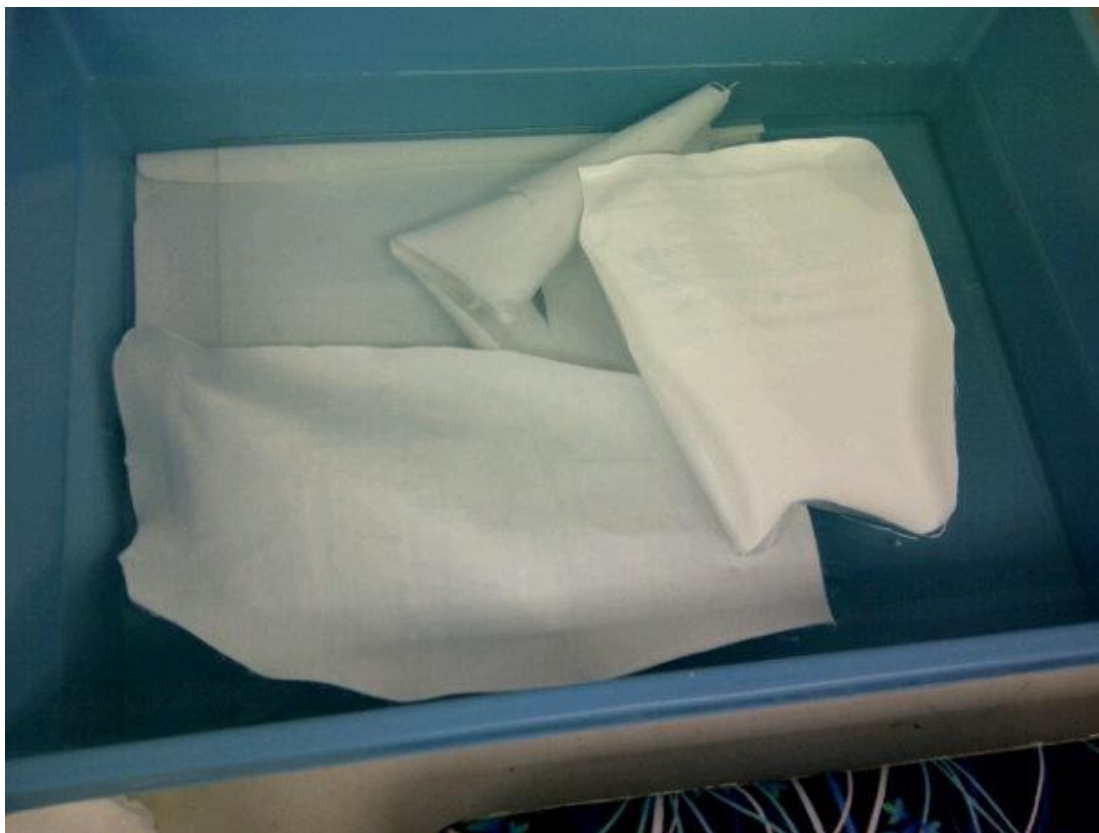


Figure A-5: Formation of membrane in water bath.

APPENDIX B

Flux calculation

i. Uncoated PES Membrane

Time taken at different pressure recorded

Pressure	Time, h
5	0.1221
4	0.1267
3	0.1389
2	0.1802
1	0.2085

Permeate volume = 0.020 L

Membrane area = 0.00287 m²

Using Eq. (2.1),

$$J = \frac{V}{A\Delta t}$$

Where, J = Membrane flux, L

A = Membrane area, m²

Δt = Filtration time, Δt

Flux for uncoated PES membrane is

Pressure	Time, h	Flux, L/ m ² .h
5	0.1221	57.0732278
4	0.1267	55.00111377
3	0.1389	50.17020241
2	0.1802	38.6717043
1	0.2085	33.42273916

ii. 2% TEOA – Polyester Membranes

Time taken at different pressure recorded

2% TEOA Membrane			
Pressure (bar)	Time, h		
	M2-30S	M2-45S	M2-60S
5	0.1357	0.1605	0.192
4	0.1383	0.1702	0.1988
3	0.1545	0.2247	0.2475
2	0.2132	0.2627	0.2715
1	0.2633	0.2935	0.3022

Permeate volume = 0.020 L

Membrane area = 0.00287 m²

Using Eq. (2.1),

$$J = \frac{V}{A\Delta t}$$

Where, J = Membrane flux, L

A = Membrane area, m²

Δt = Filtration time, Δt

Flux for 2% TEOA – Polyester Membranes is

Pressure	Flux, L/ m2.h		
	M2-30S	M2-45S	M2-60S
5	51.35328751	43.4183247	36.29500581
4	50.38786056	40.94383734	35.05352674
3	45.10447324	31.01308907	28.15612572
2	32.68593394	26.52699321	25.66718643
1	26.4665443	23.7432406	23.05969926

Final concentration dye calculation

ABS value after dye removal process,

Pressure, bar	ABS Value		
	M2-30S	M2-45S	M2-60S
1	1.753	1.699	1.685
2	1.629	1.585	1.582
3	1.560	1.350	1.480
4	1.478	1.345	1.412
5	1.425	1.279	1.269

By using straight line equation of MB standard curve,

$$Y = 0.1329x - 0.1858$$

Final concentration of dye, x is

Pressure, bar	Concentration, mg/L		
	M2-30S	M2-45S	M2-60S
1	14.57	14.17	14.06
2	13.65	13.32	13.3
3	13.13	11.57	12.54
4	12.52	11.53	12.03
5	12.13	11.04	10.97

Rejection calculation

From final concentration of dye calculated,

Rejection can be determined from Eq. (2.2)

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100$$

Where, R = Rejection percentage

C_p = Concentration of permeate

C_f = Concentration of feed

Pressure	% Rejection		
	M2-30S	M2-45S	M2-60S
1	41.7266068	43.3338844	43.750586
2	45.4173924	46.727026	46.8163192
3	47.471136	53.72166	49.852288
4	49.9118168	53.870482	51.8762672
5	51.48933	55.8349324	56.1325764