OPTIMIZATION OF ADDITIVES
CONCENTRATION IN POLYMERIC MEMBRANE
FABRICATION
(LOW POLYMER CONCENTRATION)

CHEAH CHOON HEN

UNIVERSITI TUNKU ABDUL RAHMAN
OPTIMIZATION OF ADDITIVES CONCENTRATION IN POLYMERIC MEMBRANE FABRICATION (LOW POLYMER CONCENTRATION)

CHEAH CHOON HEN

A project report submitted in partial fulfillment of the requirements for the award of Bachelor of Engineering (Hons) Environmental Engineering

Faculty of Engineering and Green Technology
Universiti Tunku Abdul Rahman

September 2015
DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at Universiti Tunku Abdul Rahman (UTAR) or other institutions.

Signature : Cheah Choon Hen

Name : CHEAH CHOON HEN

ID No. : 10AGB02485

Date : 30 September 2015
I certify that this project report entitled “OPTIMIZATION OF ADDITIVES CONCENTRATION IN POLYMERIC MEMBRANE FABRICATION (LOW POLYMER CONCENTRATION)” was prepared by CHEAH CHOON HEN has met the required standard for submission in partial fulfilment of the requirements for the award of Bachelor of Engineering (Hons) Environmental Engineering at Universiti Tunku Abdul Rahman.

Approved by,

Signature : ___________________ Signature : ___________________

Supervisor : Mr. Wong Ling Yong Co-Supervisor : Dr. Ng Choon Aun

Date : ________________ Date : ________________
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I would like to thank everyone who had contributed to the successful completion of this project. I would like to express my gratitude to my research supervisor, Mr. Wong Ling Yong and research co-supervisor, Dr. Ng Choon Aun for their invaluable advice, guidance and their enormous patience throughout the development of the research.

In addition, I would also like to express my gratitude to my loving family and friends who had helped and given me encouragement and strength to complete the project. With their constant loving and support, this project was successfully completed.

Last but not least, I would also like to thank to our lab assistants, Mr. Muhammad Zakuan Ariff Bin Mohd Zaidi, Mr. Cheah Chew Keat, Cik Noor Hazreena Binti Noor Izahar, Ms. Mirohsha a/p Mohan and Mr. Voon Kah Loon for providing plenty of assistance in the lab during the experiments.
OPTIMIZATION OF ADDITIVES CONCENTRATION IN POLYMERIC MEMBRANE FABRICATION (LOW POLYMER CONCENTRATION)

ABSTRACT

Recent developments in technology have resulted in breakthroughs in wastewater treatment and reclamation for water reuse. Membranes technology may be an option when they enable the removal of contaminants. In this study, the potential of powdered activated carbon (PAC) as an additive in polyethersulfone (PES) membrane fabrication to improve the performances of membrane was determined. Three membranes with different additive concentrations (0 wt. %, 5 wt. %, and 11 wt. %) were tested to find out the flux produced and rejection to salt water by each membrane. The results obtained showed that the membrane with 11 wt. % of PAC produced the most flux than others. Flux produced was in the range between $5.146 \times 10^{-5}$ m$^3$/m$^2$/s and $6.549 \times 10^{-5}$ m$^3$/m$^2$/s. Besides, membrane with 11 wt. % of additives also had the highest rejection for salt water, which reject 76% and 79%. Apart from that, morphology of membrane with and without additive was studied using Scanning Electron Microscopy (SEM). Structure of membrane has been shown that membrane with PAC was having more macro voids formation than without PAC. Furthermore, tensile testing for the membranes was also studied. The ultimate tensile strength (UTS) of the membrane without PAC showed a higher readings which was 2.261 MPa. Meanwhile, membrane with 5 wt. % have lower UTS compare to 0 wt. %.

Yet, the UTS was increase as there is more PAC was added into the membrane as shown in membrane with 11 wt. %.
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<thead>
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<th>Description</th>
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<tbody>
<tr>
<td>AC</td>
<td>Activated Carbon</td>
</tr>
<tr>
<td>COD</td>
<td>Chemical Oxygen Demand</td>
</tr>
<tr>
<td>DMAc</td>
<td>Dimethylacetamide</td>
</tr>
<tr>
<td>DMF</td>
<td>Dimethylformamide</td>
</tr>
<tr>
<td>DMSO</td>
<td>Dimethylsulfoxide</td>
</tr>
<tr>
<td>EPSs</td>
<td>Extracellular polymer substances</td>
</tr>
<tr>
<td>GAC</td>
<td>Granular Activated Carbon</td>
</tr>
<tr>
<td>MBR</td>
<td>Membrane Bioreactor</td>
</tr>
<tr>
<td>MF</td>
<td>Microfiltration</td>
</tr>
<tr>
<td>NaCl</td>
<td>Sodium Chloride</td>
</tr>
<tr>
<td>NF</td>
<td>Nanofiltration</td>
</tr>
<tr>
<td>NMP</td>
<td>N-Methyl-2-Pyrrolidinone</td>
</tr>
<tr>
<td>PAC</td>
<td>Powdered Activated Carbon</td>
</tr>
<tr>
<td>PES</td>
<td>Polyethersulfone</td>
</tr>
<tr>
<td>RO</td>
<td>Reverse Osmosis</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>TMU</td>
<td>Tetramethylurea</td>
</tr>
<tr>
<td>UF</td>
<td>Ultrafiltration</td>
</tr>
<tr>
<td>UTS</td>
<td>Ultimate Tensile Strength</td>
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</table>
CHAPTER 1

INTRODUCTION

1.1 Background of Study

Wastewater is spent or used water from domestic and industrial activities that contains enough harmful materials to damage the water’s quality. According to Adamu and Balarabe (2014), the major sources of the natural water pollution load are constituted from the effluents generated from these activities. Wastewater may contain contaminants like chemicals, human waste, dirt, or oil. When wastewater is untreated, it may cause serious harm to the environment and threaten human health.

Therefore, wastewater treatment take an important role in order to treat the wastewater. It is a process which partially remove and change the solids in wastewater by decomposition from highly complex, decayable, organic solids to mineral or stable organic solids (Sonune & Ghate, 2004).

Nowadays, the increase in environmental protection sensitivity compare to last decade has led to the revolution of new technologies for wastewater treatment (Stephen & Valerie, 2000). Once considered a feasible technology only for desalination, membrane processes are increasingly employed especially in filtration process for removal of bacteria, microorganisms, particulate material, and natural organic material, which can contribute color, tastes, as well as odors to the water (National Drinking Water of
Clearinghouse, 1999). According to Amjad (1993), the membrane filtration can be classified into microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO).

Apart from wastewater treatment, membranes are also being widely used in fields such as manufacture of food, devices for energy conversion like fuel cells, devices for medical like blood oxygenators, and also the productions of base chemicals (Hugo, 2015). Contemporarily, conventional technologies are being integrated with the membrane-separation processes as hybrid membrane systems from time to time in order to reduce energy consumption and minimize the environmental impact (Rajindar, 2015).

1.2 Problem Statement

Nowadays, there are more and more municipal wastewater treatment facilities are using membrane technology in their treatment. Membranes become the best option when researchers found out that it enable the removal of contaminants that other technologies cannot. Apart from that, they are also more economical and require less land area than other technologies. Membranes commonly are being used to remove organic compounds, dissolved species, nitrogen species, phosphorus, suspended solids, colloidal, as well as human pathogens like bacteria, and viruses (CDM Smith, 2015).

As membrane technology take an important role in water treatment nowadays, continual improvement is needed to increase the performance of the membrane. For example, there are high flux production but low in rejection rate in low polymer concentration membrane. In this study, hybrid membrane with different concentrations of PAC were fabricated and performance of each membrane was tested.
1.3 **Project Objectives**

In order to obtain the goal of the project, objectives need to be stated clearly. This project objectives include:

i. To fabricate membrane with different concentration of PAC as additive.

ii. To study the effect of PAC in membrane fabrication in term of flux production, salt rejection and tensile strength.

iii. To characterize the membrane structure and membrane properties of produced membrane.

1.4 **Project Scope**

The scope of this study are listed below:

i. To prepare membrane with formulation: PES/ NMP/ PAC.

ii. To fabricate membrane with different PAC concentration by using Membrane Auto Casting Machine.

iii. To determine the performance of membrane by using Dead End Membrane Test Rig.

iv. To study and characterize the membrane structure and membrane properties by using SEM.

v. To study the tensile strength of membrane by using Light Weight Tensile Testing Machine.
CHAPTER 2

LITERATURE REVIEW

2.1 Domestic Wastewater

Domestic wastewater is wastewater which derived principally from kitchen, toilet and bathroom. It may contains human waste, food waste, soap, detergent as well as oils and greases (Molloy, 2012) which suspended in range from 150 to 200 liters of wastewater daily for each person in a household (Department of Water Government of Western Australia, 2010). Domestic wastewater is the combination of grey water and municipal waste and has certain strength and pollution ability which may contain pathogens (Carpenter, et al., 1998). What is more, it has also been reported to constitute suspended solids and also solids in colloidal forms (Awadallah & Yousry, 2012). According to Carpenter, et al. (1998), the population growth in recent decades has increases the use of domestic water use and it influences water quality and aquatic ecosystem functions.

2.2 Wastewater Treatment Plant

Wastewater treatment plant is a plant build in order to treat the wastewater or to improve the effluent quality. The treatment processes are designed to achieve improvements in the quality of the wastewater. The various treatment processes can reduce the biodegradable
organics, suspended solids, pathogenic bacteria as well as nutrients (The World Bank Group, 2015).

The wastewater treatment can be categorized into three processes which are primary, secondary and tertiary treatments. Primary treatment, also known as mechanical treatment, is used to remove gross, suspended and floating solids from raw sewage through screening and sedimentation (Nathanson, 2014).

Next, the secondary treatment, also known as biological treatment, is used to removes the dissolved organic matter that escapes previous treatment by using microbes to consume the organic matter as food, and converting it into carbon dioxide, water, and energy for their own growth and reproduction (Malik, 2014).

Last but not least, the tertiary treatment, also known as advanced treatment, additional treatment. It can remove more than 99 percent of all the impurities from sewage, producing an effluent of almost drinking-water quality (The World Bank Group, 2015).

2.3 Membrane Technology

Membrane separation technology nowadays has replaced the traditional water treatment methods like physical separation techniques, biological and chemical treatments. Selectively permeable barriers with pores sized to permit the passage of water molecules, but small enough to retain a wide range of particulates and dissolved compounds are used in the membrane separation technology (European Union, 2010).
2.4 Introduction to Membrane Process

Membrane is a permselective barrier or interphase between two phases as shown in Figure 2.1. Depends on the different separation principles as well as the mechanisms, there are many different kinds of membrane process. Besides, different membrane processes can cover different size ranges of particles or even molecules (Marcel, 1996). The benefits of membrane processes are listed in the Table 2.1.

![Figure 2.1: Schematic representation of a two-phase system separated by a membrane (Marcel, 1996)](image)

Table 2.1: Benefits of Membrane Process (Sulzer Chemtech, n.d.)

<table>
<thead>
<tr>
<th>Benefits</th>
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<tr>
<td>– Improved economics through</td>
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<tr>
<td>• Low investment cost</td>
</tr>
<tr>
<td>• Optimized operating cost</td>
</tr>
<tr>
<td>– Reduction of product degradation</td>
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<tr>
<td>– Design of novel process solutions</td>
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</table>
2.5 Membrane Classification

Transport of selected species through the membrane is achieved by applying a driving force across the membrane. There are a broad classification of membrane separations in the sense of how a material was transported across a membrane. The flow of material across a membrane has to be kinetically driven and the forces can be pressure, concentration, electrical potential or temperature (Keith, et al., 1996). Membrane process can be classified as Microfiltration (MF), Ultrafiltration (UF), Nanofiltration (NF) and Reverse Osmosis (RO) as shown in Figure 2.2 and their properties are listed Table 2.3.

Figure 2.2: Application Size Range of Membrane Filtration Process (Keith, et al., 1996)
Table 2.2: Correlation of Membrane Features with Ranges of Separation
(Brett & Jabez, 2004)

<table>
<thead>
<tr>
<th>Properties</th>
<th>Types of Membranes</th>
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<tr>
<td></td>
<td>Microfiltration (MF)</td>
</tr>
<tr>
<td>Thickness</td>
<td>10 - 150 μm</td>
</tr>
<tr>
<td>Pore Size</td>
<td>100 – 1000 nm</td>
</tr>
<tr>
<td>Pressure</td>
<td>&lt; 2 bars</td>
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</table>

2.5.1 Microfiltration (MF)

Microfiltration (MF) is a process which used to separate micron-sized particles from fluids (Winston Ho & Kamalesh, 2001). It happened when a pressure-driven flow through a thin polymer film with uniform pore size and high pore density or other filter medium (Keith, et al., 1996). Generally, MF is defined to be the filtering of a suspended solids, bacteria (Brett & Jabez, 2004), suspension which containing colloidal or fine particles (Winston Ho & Kamalesh, 2001) with linear dimensions in range around 100 nm to 1000 nm (Brett & Jabez, 2004) as shown in Table 2.3. There are some limitations for MF, one of it is that disinfection is required for viral inactivation (National Drinking Water Clearinghouse, 1999).

Figure 2.3: Microfiltration Performance Characteristics
(Solomon Jebamani, et al., 2009)
2.5.2 Ultrafiltration (UF)

For ultrafiltration (UF), it involves the pressure-driven separation of materials from water using a membrane pore size of approximately 10 nm to 200 nm. It cover the region between MF and NF (Keith, et al., 1996). Materials larger than the pore size rating like suspended solids, bacteria and viruses are retained by the membrane and can be concentrated or separated while water, monovalent ions and multivalent ions will still pass through the membrane. As same with MF, UF have the same limitations which are disinfection is required for viral inactivation (National Drinking Water Clearinghouse, 1999).

![Figure 2.4: Ultrafiltration Performance Characteristics](Solomon Jebamani, et al., 2009)

2.5.3 Nanofiltration (NF)

Nanofiltration (NF) membranes have a nominal pore size of approximately 1 nm to 10 nm. Pushing water through these smaller membrane pores requires a higher operating pressure than either MF or UF, which is approximately 5 bars to 35 bars (Solomon Jebamani, et al., 2009). These membrane can remove virtually all those suspended solids, viruses, bacteria. However, it can only partially remove multivalent ions while water and monovalent ions will still pass through the membrane. Compare to MF and UF, More energy is required for NF than MF or UF, which has hindered its advancement as a treatment alternative. Another limitation of NF is that disinfection is required under regulation, and recommended as a safety measure and residual protection (National Drinking Water Clearinghouse, 1999).
2.5.4 Reverse Osmosis (RO)

Reverse Osmosis (RO) systems are compact, simple to operate, and require minimal labor, making them suitable for small systems. RO can also effectively remove suspended solids, bacteria, viruses, multivalent ions or even monovalent ions and only allow water to pass through the membrane due to its extremely small pore sizes which are at the range from 0.1 nm to 1 nm. RO can function effectively when used in multiple units of membranes. Water passing through series of RO membrane can produce water with near zero contaminant concentrations. Disinfection is recommended to ensure the safety of water (National Drinking Water Clearinghouse, 1999). Furthermore, due to the small pore size of RO membranes, the membranes are prone to fouling.
2.6 Activated Carbon

Activated carbon (AC) has been proven by Crittenden, et al. (1993) and Cook, et al. (2001) in which it is an effective adsorbent in remove a different types of organic and inorganic pollutants from either aqueous or gaseous media. It is widely used due to its exceptionally high surface area ranges which is from 500 to 1500 m$^2$/g, wide spectrum of surface functional groups, and also its well-developed internal microporosity (Abdel Hameed, et al., 2013). AC is a form of carbon which has been proven to be an effective adsorbent for the removal of a wide variety of organic as well as inorganic pollutants from aqueous or gaseous media (Foo & Hameed, 2009). It is widely used because it has a large surface area available which the ranges are from 500 m$^2$/g to 1500 m$^2$/g, well-developed internal microporosity, and wide spectrum of surface functional groups for adsorption or chemical reaction after it has been processed (Rivera-Utrilla, et al., 2011). It is often used as a filter in water treatment systems. Water is directed downwards through a stationary bed of activated carbon, organic material is left in order to accumulate at the top of the bed (Faliq Alit, 2010). According to Siti Khadijah, et al. (2012), there are two manufactured techniques which are through physical and also chemical activations to produce AC.

For physical activation, gasses with either oxidation or carbonization process or combination of both of these processes can produce AC. According to Yee Jun, et al. (2009), the carbonized materials were exposed to oxidizing atmosphere like steam, carbon dioxide or oxygen at temperatures between 600 - 1200 °C in oxidation process. On the other hand, in carbonization process, the carbon content is heated up to 600-900 °C only in an atmosphere of inert gases like nitrogen or argon.

For chemical activation process, raw materials are macerated with acid, strong base or salt before they are being carbonized at low temperatures between 450 – 900 °C. Due to chemical activation process offers a minimum time and low temperature for activating material compare to physical activation, chemical activation process is a technique which is more preferable (Yee Jun, et al., 2009).
There are different forms of AC. The most common and popular forms are powdered activated carbon (PAC) and granular activated carbon (GAC). It will be further discussed in section 2.6.1 and 2.6.2.

2.6.1 Powdered Activated Carbon (PAC)

Powdered Activated Carbon (PAC) is particularly a powder or fine granules with less than 1.0 mm size in average and has a large surface area to the volume ratio. Generally, it was used in raw water intakes, rapid mix basins, clarifiers, and gravity filters (Siti Khadijah, et al., 2009). According to H. Ma, et al. (2000), PAC has an ability to adsorb primarily those organic compounds with lower molecular weight and shows more affinity towards the hydrophobic fractions. A report reported by Jacangelo, et al. (1995) stated that PAC could adsorb organic materials and able to minimize membrane fouling. Furthermore, the PAC cake layer which formed on the membrane surface could avoid irreversible fouling of the membrane as well as slow down fouling.

With the combination of PAC and membrane filtration process, adsorption was used as a hybrid system in order to remove the organic materials as well as improving permeate flux. Generally, PAC is used in the pretreatment step before the membrane operation or combination of both PAC and membrane in the same tank. However, membrane fouling is still an existing problem in applying a PAC-MF system for wastewater even though there are improvements in membrane performance nowadays (Ramesh Thiruvenkatachari, et al., 2006).

Figure 2.7: Powdered Activated Carbon (PAC)
(European Operation of Calgon Carbon Corporation, 2015)
2.6.2 Granular Activated Carbon (GAC)

Granular activated carbon (GAC) is a good adsorbent medium like PAC as it has high surface area to surface area to volume ratio (CARBTROL® Corporation, 1992). According to Baruth (2005), its adsorption surface area is normally from 650 m\(^2\)/g to 1000 m\(^2\)/g. And according to Robert H. Perry, et al, (1997), GAC is made of tiny clusters of carbon atoms stack upon one another, and is produced by carbon source like wood, ignite, or coal in the absence of air which a high carbon content material. However, compare to PAC, it has smaller surface area to the volume ratio. It is preferred in gases and vapors adsorption (Siti Khadijah, et al., 2009).

Figure 2.8: Granular Activated Carbon (GAC)
(European Operation of Calgon Carbon Corporation, 2015)
2.7  Polymer and Solvent for Membrane Fabrication

In this study, polyethersulfone (PES) was used as a polymer and N-Methyl-2-Pyrrolidinone (NMP) was used as a solvent in membrane fabrication.

2.7.1  Dope Solution

Dope solution is a homogeneous polymer solution which is prepared by mixing a polymer, solvent and also other ingredients or additives (King Abdullah University of Science and Technology, 2015).

2.7.2  Polyethersulfone (PES)

Polyethersulfone (PES) is a common material that used as a polymer for UF membrane. It has high resistance to heat and combustibility, low smoke emission (Plastics International, n.d.), good chemical resistance and high mechanical properties (Ping, et al., 2010). Nevertheless, according to Kim, et al. (1999), pure PES membrane is vulnerable to fouling in water treatment due to the PES hydrophobic character. Membrane fouling not only limit the UF membrane performance as well as reducing its working life, but also increase the cost of operating it. According to Ping, et al. (2010), some hydrophilic cellulose fibrils can be blended with PES in order to improve the hydrophilicity of PES membranes.
2.7.3 N-Methyl-2-Pyrrolidinone (NMP)

N-Methyl-2-Pyrrolidinone (NMP) is a hygroscopic colorless to slightly yellow liquid (Office of Pollution Prevention and Toxics, 2012) with a mild amine odor (Akesson, 2001). The physical properties are shown in Table 2.3.

Table 2.3: Physical Properties of N-Methyl-2-Pyrrolidinone (NMP)
(Centers for Disease Control and Prevention, 2014)

<table>
<thead>
<tr>
<th>Properties</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boiling Point</td>
<td>202 °C</td>
</tr>
<tr>
<td>Melting Point</td>
<td>-24 °C</td>
</tr>
<tr>
<td>Relative density (water = 1)</td>
<td>1.03</td>
</tr>
<tr>
<td>Solubility in water</td>
<td>Very good</td>
</tr>
<tr>
<td>Vapour pressure, Pa at 25 °C</td>
<td>66</td>
</tr>
<tr>
<td>Relative vapour density (air = 1)</td>
<td>3.4</td>
</tr>
<tr>
<td>Auto-ignition temperature</td>
<td>270 °C</td>
</tr>
</tbody>
</table>

According to Wayne, et al. (1991), the width of the miscibility regions indicated that solvent dissolution power for PES ranks. The ranks were shown as followed: N-Methyl-2-Pyrrolidinone (NMP) > Tetramethylurea (TMU) > Dimethylacetamide (DMAc) > Dimethylsulfoxide (DMSO) > Dimethylformanode (DMF).
2.8 Membrane Bioreactor (MBR)

Membrane bioreactor (MBR) is a technology which combine a direct solid-liquid separation by membrane filtration and the biological degradation process by activated sludge. It allows complete physical retention of bacteria flocs and almost all suspended solids within the bioreactor by using MF and UF membrane technology (Pierre, et al., 2006). Additionally, MBR also has high effluent quality and volumetric loading, less sludge production, small footprint and reactor requirements, as well as good disinfection capability (Judd, 2006).

However, the MBR filtration performance ineluctably decreases with filtration time. This is due to the deposition of soluble and particulate materials onto and into the membrane from time to time, attributed to the interactions between activated sludge components and the membrane (Pierre, et al., 2006). Besides, other disadvantages include high capital and operating cost like membrane cleaning as well as high energy cost as air scouring needed in order to control bacterial growth on membranes. Last but not least, the waste sludge from MBR may have low settling rate and causes the need of chemicals in order to produce biosolids acceptable for disposal (Hermanowicz, et al., 2006).
There are two main MBR configuration which are submerged and external separation membrane as shown in Figure 2.11. Both of the MBR systems are aerated at the bottom part of the bioreactor (Melin, et al., 2006).

![Figure 2.11: Configuration of MBR systems. (a) Submerged MBR, (b) Lateral flow MBR (Melin, et al., 2006)](image)

### 2.9 Membrane Fouling

Membrane fouling is a buildup of materials such as adsorbed molecules and deposited in the membrane surface (Peinmemann & Nunes, 2010). Some typical foulants included proteins, lipids, and bacteria. Fouling is a complex phenomenon which is difficult to localize the membrane fouling clearly. It can be due to one of the following mechanisms such as cake filtration (Yan-jun, et al., 2000), standard blocking (Labbe, et al., 1990), complete blocking (Weldring & van't Riet, 1988) and intermediate blocking (Pierre, et al., 2006) as shown in Figure 2.12 and their causes and descriptions are listed in Table 2.4.

Membrane fouling basically affected by few factors, for example concentration, pH and ionic strength, component interactions, pre-filtration and the removal of
aggregates, pore size of the membrane, as well as the porosity and pore size distribution of the membrane (Pierre, et al., 2006).

Figure 2.12: Fouling Mechanism (Radjenovic, et al., 2008)

Membrane fouling may cause flux decline. In other words, the decrease in permeation through a membrane during the filtration process. The flux decline is always caused by a decreasing force and increase in resistance (Franken, 2009).

Table 2.4: Description of Fouling Mechanism (Pierre, et al., 2006)

<table>
<thead>
<tr>
<th>Law</th>
<th>Physical cause</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cake filtration</td>
<td>Boundary layer resistance</td>
<td>Deposit of particles larger than the membrane pore size onto the membrane surface</td>
</tr>
<tr>
<td>Complete blocking</td>
<td>Pore blocking</td>
<td>Occlusion of pores by particles with no particle superimposition.</td>
</tr>
<tr>
<td>Standard blocking</td>
<td>Direct adsorption</td>
<td>Deposit of particles smaller than the membrane pore size onto the pore walls, reducing the pore size.</td>
</tr>
</tbody>
</table>
2.10 Extracellular Polymeric Substances (EPSs)

According to Sleytr (1997), the EPS matrix is generally from 0.2 to 1.0 μm thick. In some bacteria species the thickness of the EPS layer does not exceed values from 10 to 30nm. EPS compounds belong to such different classes of macromolecules (Czaczyk & Nyszka, 2007) as polysaccharides, proteins, nucleic acids, glycoproteins and phospholipids (Branda, et al., 2005).

2.11 Cross-Flow Filtration

Cross-Flow filtration, also known as tangential-pass filtration is a constant turbulent flow along the membrane surface prevents the accumulation of matter on the membrane surface. Tubes with a membrane layer on the inside wall of the tube are commonly used in the filtration process. The feed flow through the membrane tube has a high flow speed to create turbulent conditions and has a elevated pressure as driving force for the process (Ahsan Munir, 2006). Unlike dead-end filtration which will be discussed in 2.12, it does not built up cake layer uncertainly. Hence, relatively high fluxes may be maintained over prolonged time periods as shown in Figure 2.13 (Winston Ho & Kamalesh, 2001).
2.12 Dead-End Filtration

Dead-end filtration is the most basic form of filtration. It is a batch process where the accumulated retained particles on the membrane decreases the filtration capacity which is caused by clogging. The retained particles are accumulated on the surface of the membrane when the complete feed flow is forced through the membrane (Ahsan Munir, 2006). As times goes by, these retained particles will form a cake layer. The cake thickness formed results in increase resistance to filtration and decrease the permeate flux rate if the pressure drop is held constant as shown in Figure 2.14 (Winston Ho & Kamalesh, 2001).
2.13 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The information about the sample external morphology (texture) can be revealed by using the signals that derive from electron-sample interactions (Swapp, 2013).

Figure 2.14: Schematic of Dead-End Filtration (Winston Ho & Kamalesh, 2001)
CHAPTER 3

RESEARCH METHODOLOGY

3.1 Membrane Fabrication and Casting

For membrane fabrication, polyethersulfone (PES) was used as polymer, N-methyl-2-pyrrolidinone (NMP) was used as the solvent, and powder activated carbon (PAC) was used as an additive. PES polymer was shown in Figure 3.1 and PAC was shown in Figure 3.2.

Figure 3.1: Polyethersulfone (PES) used as Polymer
3.1.1 Dope preparation

The apparatus needed in dope preparation included:

Table 3.1: Apparatus Needed on Dope Preparation

<table>
<thead>
<tr>
<th>Apparatus</th>
<th>Apparatus</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spatula</td>
<td>Cooling pipe</td>
</tr>
<tr>
<td>Condenser</td>
<td>Mercury Thermometer</td>
</tr>
<tr>
<td>Stopper</td>
<td>Thermometer Adapter</td>
</tr>
<tr>
<td>Heating Mantle</td>
<td>Glass Rod</td>
</tr>
<tr>
<td>Stirrer</td>
<td>3-neck Flask</td>
</tr>
<tr>
<td>Beakers</td>
<td>Reagent Bottles</td>
</tr>
<tr>
<td>Retort Stand</td>
<td>Aluminum Foil</td>
</tr>
</tbody>
</table>
In order to remove any moisture in the apparatus before the experiment started, some apparatuses such as beaker, spatula, 3-neck flask, and reagent bottle were placed in the oven at 60 °C for 24 hours. After 24 hours, the apparatus were set up as shown in Figure 3.3 in a fume hood as shown in Figure 3.4.

**Figure 3.3: Dope Preparation Apparatus Set Up**

**Figure 3.4: Fume Hood**
As this experiment was to study the effect of additives incorporated into polymeric membranes with low polymer concentration and investigate the effectiveness of the membranes produced in treating the domestic wastewater, the weight of PAC was varied. The ratio of PAC was calculated by using the method below. Other calculations were shown in Appendix A.

For membrane of 12 g of polymer with 5 % of PAC,

\[
\text{PAC concentration needed} = \text{Polymer concentration} \times \text{percentage of PAC} \\
= 12 \text{ g} \times 5\% \\
= 0.60 \text{ g}
\]

\[
\text{PES concentration needed} = \text{Polymer concentration} - \text{PAC concentration} \\
= 12 \text{ g} - 0.60 \text{ g} \\
= 11.40 \text{ g}
\]

Thus, in 100 g of dope preparation,

PES: NMP: PAC = 11.40 g: 88.00 g: 0.60 g

The ratio of PAC to PES were calculated and listed in Table 3.2. NMP was fixed in 88.00 g and used as solvent.

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Additive, PAC (%)</th>
<th>Additive, PAC (g)</th>
<th>Polymer, PES (g)</th>
<th>Solvent, NMP (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PES-PAC 0 wt. %</td>
<td>0</td>
<td>0.00</td>
<td>12.00</td>
<td>88.00</td>
</tr>
<tr>
<td>PES-PAC 5 wt. %</td>
<td>5</td>
<td>0.60</td>
<td>11.40</td>
<td>88.00</td>
</tr>
<tr>
<td>PES-PAC 11 wt. %</td>
<td>11</td>
<td>1.32</td>
<td>10.68</td>
<td>88.00</td>
</tr>
</tbody>
</table>
First of all, 88.00 g of NMP was poured into a beaker and measured by using an analytical balance. Subsequently, 11.40 g of PES was measured by using an analytical balance. After that, NMP was poured into the 3-neck flask and heated by using a heating mantle at temperatures between 50 °C and 60 °C. At the same time, the solvent was stirred by using a magnetic stirrer which was put into the 3-neck flask.

Once the temperature become stable, PES was added slowly into the flask. In other words, after one spatula of polymer was added into the 3 neck-flask, then the another spatula of polymer was added when the previous one was completely dissolved in the flask. When all the PES was completely dissolved in the solvent, the heat was turned off and the dope was allowed to cool down to room temperature. After that, the dope was poured into a reagent bottle. PAC was then measured accordingly and added into the dope prepared. Next, the mixture was placed in the sonicator bath as shown in Figure 3.5 for 8 hours until PAC and the mixture became homogeneous mixture as shown in Figure 3.6.

Figure 3.5: Sonicator Bath
3.1.2 Membrane Fabrication

The membranes were casted using the dry/wet casting technique by using a membrane auto casting machine as shown in Figure 3.7 below. The dope solution was poured slowly and evenly onto a smooth glass plate.
Membrane spacing which is the thickness of the membrane was set as 10 micrometer and was checked beforehand using a knife gap as shown in Figure 3.8. After the membrane was cast, the membrane was then submerged in a water bath immediately until the thin layer membrane totally formed. The membrane will proceed to coagulant bath which the membrane was submerged in a container for 24 hours (washing bath) as shown in Figure 3.9.

![Figure 3.8: Knife Gap](image)

![Figure 3.9: Thin Film Membrane in Coagulant Bath](image)
3.1.3 Post Treatment

The membrane was placed in a container containing methanol at least 8 hours for post treatment. During this stage, excess solvent in the membrane will be removed. The membrane was then hanged and allowed to dry at room temperature for 24 hours. After that, the membrane was cut to a diameter of 47 mm and ready to be tested.

3.2 Membrane Performance Testing

There were three analysis needed to be carried out to evaluate the performance of membrane in this study and those are measurement of pure water flux, salt solution flux, and salt rejection rate.

The tests was carried out with Dead End Membrane Test Rig as shown in Figure 3.9. The membrane sheets were cut out into circular shape with diameter of 47 mm. In this test, three membranes with different concentrations of PAC were tested.

Figure 3.9: Dead End Membrane Test Rig
Pure water flux was measured by using distilled water and it was measured under different pressure which are 0 bar, 0.1 bar and 0.2 bar in order to determine the water permeability. Pure water flux was calculated by taking the reading of time to collect 5 mL of permeate for the membranes using dead end membrane test rig.

Salt (NaCl) solution flux and rejection were carried out using dead end membrane test rig also. The flux and rejection of salt was measured by using standard 0.01M of NaCl for different feed solutions. 0.01M of NaCl was passed through the membrane under pressure of 0 bar, 0.1 bar and 0.2 bar in order to test the rejection performance. The time to collect 15 mL of solution was taken and the flux of the salt solution was measured. The volume collected was then taken to measure the conductivity in order to compare the conductivity before and after the salt solution being filtered. Be reminded that the apparatus need to be rinsed with distilled water and washed after each run was being done in order to remove the deposition on the surface of the membrane.

The flux calculation was shown in equation below:

$$\text{Flux, } J_\nu = \frac{\text{Volume permeation rate (} V\text{)}}{\text{Membrane area (} A\text{)} \times \text{time (} t\text{)}}$$

Where,

- $J_\nu$ = permeate flux of pure water or salt aqueous solution flux (m$^3$/m$^2$/s)
- $V$ = volume of permeate solution collected (m$^3$)
- $A$ = effective area of membrane (m$^2$)
- $T$ = time (s)

The rejection characteristics of membranes were described by observed rejection, $R_{\text{obs}}$ and real rejection, $R_{\text{real}}$. The rejection calculation was shown in equation below:

$$\ln \frac{1 - R_{\text{obs}}}{R_{\text{obs}}} = \ln \frac{1 - R_{\text{real}}}{R_{\text{real}}} + \frac{J_\nu}{k}$$
\[ R_{\text{obs}} = 1 - \frac{C_p}{C_b} \]
\[ R_{\text{real}} = 1 - \frac{C_p}{C_w} \]

Where,
- \( C_p \) = Salt concentration in permeate
- \( C_b \) = Salt concentration in bulk
- \( C_w \) = Salt concentration in wall

### 3.3 Dilution of Salt

The stock solution was prepared by dissolving the calculated amount of salt (in gram) in the required volume of dilution. The stock solution was prepared by using the equation below:

Molecular weight of NaCl = 58.4428 g/mol to obtain 0.01 M using 1000 mL of distilled water.

\[ m = \frac{0.01 \times 1000 \times 58.4428}{1000} \]

Where \( m = 5.84428 \) g of NaCl to be diluted in 1000 mL volumetric flask.

The dilution for each salt can be obtained by using the equation below and calculation was shown in Appendix B:

\[ m = \frac{MV \times MW}{1000} \]
Where,

\[ m = \text{weight of chemical (g) that need was dissolve or dilute in distilled water (volumetric flask) to obtain M mol} \]

\[ M = \text{Wanted molar} \]

\[ V = \text{Wanted volume in mL} \]

\[ \text{MW} = \text{Molecular weight} \]

### 3.4 Stock Solution Dilution

10 different concentrations of salt solution were selected in order to bracket the expected retentate and permeate concentration of salt rejection test based on conductivity which shown as Table 3.3. Dilution factor using 0.1M as the stock solution (known molar) at 50 mL as wanted volume. Volumetric flask of 50 mL was used for dilution. The calculations were shown in Appendix C.

\[ M_1 V_1 = M_2 V_2 \]

Where,

\[ M_1 = \text{Wanted molar} \]

\[ V_1 = \text{Wanted volume} \]

\[ M_2 = \text{Known molar} \]

\[ V_2 = \text{To pipette volume} \]
### Table 3.3: Dilution Table for Salt Solution

<table>
<thead>
<tr>
<th>Concentration (M)</th>
<th>V&lt;sub&gt;to pipette volume stock solution (mL)&lt;/sub&gt;</th>
<th>V&lt;sub&gt;distilled water (mL)&lt;/sub&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>50</td>
<td>0</td>
</tr>
<tr>
<td>0.08</td>
<td>40</td>
<td>10</td>
</tr>
<tr>
<td>0.06</td>
<td>30</td>
<td>20</td>
</tr>
<tr>
<td>0.04</td>
<td>20</td>
<td>30</td>
</tr>
<tr>
<td>0.02</td>
<td>10</td>
<td>40</td>
</tr>
<tr>
<td>0.01</td>
<td>5</td>
<td>45</td>
</tr>
<tr>
<td>0.008</td>
<td>4</td>
<td>46</td>
</tr>
<tr>
<td>0.006</td>
<td>3</td>
<td>47</td>
</tr>
<tr>
<td>0.004</td>
<td>2</td>
<td>48</td>
</tr>
<tr>
<td>0.002</td>
<td>1</td>
<td>49</td>
</tr>
</tbody>
</table>

### 3.5 Preparing Conductivity-Concentration Curve

It is important to calibrate a conductivity-concentration curve in order to obtain the concentration of permeate and retentate. In this study, NaCl was used as feed solution or inorganic electrolytes. Different range of calibration curves (0 – 0.01M) and (0.01M – 0.10M) were prepared to meet the required range of the concentration of permeate and retentate. The conductivity of the solutions were measured by conductivity meter and the unit is in milisiemens (mS).

Before determine the concentration of permeate and retentate of the solution, the conductivity need to be determined first. Then, the conductivity determined will be referred to the calibration curve in order to determine the concentration of the salt solutions. The calibration curve for the salt solutions for conductivity against concentration was shown in Appendix D.
3.6 Morphology Study of Synthesized Membrane by Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) will be used to study the surface morphology of the hybrid membrane. Through a scanning mode by using conventional SEM techniques, areas ranging from approximately 1 cm to 5 microns in width can be with magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm (Swapp, 2013). In this study, FESEM- JEOL 6701-F is used as the equipment to identify the morphology of the membranes.

3.7 Tensile Test by using Light Weight Tensile Testing Machine

Tensile testing was used to study the E. Modulus and Ultimate Tensile Strength (UTS) of the membranes. Width and gauge length of the membrane were cut into 3 mm and 10 mm respectively. It was then tested by using light weight tensile test machine, with speed of 1 mm/min, extension range up to 2000 mm and load range up to 500 N.
CHAPTER 4

RESULTS AND DISCUSSION

Based on the results obtained from the tests, results analysis were discussed in this chapter to scrutinize the effects of polymer membranes incorporate with different concentrations of PAC in terms of pure water permeability, salt rejection rate, tensile strength and morphology study of synthesized membrane.

4.1 Membranes Performance Test

4.1.1 Membranes Pure Water Flux

In pure water flux test, the permeability of membranes which have the casting condition with same thickness, shear rate, temperature and casting solution but different concentrations of PAC were tested and compared. The pure water flux was determined by using dead-end filtration with different PES-PAC wt. % membranes as function of applied pressure. The time taken to collect 5 mL of permeate was recorded, and the data was used to calculate the pure water flux. The diameter of membrane was 0.047 m, which has an effective surface area at $1.734 \times 10^{-3}$ m$^2$. The results of the test were shown in Table 4.1 and these data were used to determine whether these membranes are suitable to use for subsequent membrane testings.

According to Mustaffar, et al., (2004), the higher the polymer concentration, the lower the flux production of the membrane. Therefore, the results in this test were proven
as when there is more PAC in the membrane, the lesser the concentration polymer it is in the membrane. And hence, the higher the flux of the membrane.

Table 4.1: Pure Water Flux Productivity by Different PES-PAC wt. % and Pressures Applied on Synthesized Membranes

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Pressure (bar)</th>
<th>Time (s)</th>
<th>Volume (mL)</th>
<th>Volume (m³)</th>
<th>Flux (m³/m²/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PES-PAC</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>0 wt. %</td>
<td>0.1</td>
<td>85</td>
<td>5</td>
<td>5 × 10⁻⁶</td>
<td>3.39 × 10⁻⁵</td>
</tr>
<tr>
<td></td>
<td>0.2</td>
<td>60</td>
<td>5</td>
<td>5 × 10⁻⁶</td>
<td>4.80 × 10⁻⁵</td>
</tr>
<tr>
<td>PES-PAC</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>5 wt. %</td>
<td>0.1</td>
<td>72</td>
<td>5</td>
<td>5 × 10⁻⁶</td>
<td>4.00 × 10⁻⁵</td>
</tr>
<tr>
<td></td>
<td>0.2</td>
<td>55</td>
<td>5</td>
<td>5 × 10⁻⁶</td>
<td>5.24 × 10⁻⁵</td>
</tr>
<tr>
<td>PES-PAC</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>11 wt. %</td>
<td>0.1</td>
<td>56</td>
<td>5</td>
<td>5 × 10⁻⁶</td>
<td>5.15 × 10⁻⁵</td>
</tr>
<tr>
<td></td>
<td>0.2</td>
<td>44</td>
<td>5</td>
<td>5 × 10⁻⁶</td>
<td>6.55 × 10⁻⁵</td>
</tr>
</tbody>
</table>

Figure 4.1 shows an increasing of flux with different PES-PAC wt. % membranes against different pressures. The pure water flux is directly proportional to the pressure applied for all the membranes produced. The PES membrane without PAC has the lowest pure water flux compared with other PES membranes with PAC. These results indicated that the permeability of PES membranes increased when PAC was added. Besides, the result also showed that PES-PAC 11 wt. % membrane has the best pure water permeability compare to PES-PAC 0 wt. % and PES-PAC 5 wt. % membranes in the test. In other words, the more the concentration of PAC added in PES membrane, the higher will be the pure water permeability. Furthermore, this result also indicated that these membranes were suitable to be used for further membrane performance testing.
4.1.2 Salt Solution Flux

Three produced membranes with different concentration of PAC were gone through a solute rejection test with NaCl, 0.01M. The obtained data which presented in Table 4.2 were used to determine which PES-PAC wt. % membrane can perform the best in salt solution rejection.
Table 4.2: Salt Solution Flux of Different PES-PAC wt. % and Pressure Applied on Synthesized Membranes

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Pressure (bar)</th>
<th>Time (s)</th>
<th>Volume (mL)</th>
<th>Volume (m^3)</th>
<th>Flux (m^3/m^2/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PES-PAC</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>0 wt. %</td>
<td>0.1</td>
<td>138</td>
<td>10</td>
<td>1 × 10^{-5}</td>
<td>4.18 × 10^{-5}</td>
</tr>
<tr>
<td></td>
<td>0.2</td>
<td>92</td>
<td>10</td>
<td>1 × 10^{-5}</td>
<td>6.26 × 10^{-5}</td>
</tr>
<tr>
<td>PES-PAC</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>5 wt. %</td>
<td>0.1</td>
<td>85</td>
<td>10</td>
<td>1 × 10^{-5}</td>
<td>6.78 × 10^{-5}</td>
</tr>
<tr>
<td></td>
<td>0.2</td>
<td>67</td>
<td>10</td>
<td>1 × 10^{-5}</td>
<td>8.60 × 10^{-5}</td>
</tr>
<tr>
<td>PES-PAC</td>
<td>0.0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>11 wt. %</td>
<td>0.1</td>
<td>61</td>
<td>10</td>
<td>1 × 10^{-5}</td>
<td>9.45 × 10^{-5}</td>
</tr>
<tr>
<td></td>
<td>0.2</td>
<td>57</td>
<td>10</td>
<td>1 × 10^{-5}</td>
<td>10.11 × 10^{-5}</td>
</tr>
</tbody>
</table>

Figure 4.2 shows an increasing of salt solution flux with different PES-PAC wt. % membranes against different pressure. From the graph, it can be clearly observed that as the PAC concentration in PES membrane increase, the flux produced increase which the results trend was similar to the pure water flux. By increasing the PAC concentration to 11 wt. %, the performance of the membrane in flux production was enhanced tremendously as presented in Figure 4.2. It may because the higher the polymer concentration, the lower the flux production of the membrane as mentioned previously and supported by Mustaffar, et al., (2004).
4.1.3 Salt Rejection Rate

As shown in Table 4.3, the salt solution increase when the PAC concentration increase from 0 wt. % to 11 wt. %. It can be clearly observed that the membrane with PAC concentration of 11 wt. % was giving the highest rejection compare to membrane with 0 wt. % and 5 wt. %. The details of the calculations were shown in Appendix E.

<table>
<thead>
<tr>
<th>Pressure (bar)</th>
<th>Rejection by membrane with different concentrations of PAC (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0 wt. %</td>
</tr>
<tr>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>0.10</td>
<td>48.93</td>
</tr>
<tr>
<td>0.20</td>
<td>62.96</td>
</tr>
</tbody>
</table>

From Figure 4.3, it can be observed that the rejection rate was increased from 0.00 bar to 0.20 bar. This condition can be explained that when there is higher concentration of
PAC, there are more easily to adsorb the salt in the solution. Thus, the salt rejection was higher. However, when up to 0.2 bar of pressure was applied, the rejection rate was slightly decrease. This may due to these PAC may adsorb enough salt and there is not more area for the salt to be adsorbed to the PAC in the membrane.

![Graph Of Salt Solution Rejection Against Different Pressure (real %)](image)

**Figure 4.3:** Graph of Salt Solution Rejection against Different PES-PAC wt. % at Different Pressure Applied on Membranes

### 4.2 Morphology Study on Synthesized Membrane

The scanning electron microscopy (SEM) images of two membranes were shown in Figure 4.1. One was without PAC and another one was with PAC added into the membrane. Membrane with PAC has more pores on the surface of the membrane compare with the membrane without PAC. In other words, additives are able to enhance the marcovoid formation and there are more pores will be formed on the membrane. Hence, the flux production increased. In this study, produced membranes shows the structural properties with increment of porosity with and without PAC added as shown in Figure 4.4 and Figure 4.5.
Figure 4.4: Scanning Electron Microscopy (SEM) of Membranes without PAC
(a) × 700, (b) × 2000

Figure 4.5: Scanning Electron Microscopy (SEM) of PES-PAC 5 wt. % Membranes
(a) × 700, (b) × 2000

4.3 Tensile Testing

Tensile testing was carried out for the membranes in order to test the E. Modulus and ultimate strength of the membranes. It is important to know that how much strength the membranes are able withstand. During this test, some parameters were fixed at certain conditions such as width and gauge length of the membrane, speed of the extension, extension range, and load range as shown in Table 4.4.
Table 4.4: Fixed Parameters for Tensile Testing

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Width</td>
<td>3 mm</td>
</tr>
<tr>
<td>Gauge Length</td>
<td>10 mm</td>
</tr>
<tr>
<td>Speed</td>
<td>1 mm/min</td>
</tr>
<tr>
<td>Extension Range</td>
<td>2000 mm</td>
</tr>
<tr>
<td>Load Range</td>
<td>500 N</td>
</tr>
</tbody>
</table>

From the data obtained from Table 4.5, PES-PAC 0 wt. % has the best UTS compared with those membrane with PAC added. Besides, it was also clearly observed that the E. Modulus and Ultimate Tensile Strength (UTS) of PES-PAC 5 wt. % was dropped from 28.15 MPa to 10.35 MPa and 2.216 MPa to 1.775 MPa, respectively as compare to PES-PAC 0 wt. % membrane. However, the E. Modulus and UTS of PES-PAC 11 wt. % membrane were increased again to 21.67 MPa and 1.912 MPa, respectively as compared to PES-PAC 5 wt. % membrane. Therefore, this may because the formulation of PES-PAC 11 wt. % was just the optimum concentration for the combination of PES and PAC and hence strengthen the membrane. From the data obtained, PES-PAC 0 wt. % has the best UTS compare with those membrane with PAC added.

Table 4.5: Tensile Testing with Different Membranes

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Average Thickness (mm)</th>
<th>E. Modulus (MPa)</th>
<th>Ultimate Tensile Strength, UTS (MPa)</th>
<th>Extension (mm)</th>
<th>Force (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PES-PAC 0 wt. %</td>
<td>0.2580</td>
<td>28.15</td>
<td>2.261</td>
<td>1.15</td>
<td>1.75</td>
</tr>
<tr>
<td>PES-PAC 5 wt. %</td>
<td>0.2563</td>
<td>10.35</td>
<td>1.775</td>
<td>1.00</td>
<td>1.35</td>
</tr>
<tr>
<td>PES-PAC 11 wt. %</td>
<td>0.2240</td>
<td>21.67</td>
<td>1.912</td>
<td>1.15</td>
<td>1.30</td>
</tr>
</tbody>
</table>
CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

Some findings had been discovered in this study. All the results will be concluded in this chapter in order to clarify the findings. Besides, some recommendations also being given to others so that this study can be further with different aspects.

5.1 Conclusion

Hybrid polymeric (low polymer concentration) membrane with PAC was successfully fabricated in this study. Produced membranes with different PAC concentrations, 0 wt. %, 5 wt. %, and 11 wt. % were evaluated based on pure water and salt solution permeability, salt rejection rate, tensile test and morphology test. Through these tests, the optimum PAC concentration used in membrane fabrication was determined which was PES-PAC 11 wt. % membrane. Hence, it prove that PAC as additive able to improve the membrane performances in term of permeability, and rejection rate. Meanwhile, the tensile strength test may needed for further study in order to understand the strength of PES-PAC membrane in details. SEM image revealed that the porosity of the produced membranes improved with the increment of PAC concentration. Therefore, the overall performances of membrane improved with the increase of PAC concentrations in this study.
5.2 Recommendation

After went through this membrane performance study, some recommendations had been made in order to make further improvement in future study. These works includes:

i. Determine the fouling rate of different PES-PAC wt. % membranes.
ii. Determine the pH resistance of different PES-PAC wt. % membranes.
iii. Determine COD and colour removal efficiency in MBR application.
REFERENCES


Franken, A., 2009. Prevention and Control of Membrane Fouling: Practical Implications and Examining recent Innovations.[Online] Available at:


Mustaffar, M., Ismail, A. & Illias, R., 2004. Study on the effect of polymer concentration on hollow fiber ultrafiltration membrane performance and morphology, Johor Bahru: Membrane Research Unit, Faculty of Chemical & Natural Resources Engineering, Universiti Teknologi Malaysia.


Northeast Georgia Regional Development Center, 1997. Description of Commonly Considered Water Quality Constituents, s.l.: s.n.


Appendix A

Calculation for amount of PES, PAC, and NMP needed in membrane fabrication.

The ratio of PES to NMP is 12:88 and the percentage of PAC was varied during membrane fabrication as shown in Table 3.2. The calculations were shown below.

**PES-PAC 0 wt. % Membrane**

PAC concentration needed = Polymer concentration × percentage of PAC

= 12 g × 0%

= 0.00 g

PES concentration needed = Polymer concentration – PAC concentration

= 12 g – 0.00 g

= 12.00 g

Thus, in 100 g of dope preparation,

PES: NMP: PAC = 12.00 g: 88.00 g: 0.00 g

**PES-PAC 5 wt. % Membrane**

PAC concentration needed = Polymer concentration × percentage of PAC

= 12 g × 5%

= 0.60 g

PES concentration needed = Polymer concentration – PAC concentration

= 12 g – 0.60 g

= 11.40 g

Thus, in 100 g of dope preparation,

PES: NMP: PAC = 11.40 g: 88.00 g: 0.60 g
PES-PAC 11 wt. % Membrane

PAC concentration needed = Polymer concentration × percentage of PAC
= 12 g × 11%
= 1.32 g

PES concentration needed = Polymer concentration – PAC concentration
= 12 g – 1.32 g
= 10.68 g

Thus, in 100 g of dope preparation,

PES: NMP: PAC = 10.68 g: 88.00 g: 1.32 g
Appendix B

Calculation for preparation of 0.1 M NaCl stock solution.

To prepare 1 L of 0.1 M NaCl stock solution,

\[ m = MW \times M \times V \]
\[ = 58.45 \text{ g/mol} \times 0.1 \text{ M} \times 1 \text{ L} \]
\[ = 5.845 \text{g of NaCl} \]
Appendix C

Calculation for stock solution dilution.

The required concentration of salt solutions were 0.08 M, 0.06 M, 0.04 M, 0.02 M, 0.01 M, 0.008 M, 0.006 M, 0.004 M, and 0.002 M.

\[ M_1V_1 = M_2V_2 \]
\[ V_1 = \frac{(M_2V_2)}{M_1} \times 1000 \text{ mL/L} \]

For 0.08 M of salt solution,
\[ V_1 = \frac{(0.08 \times 0.05 \text{ L})}{0.1 \times 1000 \text{ mL/L}} \]
\[ = 40 \text{ mL} \]

For 0.06 M of salt solution,
\[ V_1 = \frac{(0.06 \times 0.05 \text{ L})}{0.1 \times 1000 \text{ mL/L}} \]
\[ = 30 \text{ mL} \]

For 0.04 M of salt solution,
\[ V_1 = \frac{(0.04 \times 0.05 \text{ L})}{0.1 \times 1000 \text{ mL/L}} \]
\[ = 20 \text{ mL} \]

For 0.02 M of salt solution,
\[ V_1 = \frac{(0.02 \times 0.05 \text{ L})}{0.1 \times 1000 \text{ mL/L}} \]
\[ = 10 \text{ mL} \]
For 0.01 M of salt solution,
\[ V_1 = \frac{(0.01 \times 0.05 \text{ L})}{0.1 \text{ M} \times 1000 \text{ mL/L}} = 5 \text{ mL} \]

For 0.008 M of salt solution,
\[ V_1 = \frac{(0.008 \times 0.05 \text{ L})}{0.1 \text{ M} \times 1000 \text{ mL/L}} = 4 \text{ mL} \]

For 0.006 M of salt solution,
\[ V_1 = \frac{(0.006 \times 0.05 \text{ L})}{0.1 \text{ M} \times 1000 \text{ mL/L}} = 3 \text{ mL} \]

For 0.004 M of salt solution,
\[ V_1 = \frac{(0.004 \times 0.05 \text{ L})}{0.1 \text{ M} \times 1000 \text{ mL/L}} = 2 \text{ mL} \]

For 0.002 M of salt solution,
\[ V_1 = \frac{(0.002 \times 0.05 \text{ L})}{0.1 \text{ M} \times 1000 \text{ mL/L}} = 1 \text{ mL} \]
Appendix D

Calibration curve for salt solution for conductivity against concentration.

<table>
<thead>
<tr>
<th>Concentration (M)</th>
<th>Conductivity (mS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.000</td>
<td>0.001</td>
</tr>
<tr>
<td>0.002</td>
<td>0.217</td>
</tr>
<tr>
<td>0.004</td>
<td>0.424</td>
</tr>
<tr>
<td>0.006</td>
<td>0.609</td>
</tr>
<tr>
<td>0.008</td>
<td>0.831</td>
</tr>
<tr>
<td>0.010</td>
<td>1.040</td>
</tr>
</tbody>
</table>

\[ y = 103.14x + 0.0047 \]

\[ R^2 = 0.9996 \]

Graph of conductivity against concentration (NaCl)
<table>
<thead>
<tr>
<th>Concentration (M)</th>
<th>Conductivity (mS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>0.001</td>
</tr>
<tr>
<td>0.01</td>
<td>1.040</td>
</tr>
<tr>
<td>0.02</td>
<td>2.078</td>
</tr>
<tr>
<td>0.04</td>
<td>3.950</td>
</tr>
<tr>
<td>0.06</td>
<td>5.920</td>
</tr>
<tr>
<td>0.08</td>
<td>7.630</td>
</tr>
<tr>
<td>0.10</td>
<td>10.010</td>
</tr>
</tbody>
</table>

Graph of conductivity against concentration (NaCl)

\[ y = 97.966x + 0.0371 \]

\[ R^2 = 0.9988 \]
Appendix E

Calculation for percentage of salt rejection by different PES-PAC wt. % membrane at different applied pressure.

**PES-PAC 0 wt. %**

<table>
<thead>
<tr>
<th>Pres Bar</th>
<th>Press-1</th>
<th>time</th>
<th>time</th>
<th>Kond Suapan</th>
<th>Kond Permeate</th>
<th>Kond Retentate</th>
<th>Flux</th>
<th>flux (Jv/k) exp(G)</th>
<th>Cf</th>
<th>Cp</th>
<th>Cr</th>
<th>Cb</th>
<th>Cw</th>
<th>Robs</th>
<th>Rreal</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>0.00E+00</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>0.1</td>
<td>1.01E+04</td>
<td>2</td>
<td>18</td>
<td>138.0</td>
<td>1.140</td>
<td>0.850</td>
<td>1.077</td>
<td>4.19E-05</td>
<td>41.87</td>
<td>0.9361</td>
<td>0.01094</td>
<td>0.00774</td>
<td>0.0106</td>
<td>0.0151</td>
<td>27.3136</td>
</tr>
<tr>
<td>0.2</td>
<td>2.03E+04</td>
<td>1</td>
<td>32</td>
<td>92.0</td>
<td>1.140</td>
<td>0.925</td>
<td>1.077</td>
<td>6.28E-05</td>
<td>62.830</td>
<td>1.4041</td>
<td>0.01094</td>
<td>0.00751</td>
<td>0.01034</td>
<td>0.0106</td>
<td>0.0203</td>
</tr>
</tbody>
</table>

**PES-PAC 5 wt. %**

<table>
<thead>
<tr>
<th>Pres Bar</th>
<th>Press-1</th>
<th>time</th>
<th>time</th>
<th>Kond Suapan</th>
<th>Kond Permeate</th>
<th>Kond Retentate</th>
<th>Flux</th>
<th>flux (Jv/k) exp(G)</th>
<th>Cf</th>
<th>Cp</th>
<th>Cr</th>
<th>Cb</th>
<th>Cw</th>
<th>Robs</th>
<th>Rreal</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>0.00E+00</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
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<td>0</td>
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<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>0.1</td>
<td>1.01E+04</td>
<td>2</td>
<td>25</td>
<td>85.0</td>
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<td>68.004</td>
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<td>0.0228</td>
<td>32.0168</td>
</tr>
<tr>
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<td>2.03E+04</td>
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<td>7</td>
<td>67.0</td>
<td>1.140</td>
<td>0.823</td>
<td>1.077</td>
<td>8.63E-05</td>
<td>86.274</td>
<td>1.9281</td>
<td>0.01094</td>
<td>0.00749</td>
<td>0.01034</td>
<td>0.0106</td>
<td>0.0292</td>
</tr>
</tbody>
</table>

**PES-PAC 11 wt. %**

<table>
<thead>
<tr>
<th>Pres Bar</th>
<th>Press-1</th>
<th>time</th>
<th>time</th>
<th>Kond Suapan</th>
<th>Kond Permeate</th>
<th>Kond Retentate</th>
<th>Flux</th>
<th>flux (Jv/k) exp(G)</th>
<th>Cf</th>
<th>Cp</th>
<th>Cr</th>
<th>Cb</th>
<th>Cw</th>
<th>Robs</th>
<th>Rreal</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
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<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
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<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>0.1</td>
<td>1.01E+04</td>
<td>1</td>
<td>1</td>
<td>61.0</td>
<td>1.140</td>
<td>0.835</td>
<td>1.077</td>
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<td>94.760</td>
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<td>0.01094</td>
<td>0.00760</td>
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<td>0.0106</td>
<td>0.0329</td>
</tr>
<tr>
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<td>2.03E+04</td>
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<td>57</td>
<td>57.0</td>
<td>1.140</td>
<td>0.84</td>
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<td>1.01E-04</td>
<td>101.410</td>
<td>2.2663</td>
<td>0.01094</td>
<td>0.00764</td>
<td>0.01034</td>
<td>0.0106</td>
<td>0.0366</td>
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