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FABRICATION OF CHITOSAN MEMBRANE:
THE EFFECTS OF DIFFERENT CHITOSAN COMPOSITIONS ON MEMBRANE PERFORMANCE IN TREATING OILY WASTEWATER

NURULHUDA MD.NOH

A thesis submitted in fulfillment of the
requirements for the award of the degree of
Bachelor of Chemical Engineering

Faculty of Chemical Engineering and Natural Resources
Universiti Malaysia Pahang

MAY 2008

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To my beloved mother and father

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ACKNOWLEDGMENT

In the name of Allah swt the Beneficent, the Merciful

The completion of this thesis has required the help and support of numerous people. They have to contribute toward our understanding and thoughts. In particular, I would like to thank Miss Siti Kholijah Abdul Mudalip for her help and guidance throughout my research. I have learnt so much from her. Without continued support and interest, this thesis would not have been the same as presented here. I would also like to thank the members of my research for everything that they have done for me and all of their help with my research.

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ABSTRACT

In this research four fabricated chitosan membrane was produced with different chitosan compositions. The membrane produced using the solvent casting method. All the fabricated membrane then was characterized using the scanning electron microscope (SEM) and tested using the different molecular weight (MWCO) to determine the pore size. The result of SEM shows that the fabricated membranes have an asymmetric membrane. The MWCO of the fabricated membrane is 1000 where it crosses the 90 percent the rejection rate. And lastly, the method of oil swelling was used to determine the favorable fabricated membrane to treat the oily wastewater. As a conclusion, the CHI-4 was the one favorable membrane to solve the oily problem with degree of oil swelling up to 227.80 % and also this fabricated membrane has a higher degree of oil swelling compare to the others membrane.

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ABSTRAK

Dalam kajian ini, empat jenis membran difabrikasi mengikut komposisi chitosan yang berbeza-beza. Kesemua membran ini akan di uji mengunakan *Scanning electron microscope* untuk mendapatkan keratan rentas dan diuji dengan PEG yang berbeze-beza berat untuk menentukan julat saiz lubang membran yang dihasilkan. Daripada keputusan ujian SEM menunjukan membran yang dihasilkan adalah *asymmetric membrane*. Julat *molecular weight cut off* membran yang dihasilkan adalah 1000 dimana ia melalui 90 peratus kadar pengeluaran membran. Bagi menentukan membran yang terbaik untuk menyingkirkan sisa buangan berminyak, *oil swelling method* akan diguna pakai. Secara kesimpulanya, CHI-4 merupakan membran yang terbaik dengan kadar penyerapan minyak yang tertinggi hingga 227.80 % bagi merawat permasalahan sisa buang berminyak ini.

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LIST OF SYMBOLS

Cp	-	Permeate concentration
Cf	-	Feed concentration
Wdt	-	Dry weight of the membrane
Wst	-	Swelling weight of the membrane

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LIST OF FIGURES

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Work by Karukulski *et al*.(1995) has stated that oily wastewaters are the main pollutants discharged to environment. Oily wastewaters are generated by ship mainly in engine-rooms in amounts of millions of tons annually. This waste cannot be drained to the sewage system without proper treatment even if they are much diluted. Although in lower volume, the oily wastewater still higher concentration of pollutants. Thus, oily wastewater treatment is important.

Now days, membrane are widely used to treat oily waste. These membranes are microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO). The membrane process can be classified in a number of different ways including (i) the type of material which membrane is made, (ii) the nature of driving force, (iii) the separation mechanism, and (iv) the nominal size of the separation achieved (Grey,2002).

An application of chitosan in ultrafiltration membrane is an attractive method to treat oily wastewater. This is due to the excellent coagulating, flocculating and metal chelating properties of chitosan originating from the high density of amino groups on its polymer chains. Chitosan is found to be one of the most powerful heavy metal ion binders with binding capacities of some metals by far exceeding activated carbon.

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Membrane or semi permeable membrane is a thin layer of material that is capable of separating material as a function of their physical and chemical properties when driving force is applied across the membrane. Ultrafiltration membrane is commonly used for separating oil from water. Treatment by ultrafiltration membrane is a versatile treatment method for oily wastewater now a day. In addition, membrane filtration process carried out at room temperature; without any yield decrease (Krajewska, 2005). The ultrafiltration membrane process is considered a cost-effective option in terms of higher permeate flux compared to others. One of the advantages using membrane filtration is that permeates from ultrafiltration can be considered microorganism-free, due to the pore size as compared with the size of the micro-organism (Peeters, 2006).

1.2 Chitosan Ultrafiltration Membrane

Ultrafiltration has been shown as a promising alternative for removal of trace oil from water (oily waste). The unbound oily pass through the membrane and the oil is retained. The advantages of this method are the low-energy requirement involved in ultrafiltration and the high removal efficiency because of effective binding (Verbych and Bryk, 2005).

Chitosan is a partially acetylated glucosamine polymer encountered in the cell wall of some *fungi* such as Mucorales strains, but it mainly results from deaceylation of chitin. It is basically soluble in acidic solutions and often used as a coagulant or flocculant in water treatment processes. In addition, chitosan is a well known solid sorbent for transition metal because the amino groups on chitosan chain can serve as coordination sites. In addition to binding ability, it has a high content of functional groups and is produced at lower cost since chitin is the second abundant biopolymer in nature next to cellulose (Juang and Shiau 1999).

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1.3 Problem Statements

More and more oily wastewater has been generated from the development of oil fields, especially for those using water injection technologies (Yu 2005). It is necessary to purify this water so that it can be reused to save water resources and to protect the environment. Conventional oily wastewater treatment method include gravity separation and skimming, dissolved air floatation de-emulsification, coagulation and flocculation, which have several disadvantages such as low efficiency, high operation costs, corrosions and recontamination problems (Grey, 2002). Also, these methods are not effective in removing smaller oil droplets and emulsion. These disadvantages have promoted the development of new process for oily wastewater treatment.

Membrane filtration is playing a more prominent role in the treatment of oily wastewater due its advantages such as: (i) No chemical additives are needed to break the emulsion; (ii) Higher COD removal efficiencies and (iii) Treatment facilities are quite compact and fully automated (Krajewska, 2004). In this research, chitosan, which can be spread on water to absorb oil or grease, will be added to the membrane. Chitosan also carry a strong positive charge that allows them to chemically bond with certain compounds (Tsai and Chen, 2005).This binding action had drawn the interest of numerous industries especially to oily wastewater treatment. Chitosan causes the fine sediment particles to bind together and it subsequently removed with the sediment during the filtration on membrane.

In this work, a new method by using chitosan in ultrafiltration membrane is suggested for tackling this oily wastewater problem. It is due to the advantages of chitosan in treating the oily wastewater besides it material is so natural and environmentally safe.

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1.4 Research Objective

The main objective of this research is to fabricate the chitosan membrane and to study the effects of different chitosan compositions on membrane performance in oily wastewater treatment.

1.5 Scope of Research

In conjunction with the problem statement and objective of the experiment, the scope of the experiment can be described as:-

i) To fabricate the chitosan membrane
ii) To characterize the fabricated membrane
iii) To treat the oil wastewater using fabricated membrane
iv) To study the effects of different chitosan compositions on membrane performance
v) To analyze and compare the membrane performance

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CHAPTER 2

LITERATURE REVIEWS

2.1 Oily Wastewater

Oil/water separation covers a broad spectrum of industrial process operations. There are many techniques employed depending on the situation. The oily wastewater application can be broken down into categories determined by the type of user and the oil/water separation desired. Oil is not soluble in water but can exist evenly dispersed as globules in water (Li, 2005). The concentration of these globules is a function of mixing or stirring. If allowed to stand the emulsion will separate because oil is lighter than water, although, some amount of oil globules will remain in the water. Another interesting fact is that this emulsion can exist two days.

2.2 Definition of Membrane

A precise and complete definition of a membrane which covers all its aspects is rather difficult, even when the discussion is limited to synthetic structure (Kuan, 2005). In the most general sense, a synthetic membrane is a barrier which separate two phase and restrict the transport of various chemical species in a rather specific manner. A membrane can be homogenous or heterogeneous, symmetric or asymmetric in structure; it may be solid or liquid; it may be neutral, may vary between less than 100 nm to more

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than a centimeter. The electrical resistance may vary from several megohms to fraction of an ohm. Mass transport through a membrane may be caused by convection or by diffusion of individual molecules, induced by electric field, or s concentration, pressure or temperature gradient.

The term membrane therefore, includes a great variety of material and structures, and a membrane can often be better describe in term of what it does rather than what it is. Some material, though not meant to be membranes, show typical membrane properties, and in fact are membrane e.g., protective coating, or packaging materials. All material functioning as membrane has on characteristics properties in common: which is restricting the passage of various chemical species in a very specific manner.

2.3 Membrane Technology

Membrane processes such as microfiltration, ultrafiltration, nanofiltration and reverse osmosis are increasingly being applied for treating oily wastewater (Cheryan and Rajagopalan, 1998). Of the three broad categories of oily wastewater-free floating oil, unstable oil/water emulsion, high stable oil/water emulsion-membrane are most useful with stable emulsion, particularly water soluble oily waste. Free oil, on the other hand, can be readily removed by mechanical separation devices which use gravitational force as the driving force. Unstable oil or water emulsion can be mechanically or chemically broken and then gravity separated. Figure 2.1 is a general schematic diagram for ultrafiltration based system for treatment of oily wastes. Pretreatment to remove large particles and free oil is needed, especially if thin-channel membrane equipment is used. The membrane unit is usually operated in semi batch recycle. The waste water feed is added to the process tank at the same rate as clean permeate is withdrawn, thus keeping at constant level in the tank. The retentate containing the oil and grease and other suspended matter reach a certain predetermined concentration in the tank, the feed is stopped and the retentate allowed concentrating. Usually, this result in final concentrate

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volume that is only 3-5 % of the initial volume of oily wastewater fed to the process tank .

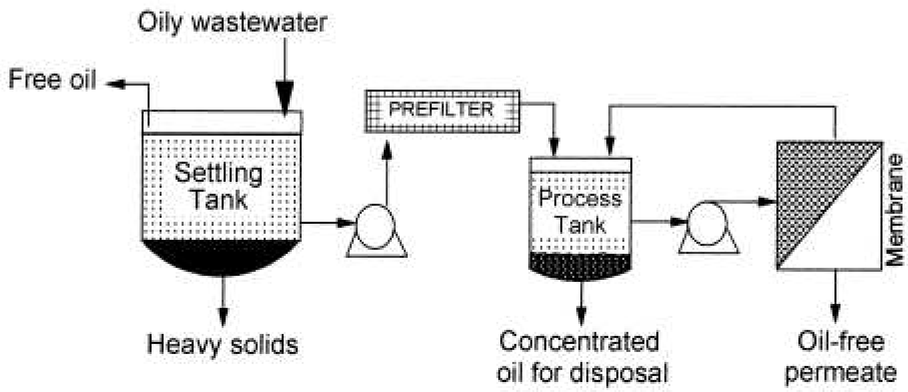

Figure 2.1: Schematic diagram for ultrafiltration based system for treatment of oily wastes (Cheryan and Rajagopalan, 1998)

Membrane has several advantages (Cheryan and Rajagopalan, 1998), among them are:

1. The technology is more widely applicable across a wide rage of industries
2. The membrane is a positive barrier to rejected components. Thus, quality of treated water (the permeate) is more uniform regardless of influent variations. These variations may decrease flux, but generally does not affect quality of its output.
3. No extraneous chemical are needed, making subsequent oil recovery easier
4. Membrane can be used in-process to allow recycling of selected waste stream within a plant.
5. Concentrates up to 40-70% oil and solids can be obtained by ultrafiltration or microfiltration. Since most oily waste contain 0.1-1 % oil, this means the volume of waste that has to be subsequently hauled away or treaded is reduced to 1/40-1/200 the initial feed volume.
6. Membrane equipment has smaller foot print.
7. Energy costs are lower compared to thermal treatments.
8. The plant can be highly automated and does not require highly skilled operators

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Membrane processes also have some limitations. There are:

1. Scale-up is almost linear above a certain size. Thus, capital cost for very large effluent volumes can be high
2. The polymeric membrane suffers from fouling and degradation during use. Thus they may have to replace frequently, which increase operating can cost significantly (Cheryan and Rajagopalan, 1998).

In spite of the disadvantages, membrane processing of oily wastewaters, some time in conjunction with other methods for treating the residuals, is a commercial success. Membrane are gaining wider acceptance for two reasons: 1) It consistently produces effluents of acceptable discharge quality, and 2) it perceived to be simple process from operational viewpoint.

2.4 Membrane Types in Oily Wastewater Treatment

2.4.1 Nanofiltration Membranes

This membrane is used to separate materials on the order of nanometers. These membranes are not usually rated based on their pore size because the pores are very small and difficult to measure accurately. Instead they rated based on the approximate molecular weight of the components that they reject or the % of salts they can removed from a stream. These membranes are used predominately for wastewater treatment but also used to concentrate material that has a wide range of particle sizes (Grey,2002).

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2.4.2 Ultrafiltration Membrane

Conventional ultrafiltration membrane is composed of some type of polymer material with pores ranging from a little less than 0.01µm to 0.1µm. These membranes are used for many different separations including oily wastewater treatment, protein concentration, colloidal silica concentration and treatment of various wastewaters in the pulp and paper industry (Grey,2002).

2.4.3 Microfiltration Membrane

These membranes tend to be pores grater than 0.1µm.These types of membrane are used to separate larger particulate matter from liquid phase. Some example would be coarse minerals or paint particles, which need to be concentrated from an aqueous solution (Grey,2002).

2.4.4 Reverse Osmosis Membrane

These are the tightest membrane for separating materials. They are generally rated on the % of salt that thye can be removed from a feed stream. However, they can also be specified by molecular weight cutoff. An example of their use would be for filtering seawater in order to removed salt. They also used to remove color, fragrance and flavor from water streams. Reverse osmosis don't have structural pores. Filtration occurs as ionic species are able to diffuse their way through the membrane itself (Grey,2002).

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2.5 Chitosan

Chitin and chitosan are fibers derived from marine animals. Chitin is polysaccharides (a string of sugar molecules) that are naturally occur in the hard outer shell of insects, shellfish such as crab, lobster and shrimp and marine coral. Chitin is chemically similar to cellulose and starch, the abundant plant fibers (Krajewska, 2005).

The chemical structure of chitin and chitosan are similar. Chitin is made up of a linear chain of acetylglucosamine groups while chitosan is obtained by removing enough acetyl group (CH3-CO) for the molecule to be soluble in most diluted acids. This process is called deacetylation. Chitosan having free amino groups is the most useful derivative of chitin (Krajewska, 2005).

Found in the shell of crustaceans, the exoskeletons of insect and the cell wall of fungi where it plays a structural ole, chitin is counted among the most plentiful, renewable organic resources in nature. Chemically, it is composed of β linked 2-acetamido-2-deoxy- β-D-glucose units forming a long chain linear polymer. It is a hydrophilic, tough and inert solid, insoluble in water and most ordinary solvent. Chitosan, the primary derivatives of chitin, is obtained by N-deacetylation to a varying extent that is characteristic by the degree of deactylation, and is consequently a copolymer of N-acetyglucosamine and glucosamine. Insoluble in water, chitosan readily dissolves in acidic solutions, which is due to the presence of amino groups in its molecules, the degree of deactylation necessary to obtain a soluble product being 80-85% or higher. Fully acetylated chitin, and chitosan, fully deacetylated chitin, can be chemically considered as analogues of cellulose, in which the hydroxyl at carbon-2 has been replaced by acetamido and amino groups.

Commercially, chitin and chitosan are obtained at a relatively low cost from the shell of shellfish (mainly crabs, shrimps, lobsters and krills) the waste of seafood processing industry. Basically the process consists of deproteinization of the raw shell

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material with dilute NaOH solution and decalcification with a dilute HCl solution. To afford chitosan, the obtained chitin is subjected to N-deacetylation by treatment with a 40-45% NaOH solution, followed by purification procedures. Depending on the origin and manufacture process the obtained products may vary in composition and properties.

Chitosan celates five to six times greater amounts of amino metal than chitin. This is attributed because of the free amino groups exposed in chitosan because of deacetylation of chitin. The biosorbent material, chitosan, is slightly soluble at low pHs and poses problems for developing commercial applications. It is also soft and has a tendency to agglomerate or form a gel in aqueous solutions. Figure 2.2 shows the conversion of chitin to chitosan by deacetylation process:

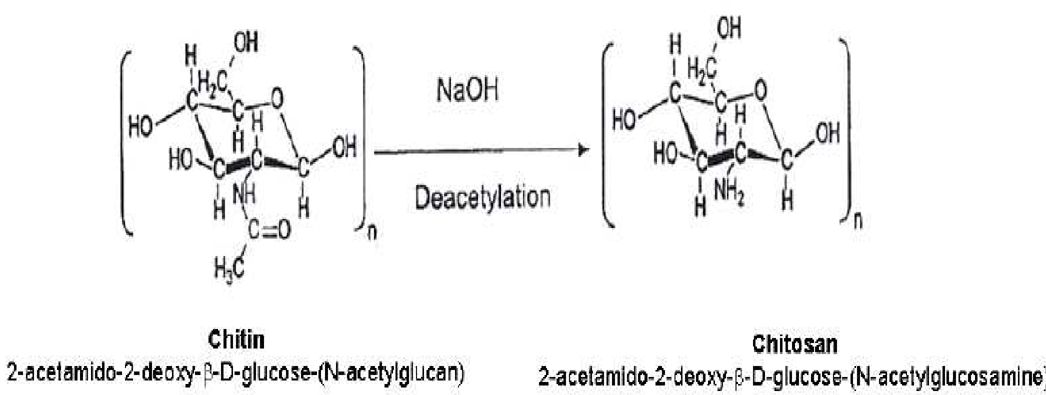

Figure 2.2 : Conversion of chitin to chitosan by deacetylation (Coughlin, 1990)

Many of the methods reported for converting chitin in crustacean shell to chitosan are slow and consume significant amounts of reagents. A relatively rapid and mild deacetylation method proposed by Coughlin *et al.*(1990) with slightly modification where the deacetylation process was subjected to microwave irradiation was used in this work. During microwave irradiation, polar molecules such as water align with the continuously changing magnetic field generated by microwaves.

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2.6 Characteristic of Chitosan

Chitosan is a non toxic, biodegradable polymer of high molecular weight and is a very much similar to cellulose a plant fiber. The only difference between chitosan and cellulose is the amine (-NH2) group in the position C-2 of chitosan instead of the hydroxyl (-OH) group found in cellulose. However chitosan possesses positive ionic charges, which give it the ability to chemically bind with negatively charged fats, lipids, cholesterol, metal ions, proteins and macromolecules.

2.6.1 Degree of Deacetylation (DD)

The process of deacetyaltion involves the removal of acetyl groups from the molecular chain of chitin, leaving behind a compound (chitosan) with a degree chemical reactive amino group (-NH2) .This make the degree of deacetylation (DD) and important property in chitosan production as its affects the physicochemical properties, hence determines its appropriate applications. Deactylation also affects the biodegradability and immunological activity (Tolaimate *et al*, 2000). The degree of deactylation can be employed to differentiatiate between chitin and chitosan because it determines the content of free amino groups in the polysaccharides. The degree of deactylation chitosan ranges from 15% -to 99% with an average of 80%, depending on the crustacean species and the preparation methods. Chitin with the degree of deacetylation of 75% or above is generally known as chitosan.

2.6.2 Molecular Weight

Chitosan is a biopolymer of high molecular weight. Like its composition, the molecular weight of chitosan varies with the raw material sources and the method of preparation. In general, high temperature, dissolved oxygen and shear stress can cause

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degradation of chitoson occurs and polymer chains rapidly break down, thereby lowering molecular weight (Kuan, 2005).

2.6.3 Viscosity

Viscosity is an important factor in the conventional determination of molecular weight of chitosan and in determining its commercial application in complex biological environments. Higher molecular weight chitosans often render highly viscous solutions, which may be desirable for industrial handling (Kuan, 2005).

2.6.4 Solubility

Chitosan soluble in dilute acids solution below pH 6.0. Organic acids such as acetic, formic, and lactic acids are used for dissolving chitosan. Chitosan is soluble in 1% hydrochloric acids but insoluble in sulfuric and phosphoric acids. Solubility of chitosan in inorganic acids is quite limited. Concentrated acetic acids solution can cause depolymerization of chitosan (Roberts and Domszy, 1982). Above pH 7.0 chitosan solubility is poor. The solubility is controlled by the degree of deactylation.

2.7 Scanning Electron Microscope (SEM)

The scanning electron microscope (SEM) is a type of electron microscope that creates various images by focusing a high energy beam of electrons onto the surface of a sample and detecting signals from the interaction of the incident electrons with the sample surface. Due to the manner in which this image is created, SEM images have

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great depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. In membrane, SEM is widely used to represent the cross section or morphology of a flat sheet membrane that produced. Figure 2.3 show the SEM (Kuan, 2005)

Figure 2.3: Scanning electron microscope

2.8 Pore Size Determination

It is difficult to measure the pore size directly by any of the techniques. The anisotropic structure and the wide distribution of pore size make this almost impossible. The molecular weight of the macro-molecules which retained by or pass through the membrane is used to determine the pore size of the membrane produced. The convention states that the molecular weight cut-off the membrane is equal to the molecular weight of globular protein which are 90% retained by the membrane.(Kuan, 2005)

Figure 2.4 shows how the molecular weight cut off is determined. The retention values of a series of globular protein are measured on the same membrane. The molecular weight at which the retention curve crosses a retentivity of 90% is the

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“molecular weight cut-off” of the membrane. This means that larger molecules are said to be retained by the membrane and smaller molecules are said to pass.

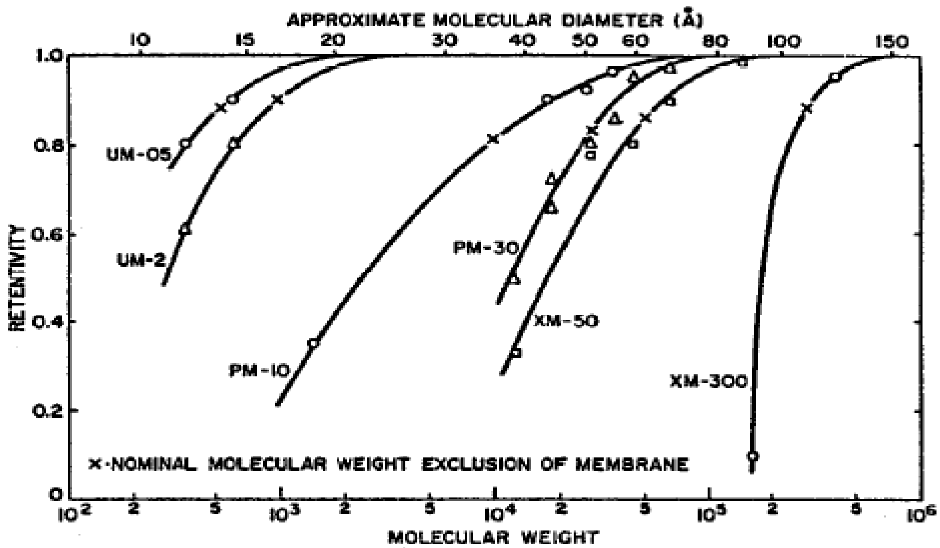

Figure 2.4: Retentivity of a series of globular proteins on various UF membrane (Kuan, 2005)

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CHAPTER 3

METHODOLOGY

3.1 Introduction

The research methodology carried in out in this study will be detail in this chapter. All the material, equipment used during the experiment is stated here. In this chapter, the methodology to prepare the chitosan membrane and the characterized of the fabricated membrane will be discussed detail here.

3.2 Material

The materials used in this research consist of chitosan powder as a main material, with the degree of deacetylation around 70-95%. Acetic acid around 10 ml was used as a solvent and 5 ml of distilled water in preparing the dope solutions.

3.3 Equipment

The equipments used in this research are as follow:

1) Casting knife
2) Scanning electron microscope

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3) Hot plate and magnetic stirrer

3.4 The Overall Method to Fabricate the Chitosan Membrane

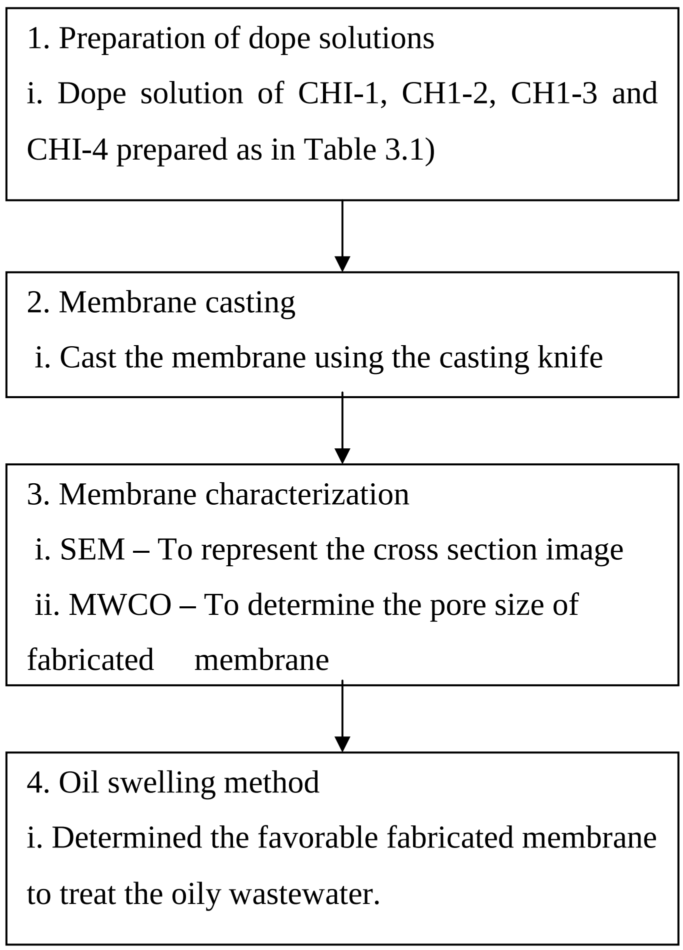

Figure 3.1: The overall method to fabricate the chitosan membrane

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3.5 Preparation of Dope Solution

Four different composition of dope solution as illustrated in Table 3.1 was prepared as in Figure 3.2. The dope solution was prepared using the hot plate and magnetic stirrer, where 10 ml acetic acid was poured first, followed by 0.2 g chitosan powder. The dope solution was heated to approximately 70 ^{0}C this is because at lower temperature, the dissolution of chitosan will take a longer time. However, too high temperature (>80^{o}C) may causes acetic acid to evaporate (Kuan, 2005).

Table 3.1: Composition of dope solution

Membrane Code	Chitosan Composition (gram)	Acetic Acid (solvent)(ml)	Water (ml)
CHI-1	0.2	10	5
CHI-2	0.4	10	5
CHI-3	0.6	10	5
CHI-4	0.8	10	5

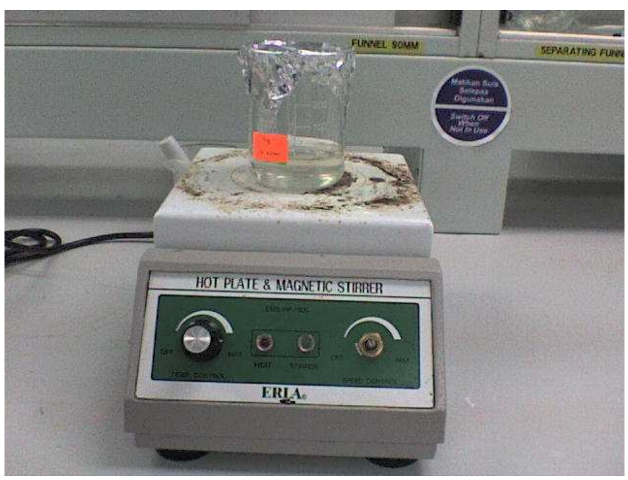

Figure 3.2: Preparation of dope solution using the hot plate and magnetic stirrer

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The chitosan powder was poured bit by bit, to ensure it dissolved properly. During the whole process, the dope solution was stirred, to encourage good dissolution of the polymer and ensure a homogenous mixing in the solution. From time to time, the distilled water was added slowly into the dope solution to avoid any agglomeration.

When the entire polymer is completely dissolved, indicated by the clear solution obtained as in Figure 3.3, it is cooled and poured into a storage bottle. Subsequently, the solution was degassed for about two hours and had to be kept away from sunlight to slow down its aging process (Kuan, 2005).

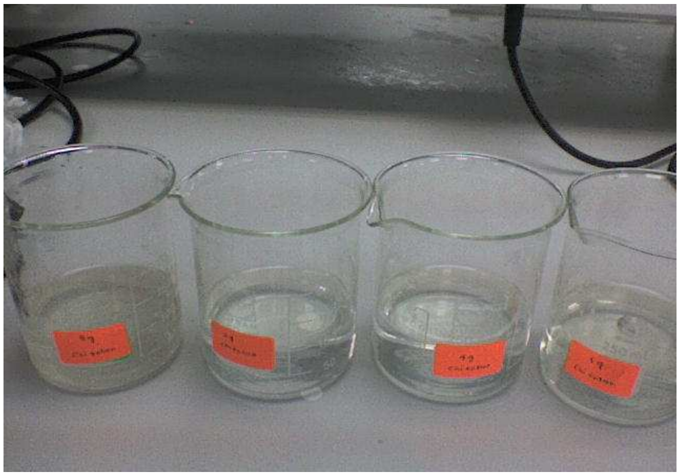

Figure 3.3: Dope solutions of different chitosan compositions

3.6 Membrane Casting

The membrane was cast using a casting knife as in Figure 3.4 A suitable amount of the solution was poured into the center well of the casting blade, which placed on the glass plane (Kuan, 2005).

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It takes about a week for this solution to evaporate and as a result of that; a thin dense chitosan membrane was produced. The Petri dish should be put on horizontal place to make the uniform thickness across the membrane area. The dried membrane was then gauged out by a small thin picker (Liangdeng, 2007). Next, the membrane was immersed into water bath to complete the phase separation, where the exchange of phases occurs between the solvent and water.

Figure 3.4 Casting flat sheet membrane using the casting knife

The membrane was then transferred to another container containing glycerol for post treatment to remove the excess acetic avid from membrane. Eventually, the membrane was transferred to another container that containing distilled water until it was used for experimental testing.

3.7 Membrane Characterizations

The fabricated membrane produced as in Figure 3.5 was characterized using SEM to get the cross section image each of the membrane and determination the pore size using the different molecular weight of PEG.

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Figure 3.5 The fabricated chitosan membrane (CHI-4)

3.7.1 Scanning Electron Microscope (SEM)

In order to explain the result obtained, the cross section image of the flat sheet membrane prepared were obtained using the high voltage scanning electron microscope. The membranes were snapped under liquid nitrogen, which gave a generally consistent and clean break. The membrane was mounted on a brass plate using double-sided adhesion tapes in a lateral position. The SEM result, will represent the morphology of the membrane and to observe the cross section area each of the membrane in order to characterize the membrane produced.

3.7.2 Pore Size Determination

The pore size of the membrane produced was determined by the molecular weight cut off using the different molecular weight of PEG (Polyethylene glycol). In this research, the PEG 200, PEG 400, PEG 600, PEG 800 and PEG 1000 were used to determine the pore size of the membrane produced.

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This experiment was performed over a time period at 31/2 hours (210 minutes). Each of the membrane was tested using the different weight of PEG to obtain the permeate concentration. The feed concentration is maintained at 5 mg/L for each of the PEG concentration. The permeate concentration each of the PEG used are listed in Table 3.2. The rejection rate (%) then determined using the equation 1.

$$\text{Rejection rate (\%)} = \left[1 - \frac{Cp}{Cf}\right] \times 100 \qquad (1)$$

Where *Cp* is permeate concentration (mg/L) and *Cf* is feed concentration (mg/L). The rejection rate of the membrane was calculated using the rejection rate equation and represent in Table 3.3.

3.8 Swelling Test

In this experiment, the swelling test was used to analyze the amount of oil content in oily waste water. POME was used to analysis the treatment of oily wastewater using the fabricated chitosan membrane.

The weight remaining profile of fabricate chitosan membrane was measured by immersed in POME waste water in order to study the swelling of oil waste using the fabricated chitosan membrane. Here the weight of chitosan membrane, before and after immersed in POME was measured to get the value of the oil absorb in the membrane. The weight of oil absorb by each of the membrane every 10 minutes obtained as in Table 3.4.

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The oil content absorb by the chitosan membrane then was represent in term of swelling of the oil. The swelling of the oil was measured for different time interval. Each sample, after immersed in POME waste for 10 minutes, was taken out and placed in Petri dish and then measured the weight. The degree of swelling (%) of each sample was then calculate according to the following equation:

$$\text{Degree oil swelling (\%)} = \frac{(Wst - Wdt)}{Wdt} \times 100 \qquad (2)$$

Where *Wdt* denotes the weight sample in dry state and *Wst* denotes the weight of sample in wet state after immersed in POME for an arbitary time interval. The oil swelling for each of the membrane was calculated using the equation 2 and tabulated in Table 3.5.

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CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Properties of the Fabricated Membrane

Four fabricated of chitosan membrane as in Table 4.1 were fabricated using the solvent-casting method described in Chapter 3, each membrane with the weight of 0.38-0.45 g and diameter of 7 cm. The differences in weight of the membrane are depends on the amount of the dope solution when it cast in the glass plate.

Table 4.1: Properties of chitosan membrane

Membrane Code	Weight (g)	Diameter (cm)
CHI-1	0.38	7
CHI-2	0.40	7
CHI-3	0.42	7
CHI-4	0.45	7

4.2 Membrane Morphology

Figure 4.1 shows SEM micrographs of the intermediate layer for three type of fabricated membrane which is CHI-1, CHI-3 and CHI-4. The SEM result for CHI-2 can not be done due to the less thick of the membrane.

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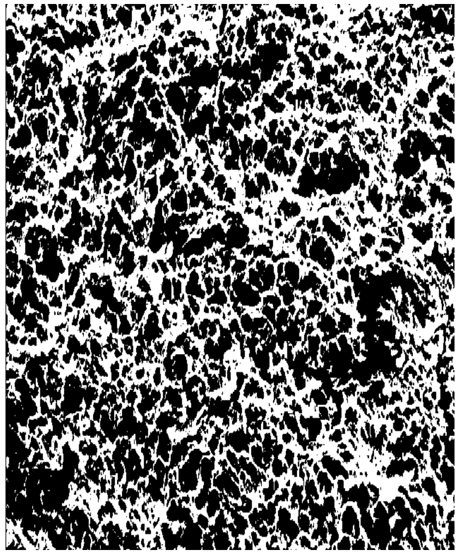
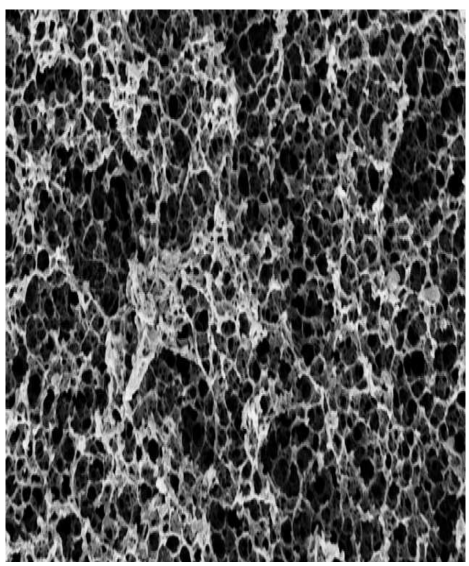


(a)



(b)

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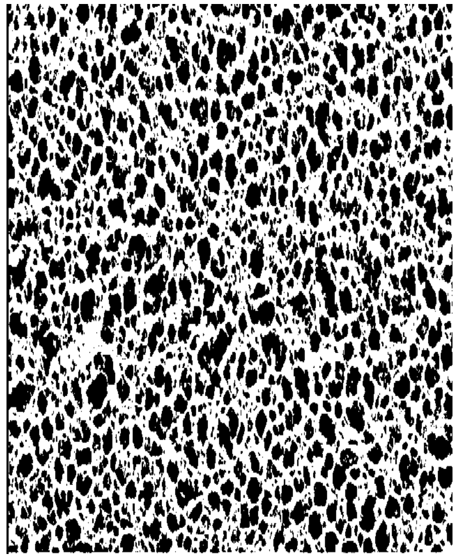

(c)

Figure 4.1: The SEM for (a) CHI-1, (b) CHI-3 and (c) CHI-4

From Figure 4.1, it is observed that the morphology of the membrane is very much influenced by the different of chitosan compositions. The dark regions in Figure 4.1 attribute to the porous in the membrane. The pore size for CHI-1 is much larger compare to the CHI-3 and CHI-4. The differences in pore size are influence by the amount of the concentration. The CHI-1 concentration is lowest, thus the pore size is the largest. Work done by Xue (2001) has indicated that the lower the solution concentration, the larger the membrane pore size and the membrane pore size decrease with increasing solution concentration. It also can be said that, the smaller the pore size, the oil remain are higher. Figure 4.1 show that the CHI-4 has the smallest pore size. Thus, CHI-4 is the good membrane to treat the oily waste water as compared to the CHI-1, CHI-2 and CHI-3.

Besides chitosan composition, the amounts of distilled water also affect the morphology of the membrane. The addition of water in the dope solution increases the skin porosity and thus allows the solute to pass through the membrane easily. The presence of water as the non-solvent in the dope solution will eventually transform from delayed demixing, which implies a non-porous membrane structure to instantaneous

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demixing, which implies a non-porous membrane structure to instantaneous demixing, where promotes a porous membrane structure (Kuan, 2005).

4.3 Molecular Weight Cut Off of the Membrane (MWCO)

Molecular weight cut off for each the membrane was tested using different molecular weight of PEG between 200 and 1000. The result of permeation concentration for each of the membrane is tabulated in Table 4.2.

Table 4.2: Permeation concentration of different PEG

Membrane Code	Feed Con *Cf* (mg/L)	PEG 200	PEG 400	PEG 600	PEG 800	PEG 1000
		Cp (mg/L)	*Cp* (mg/L)	*Cp* (mg/L)	*Cp* (mg/L)	*Cp* (mg/L)
CHI-1	5.00	3.00	2.50	2.90	1.80	0.50
CHI-2	5.00	3.30	2.30	2.24	1.58	1.00
CHI-3	5.00	3.41	3.00	2.67	1.41	0.65
CHI-4	5.00	3.56	2.50	2.50	1.21	0.77

The rejection rate percentage each of the fabricated membrane was determined using the equation 1 as mentioned in Chapter 3. The percentage of rejection rate, are shown in Table 4.3 and Figure 4.2. All the calculations to determine the rejection rate percentage are shown in appendix A.

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Table 4.3: Rejection rate percentage of the membrane with different PEG

Membrane Code	Rejection Rate, %				
	PEG 200	PEG 400	PEG 600	PEG 800	PEG 1000
CHI-1	40.0	50.0	58.0	64.0	90.0
CHI-2	34.0	54.0	44.8	68.0	80.0
CHI-3	31.8	40.0	46.0	71.8	87.0
CHI-4	28.8	48.2	50.0	75.8	84.6

From both of Table 4.2 and Table 4.3 above, it can be seen that the less permeation concentration, the rejection rate percentage is much higher. This may due to the more molecular weight of the PEG used. When the permeability less change, it can say that there are no strong membrane-chitosan interactions occurs.

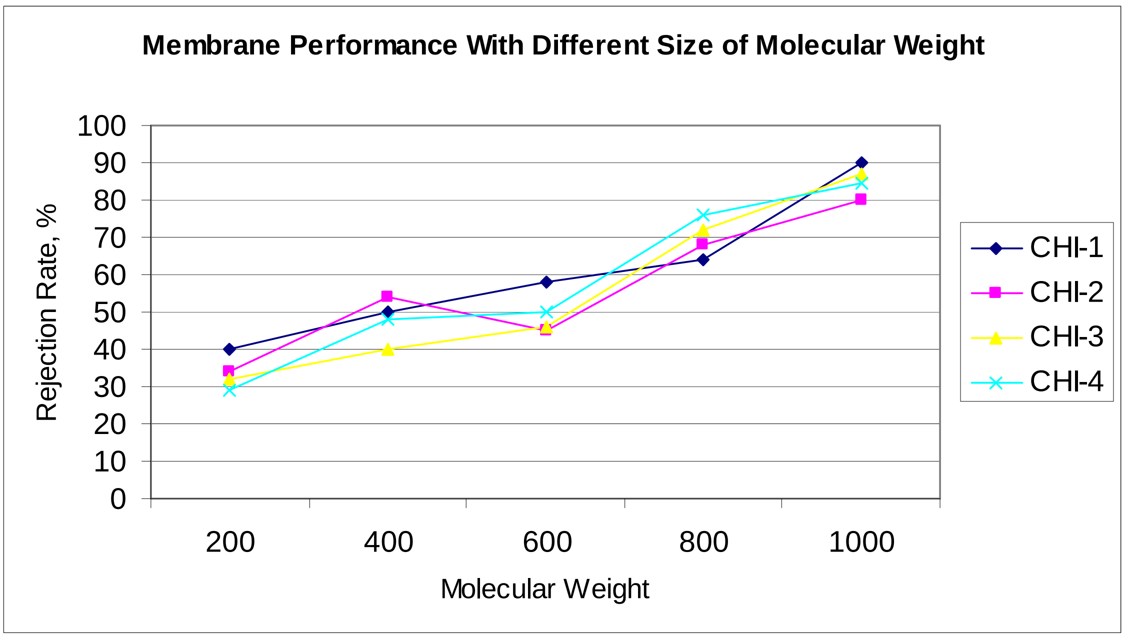

Figure 4.2: The membrane performance with different size of molecular weight

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In order to determine the MWCO of the membrane produced, the graph of rejection rate percentage versus molecular weight of PEG was draw. The MWCO is typically defined as the retained mass of the PEG that is 90% rejected by the membrane. It was observed that in Figure 4.2 the molecular weight cut off of the CHI-1 produced is 1000 where it crosses the 90% of the rejection rate. The molecular weight cut off for the CHI-2, CHI-3 and CHI-4 can not be done due the problems with the PEG. To obtain the molecular weight for the other membrane, more PEG with the higher molecular weight is to be use. The rejection rate of the chitosan membrane increased almost linearly with increase in molecular weight of the PEG used. The high molecular weight PEG is highly soluble than lower molecular weight PEG and therefore, it can be washed out together with the solvent from the membrane film to the coagulation bath or vice versa. The rates of permeation of higher molecular PEG are much slower than the lower PEG. Therefore, the higher molecular weight PEG in solvent take more time to reach and this will give ample time for the polymer aggregates on top of it to form a thicker and dense layer.

4.4 Oil Swelling of Chitosan Membrane

To analyze the oil content in oily waste water, method of swelling was used to determine the oil percentage after treated by fabricated chitosan membrane. The dry weight of chitosan membrane is 0.3 g each of the membrane. The wet weight of the chitosan membrane after swelling in POME was measured every 10 minutes for about 5 times. The result is show in Table 4.4.

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Table 4.4: The wet weight of the chitosan membrane after swelling in POME

Membrane Code	Oil Weight (gram)				
	t=10 min	t=20 min	t=30 min	t=40 min	t=50 min
CHI-1	0.3113	0.3133	0.3229	0.3272	0.3304
CHI-2	0.3400	0.3946	0.4170	0.4633	0.4643
CHI-3	0.3300	0.3846	0.4070	0.4533	0.5115
CHI-4	0.4480	0.5230	0.6660	0.8194	0.9834

The oil swelling percentage each of the fabricated membrane was determined using the equation 2. The result of oil swelling percentage, are listed in Table 4.5 and all the calculated to determine percentage of the oil swelling using the fabricated membrane are shows in appendix B.

Table 4.5: Percentage of the oil swelling using the fabricated membrane

Membrane Code	Degree of Oil Swelling (%)				
	t=10 min	t=20 min	t=30 min	t=40 min	t=50 min
CHI-1	3.76	4.43	7.63	9.07	10.13
CHI-2	13.30	31.53	39.00	54.43	54.77
CHI-3	10.00	28.20	35.67	51.10	70.50
CHI-4	49.33	74.33	122. 00	173.13	227.80

Table 4.5 shows the calculated the degree of oil swelling for all analyzed chitosan membrane. One can observed an increase in swelling every 10 minutes each of the fabricated membrane. This is due to the chitosan compositions added in the membrane.

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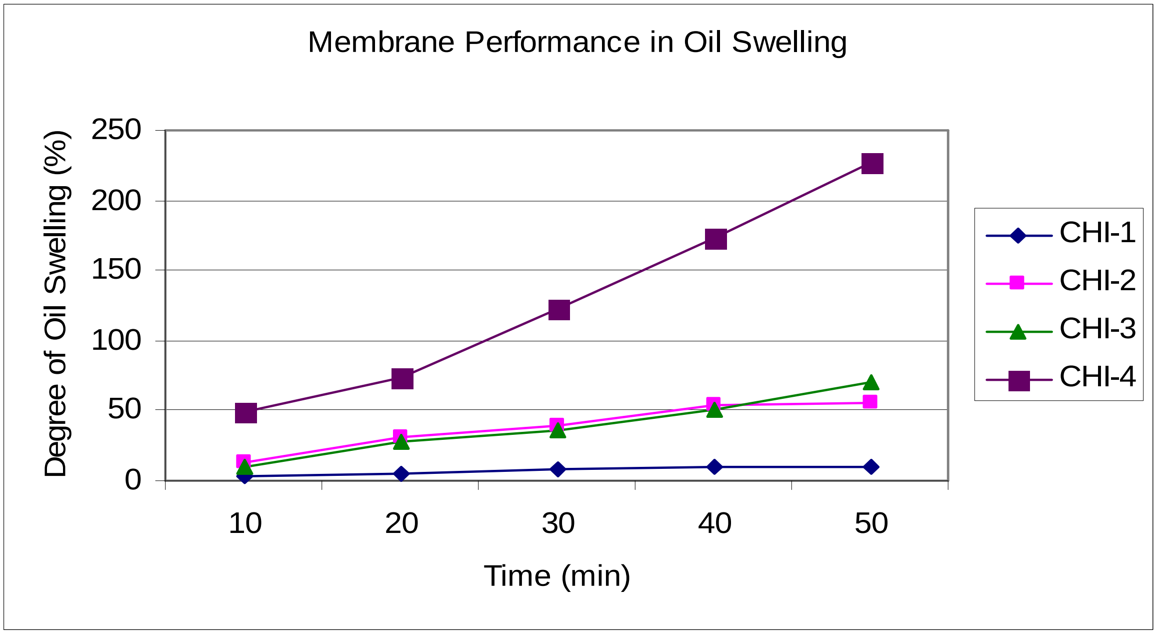

Figure 4.3: Membrane performance in oil swelling

The results from the oil swelling of the fabricated membrane, at different time are plotted in Figure 4.3. The performance of the fabricated membrane can be explained by the degree of oil swelling of the membrane. That is, the degree of oil swelling of the membrane increase with increasing chitosan compositions added in the fabricated membrane. The degree of oil swelling for four of the fabricated membrane increased initially with submersion period but for the CHI-1 it became less unchanged after a certain time period. This may due process erosion which means that, although chitosan is not water soluble, at longer times a small amount of this material could be solubilized, causing the observed weight loss. For CHI-2 and CHI-3, the degree of oil swelling is increase every interval minutes as shows in Figure 4.3 but it can be seen that obviously, the swelling of the CHI-4 increased very rapidly in minutes 20 and more increased in next minutes after with the highest degree of oil swelling about 227.80 %. Such increase in oil swelling in CHI-4 could be caused by the more of the chitosan composition added in the fabricated membrane. This obviously increasing of oil swelling may due to the electrostatic repulsion between the different charge of oil waste (anionic) and chitosan (cationic). The non protonated amino groups in chitosan, having unshared electron pair on the nitrogen atom, are capable of forming donors bond with the anionic from the oily

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waste. Thus, the more the chitosan compositions added in the fabricated membrane, the higher the degree of oil swelling and the more effective the membrane performance in treating the oily wastewater problems.

4.5 Problems Encountered During the Experiment

There are some of the problems encountered while doing the experiment since the starting. All of the problems somehow affected the fabricated membrane produced, the result for the characterization of the membrane and also result for the treating the oily wastewater. The problems encountered are:-

1. When fabricate the chitosan membrane, the cross linking agent which is Tetraetroxysilane are supposed to be added in the dope solution. The function of the Tetraetroxysilane is to produce a high strength and resilient fabricated chitosan membrane but unfortunately, Tetraetroxysilane are not available in our lab while prepared the membrane. This cross linking agent just now arrived one week before the due date to finish the experiment. Due to that problem, I am just started the experiment without the Tetraetroxysilane. Because of that, the membranes produced are low strength and more brittle.

2. Another problem is the chitosan powder, which is the main material in this experiment. Due to the same reason of the Tetraetroxysilane, chitosan powder also arrived late. I am just used the chitosan flakes, blended it first to make it more smaller size than before. I found that, the chitosan flakes are hard to soluble in acetic acid even it stirred for quite long time and as a result, the dope solution prepared are not good enough. The surface of fabricated membrane used by chitosan flakes is not smooth as can be seen in Figure 4.4.

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Figure 4.4: Fabricated membrane using the chitosan flakes

3. The fabricated membrane without the cross linking agent, resulted it became brittle and low strength. Because of that problem, the method recently used to analyze the oil content had been changed to the oil swelling. This due to the fabricated membrane, when filtered with POME waste, it split into a pieces. The water analyze can not be done because of that problem and changed to swelling test.

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CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The fabricated of the chitosan membrane is based on the solvent casting method. Four fabricated membrane was produced here to obtain the favorable fabricated chitosan membrane to treat the oily wastewater. The SEM results shows that the fabricated membrane is asymmetric membrane where the pores each of the membrane are not uniform and have a micro pore size. The MWCO of the fabricated chitosan membrane is 1000, where it crosses the 90% of the rejection rate percentage. From the method of oil swelling showed a result that CHI-4 is with degree of oil swelling up to 227.80 % is the favorable chitosan membrane to treat the oily problem. This fabricated membrane has a higher degree of oil swelling compare to the others. By using the fabricated chitosan membrane, the oily wastewater problem can be solved easily and it safely to the environment beside it more economic compared to the others method to treat the oily wastewater.

5.2 Recommendations

Even though a through study was done to fabricate the most favorable chitosan membrane, there still room for improvement and modification to be taken.

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It is recommended that, to fabricate the high strength and resilient chitosan membrane the cross-linking agent are supposed to be added. This is to increase the performance of the membrane.

The POME waste that be used in this research as oily waste, should be replaced to waste cooking oil or water mix with the oil, where the oil content is more higher compare to the POME. Because the oil percentage in the POME waste is about 16% only. Thus, the result of oil swelling is more accurate.

Last but not least, to enhance the solubility in water, chitosan can be modified by chemical reaction or copolymerized with hydrophilic polymers such as poly ethylene glycol (PEG). PEG is the most interest, owing to it hydrophilicity, biocompatibility and biodegradability.

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APPENDIX A

CALCULATION FOR MOLECULAR WEIGHT CUT OFF

The rejection rate percentage each of the fabricated membrane was determined using the equation as equation 1:-

For CHI-1

1. PEG 200

$$\text{Rejection rate (\%)} = \left[1 - \frac{Cp}{Cf}\right] \times 100 \qquad (1)$$

Where *cp* = 5.00 mg/L and *cf* = 3.00 mg/L.

Solving these equations simultaneously,

The rejection rate (%) = 40 %

2. PEG 400

$$\text{Rejection rate (\%)} = \left[1 - \frac{Cp}{Cf}\right] \times 100$$

Where *cp* = 5.00 mg/L and *cf* = 2.50 mg/L.

Solving these equations simultaneously,

The rejection rate (%) = 50 %

3. PEG 600

$$\text{Rejection rate (\%)} = \left[1 - \frac{Cp}{Cf}\right] \times 100$$

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Where *cp* = 5.00 mg/L and *cf* = 2.90 mg/L.

Solving these equations simultaneously,

The rejection rate (%) = 58 %

3. PEG 800

Rejection rate (%) = $\left[1-\frac{Cp}{Cf}\right]\times 100$

Where *cp* = 5.00 mg/L and *cf* = 1.80 mg/L.

Solving these equations simultaneously,

The rejection rate (%) = 64 %

4. PEG 1000

Rejection rate (%) = $\left[1-\frac{Cp}{Cf}\right]\times 100$

Where *cp* = 5.00 mg/L and *cf* = 0.50mg/L.

Solving these equations simultaneously,

The rejection rate (%) = 90 %

For rejection rate percentage for CHI-2, CHI-3 and CHI-4 membrane are calculated in similar manner.

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APPENDIX B

CALCULATION FOR DEGREE OF OIL SWELLING

The degree of oil swelling each of the fabricated membrane was determined using the equation as equation 2:-

<u>For CHI-1</u>

1. At t=10 min

Degree oil swelling (%) $= \frac{(Wst - Wdt)}{Wdt} \times 100$ (2)

Where Wdt = 0.30g and Wst = 0.3113g

Solving these equations simultaneously,

Degree oil swelling (%) = 3.76 %

2. At t=20 min

Degree oil swelling (%) $= \frac{(Wst - Wdt)}{Wdt} \times 100$

Where Wdt = 0.30g and Wst = 0.3133g

Solving these equations simultaneously,

Degree of oil swelling (%) = 4.43%

3. At t=30 min

Degree oil swelling (%) $= \frac{(Wst - Wdt)}{Wdt} \times 100$

Where Wdt = 0.30g and Wst = 0.3229g

Solving these equations simultaneously,

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Degree of oil swelling (%) = 7.63%

4. At t=40 min

Degree oil swelling (%) $= \frac{(Wst - Wdt)}{Wdt} \times 100$

Where $Wdt = 0.30$g and $Wst = 0.3272$g

Solving these equations simultaneously,

Degree of oil swelling (%) = 9.07%

5. At t=50 min

Degree oil swelling (%) $= \frac{(Wst - Wdt)}{Wdt} \times 100$

Where $Wdt = 0.30$g and $Wst = 0.3304$g

Solving these equations simultaneously,

Degree of oil swelling (%) = 10.13 %

Degree oil swelling percentages for CHI-2, CHI-3 and CHI-4 membrane are calculated in similar manner.

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