FABRICATION OF CHITOSAN BLEND POLYVINYL ALCOHOL(PVA) MEMBRANE WITH DIFFERENT CONCENTRATION OF PEG 400 AS ADDITIVE

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JUDUL **FABRICATION OF CHITOSAN AND POLYVINYL ALCOHOL BLEND MEMBRANE WITH DIFFERENCE CONCENTRATIONS PEG 400 AS ADDITIVE**

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WINETTA ARIANTY BT ARIFFIN

A report submitted in partial fulfilment
of the requirements for the award of the degree of
Bachelor of Chemical Engineering (Biotechnology)

Faculty of Chemical and Natural Resources Engineering
Universiti Malaysia Pahang

MAY 2008

STUDENT'S DECLARATION

I declare that this thesis entitled "*Fabrication of Chitosan Blend Polyvinyl Alcohol (PVA) Membranes with Different Concentrations of PEG 400 as additive*" is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree

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To my beloved mother and father

Allahyarham Muhalifin @ Ariffin Arif

Jausin @ Jacy bt Kijai

And my dearest siblings

Fandey McCoy bin Ariffin Arif

Allahyarham Arie Renaldo bin Ariffin Arif

Della Ferianna bt Ariffin Arif

Norma Jean bt Ariffin Arif

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ABSTRACT

In this research, Chitosan and Polyvinyl alcohol (PVA) blend membrane is made by adding additive which is polyethylene glycol or known as PEG and PEG concentration used in this research was PEG 400. The objective of this research is to study the performance and morphology of Chitosan and PVA blend with PEG 400 as additive using SEM, AFM, pure water permeation (PWP) and solute separation permeability. The membrane was prepared using dope solution method by blending Chitosan, PVA and PEG 400 together. Two difference concentration of PEG 400 which is 4 wt% and 6 wt% are use. Casting method take part after the blending process was finished. After the membrane is dried, it would be easy for the membrane to be peeled off. The membrane was characterized and distinguished by its performance in term of its pure water permeation (PWP) and solute separation and by its morphology using SEM and AFM. From the results, the pure water permeation rate of the membrane is higher by using high concentration of PEG 400 because of the increasing of the hydrophilicity of the membrane but decreasing its selectivity in solute separation. The morphology of the membrane at its cross-section can be seen using scanning electron microscopy (SEM). Characterization of membrane base on its surface section is done using atomic force microscopy (AFM).

ABSTRAK

Dalam kajian ini, Chitosan dan Polyvinyl alcohol(PVA) dibuat dengan menambahkan bahan tambahan iaitu Polyethylene Glycol atau lebih dikenali sebagai PEG dan berat molekul PEG yang digunakan dalam kajian ini ialah 400. Objektif kajian ini ialah untuk mengkaji prestasi dan morfologi campuran Chitosan dan PVA dalam membran dengan PEG 400 sebagai bahan tambahan melalui SEM, AFM, penghasilan air bersih dan pemisahan bahan terlarut. Membran disediakan dengan menggunakan kaedah larutan Dope dengan cara mencampurkan Chitosan, PVA dan PEG 400 bersama-sama. Dua kepekatan PEG 400 akan digunakan sebagai parameter iaitu 4% peratus berat dan 6% peratus berat. Kaedah Casting (pembentukan dalam acuan) atau penghasilan helaian membran berlaku selepas proses pencampuran selesai. Selepas membran itu kering, ia memudahkn membran itu untuk di pisahkn daripada acuannya. Membran dicirikan dan dibezakan melalui prestasinya dari segi ketelapan membran melalui bilangan air yg boleh lalu melalui membran tersebut dan kadar pemisahan bahan terlarut dan juga melalui morfologinya dengan menggunakan SEM dan AFM. Kadar ketelapan membran melalui peningkatannya jumlah air yg lalu melalui membran tersebut membuktikan bahawa interaksi membran tersebut terhadap air adalah tinggi apabila kepekatan PEG 400 ditingkatkan. Morfologi membran dapat dilihat dengan menggunakan SEM. Manakala untuk melihat morfologi permukaan membran tersebut, penggunaan AFM adalah yang terbaik.

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LIST OF SYMBOLS

PVA	-	Polyvinyl Alcohol
CA	-	Chitosan
PEG 400	-	Polyetylene glycol 400
BSA	-	Bovine serum albumin
SEM	-	scanning electron microscopy
AFM	-	Atomic force microscopy
J	-	Flux through the membrane
V	-	Volume
A	-	Area
Wt	-	Weight percent
Ppm	-	Part per million
g/L	-	Gram per litre
µg/L	-	Microgram per litre
PWP	-	Pure water permeation

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CHAPTER 1

INTRODUCTION

1.1 Background of study

These days, industries focus more on membrane technology for separation process especially in the chemical industry in order to overcome several constraints associated with conventional techniques (*Razdan et al, 2003*). It has been found to be an attractive and most suitable alternative approach for separation because the process is faster, energy efficient and does not involve any phase change (*Idris et al, 2006*). The advantage of having membrane is their selective permeation (*Razdan et al, 2003*). Such membranes have applications in petroleum, chemical, pharmaceutical, fertilizer industry, treatment of waste stream, effluent and hazardous streams and vice versa(*Razdan et al, 2003*).

To carry out such applications, it needs type of membrane that has strong mechanical strength where it can withstand a high pressure and high temperature and didn't change its chemical structure when applied to it. Separation system by permeation through polymeric and organic blend membrane can carry out the entire characteristic needed in industrial membrane technology. Blend membrane are prepared by phase inversion technique by casting it first. The presence of additive such as PEG 400 is used in order to enhance the effectiveness of the membrane and to adjust and modified the structure of the membrane property.

Polymer which is Polyvinyl alcohol or known as PVA is a non-toxic, water soluble, bio-compatible and biodegradable synthetic polymer have been widely used in biomedical field(*Jia et al, 2007*). PVA has been better fiber-forming and highly hydrophilic properties and its fibers have been commercialized since the 1950s(*Jia et al, 2007*). For organic material, Chitosan (poly-β(1,4)-D-glucosamine) a cationic polysaccharide, is obtained by alkaline deacetylation of chitin, the principle exoskeletel component of crustaceans(*Yang et al, 2004*). Chitosan has its own advantages such as water binding capacity, fat binding capacity, bioactivity, biodegrability, nontoxicity, biocompatibility and antifungal activity. By blending these polymer and organic component, a membrane can be made.

CS and PVA were blended together in dope solution and fabricate using casting method. In order to enhance the permeability of flux, additive was added which is polyethylene glycol 400 (PEG 400). PEG 400 can be used very well as polymeric additive (*Idris et al, 2007*). It was also reported that PEG 400 also act as microvoids suppressor and give the membrane its hydrophilic character (*Idris et al, 2007*). Acetic acid was used as the co-solvent for CS and and distilled water for PVA in order to prepare the dope solution and NaOH solution was used as both the external and internal coagulants. The separation performance of the blending of CS/PVA blend membrane was evaluated through the separation of bovine serum albumin (BSA) and Lysozime in water.

1.2 Problem Statement

Chitosan and Polyvinyl alcohol (PVA) is already widely used especially in membrane separation. Although its readily known, the suitability of these Chitosan by blending it with Polyvinyl alcohol (PVA) to form blend membrane still not enough because of its non-compatibility between these two components(*Yang et al, 2004*). To overcome this problem, crosslinking between these two components have to be done. For these purpose, formaldehyde is used as crosslink agent (*Yang et al, 2004*). Blending of two components into a membrane to separate mixture in high percentage of separation

also has its disadvantages. In order to overcome this problem, additive is used to increase the separation between mixtures. For this research, PEG 400 is selected to improve the permeation, to improve flux, reduce fouling and the most important is to improve separation.

1.3 Objectives of the Project

The objective of this project is to study the performance and morphology of Chitosan blend Polyvinyl alcohol (PVA) membranes with PEG 400 as additive by using Scanning electron microscopy (SEM), Atomic force microscopy (AFM), pure water permeation(PWP), and solute separation permeability.

1.4 Scopes of the Project

The scope of this research are listed:

i. Blending Chitosan and Polyvinyl alcohol (PVA) blend membrane with PEG 400 as additive.
ii. Characterize the membrane performance by using bovine serum albumin and lysozime as a solute in solute separation permeability and in pure water permeation (PWP).
iii. Characterize the morphology of Chitosan blend Polyvinyl alcohol (PVA) membrane using Scanning electron microscopy (SEM) and Atomic force microscopy (AFM).

CHAPTER 2

LITERATURE REVIEW

2.1 Definition of membrane

Membrane is a permeable or semi-permeable phase, often a thin polymeric solid, which restricts the motion of certain species (*Scott and Hughes*, 1997). This added phase is essentially a barrier between the feed stream for separation and one product stream. This membrane or barrier controls the relative rates of transport of various species through itself and thus, as with all separations, gives one product depleted in certain component at the feed stream and a second product concentrated in the product stream component (*Scott and Hughes*, 1997). The main uses of membranes in industry are (*Scott and Hughes*, 1997):

i. The filtration of micron and submicron size suspended solid (and dispersed liquid) from liquid and gases containing dissolved solid.
ii. The removal of macromolecules and colloids from liquids containing ion species.
iii. The separation of mixtures of miscible liquid.
iv. The selective separation of gases and vapors from gas ad vapor stream.
v. The selective transport of ionic species only.
vi. The virtually complete removal of all material suspended and dissolved from water.

Generally these membranes can be classified into three types (*Scott and Hughes*, 1997):

i. Synthetic polymer;
ii. modified natural product; cellulose-based,
iii. Miscellaneous; include inorganic, ceramic, metals, dynamics and liquid membranes.

Ideal membrane materials posses the following properties (*Cai et al*, 2008):

i. Chemical resistance(to both feed and cleaning fluids)
ii. Mechanical stability
iii. Thermal stability
iv. High permeability
v. High selectivity
vi. Stable operation.

2.2 Membrane Structure

The functioning of the membrane will depend on its structure as this essentially determines the mechanisms of separation and application. Two types of structures are generally found in membrane which is symmetric and asymmetric as following (*Mulder*, 1996):

2.2.1 Symmetric membranes;

i. Symmetric membranes are of three general types which is with approximate cylindrical pores, porous, and non-porous (homogeneous) as shown in figure 1.
ii. It was produced by one of the following method:
iii. Sintering or stretching – for the manufacture of microporous membranes.

iv. Casting – for the manufacture of ion exchange membranes and membranes for pervaporation.

v. Phase inversion and etching – the manufactured metrials function as pore membranes and are used in MF, UF and dialysis.

vi. Extrusion – materials produced by this method function as diffusion membranes for gas permeation and pervaporation.

vii. Microporous membranes are the simplest of all the symmetric membranes in term of principle of operation. Microporous membranes have defined pores or holes and separation is achieved by a sieving action.

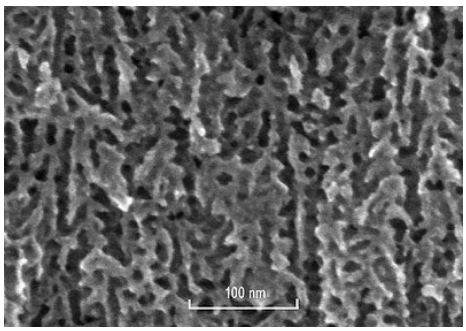


Figure 2.1: scanning electron micrograph cross section in silicon-porous membrane

2.2.2 Asymmetric membranes

Asymmetric membranes are characterized by a non-uniform structure comprising an active top layer or skin supported by a porous support or sublayer(*Schoch et al,* 1976) as shown in figure 2.2 below. There are several types of membrane which is porous, porous with a top layer and composite. Asymmetric membranes was produced either by phase inversion from single polymers or as composite structures (*Ford et al,* 1968). Asymmetric structure can be seen by its wider pores and farther away from the surface to prevent the pores from being plugged (*Ford et al,* 1968). This provides good fouling resistance since foulants have a tendency to either be totally rejected or to pass all the way through the membrane (*Wagner,* 2001)

Phase inversion porous structure is formed by precipitation from homogeneous polymer solution (*Wagner*, 2001). The phase-inversion process membrane is formed by dissolving polymers in a suitable solvent to form a viscous solution which is cast to form a asymmetric membrane (*Razdan et al,* 2003). The membrane then immersed on cold water-bath for phase inversion from sol to porous gel (*Razdan et al,* 2003). The membranes are created with a relatively thick porous support layer range from 0.2 to 0.5mm with a dense active 'skin layer' with thickness less than 1µm(*Wagner*,2001). Various additives can be added to create more suitable pore size and pore-size distribution (*Razdan et al,* 2003).

Composite membranes are different from phase inversion by its skin and support layer because of its different materials. Composite membrane consists of two different layers with different kind of material where the compatibility of these two materials is cross linked together for enhancement of separation and compatibility (*Chen et al,* 2004). This makes a certain amount of tailoring of membrane function for specific applications and thus, gives potential improvements over phase inversion (*Chen et al,* 2004).

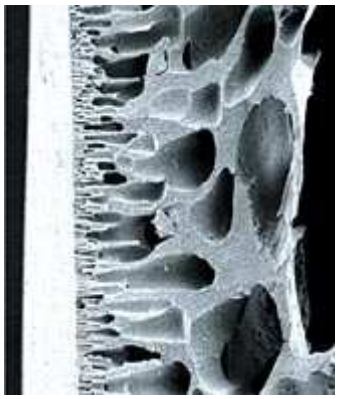


Figure 2.2: Scanning electron micrograph cross-section (50 micron scale) of asymmetric polymer membrane for biohybrid organs based on polyacrylonitrile.

2.3 Membrane process

Membrane processing is a technique that permits concentration and separation without the use of heat (*Goff et al*, 1995). Particles are separated on the basis of their molecular size and shape with the use of pressure and specially designed semi-permeable membranes. When a solution and water are separated by a semi-permeable membrane, the water will move into the solution to equilibrate the system and this is known as osmotic pressure and if a mechanical force is applied to exceed the osmotic pressure such as up to 700 psi(*Goff et al*, 1995), the water is forced to move down the concentration gradient such an example, from low to high concentration. Permeate designates the liquid passing through the membrane, and retentate (concentrate) designates the fraction not passing through the membrane.

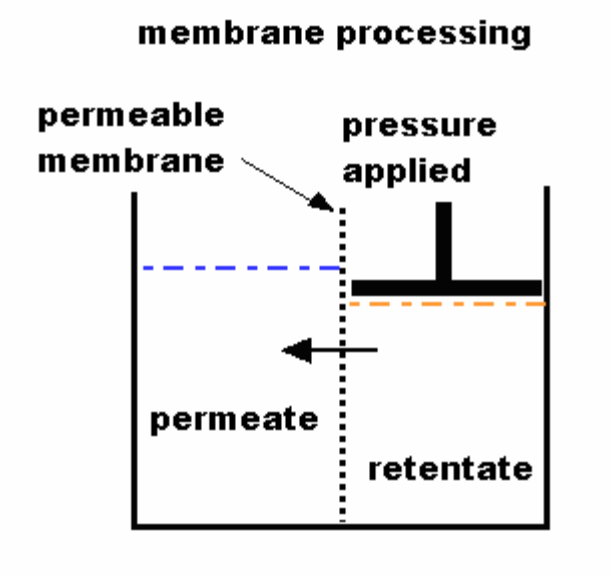


Figure 2.3: diagram on the principle of operation of membrane processing

There are several types of flow in membrane separation which can be used to separate solvent from the solution (*Koros et al*, 1996). These types of flow help to maximize the separation by applying pressure speed of the rotation especially in completely-mix flow separation, and optimum flow rate for absorption of permeate through the membrane (*Goff et al,* 1995). Types of flow are described as below where R

indicates the retentate, P is permeate, F is feed, S is sweep stream which is a nonpermeating stream directed past the downstream membrane face to reduce downstream permeant concentration and where optionally not always present in the separation process and M for membrane(*Koros et al*, 1996):

i. Co-current flow; where the flow pattern through a membrane module in which the fluids on the upstream and the downstream sides of the membrane are moved parallel to the membrane surface and in the some directions.

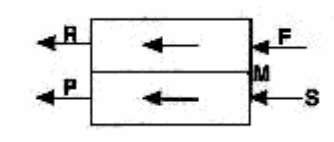


Figure 2.4 : Co-current Flow

ii. Completely-mixed (perfectly-mixed) flow where it flow through a membrane module in which fluids on both the upstream and downstream sides of the membrane are individually well-mixed.

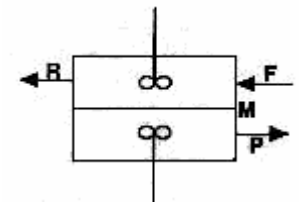


Figure 2.5 : Completely-mixed flow

iii. Counter-current flow where it flow through a membrane module in which the fluids on the upstream and downstream sides of the membrane move parallel to the membrane surface but in opposite directions.

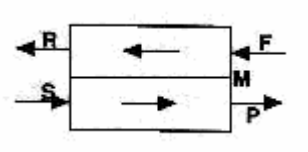


Figure 2.6 : Counter-current Flow

iv. Cross flow where it flow through a membrane module in which the fluid on the upstream side of the membrane moves parallel to the membrane surface and the fluid on the downstream side of the membrane moves away from the membrane in the direction normal to the membrane surface.

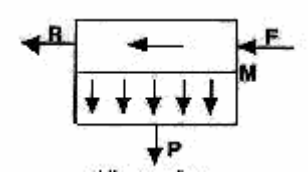


Figure 2.7 : Cross-flow

v. Dead-end flow where it flow through a membrane module in which the only outlet for upstream fluid is through the membrane.

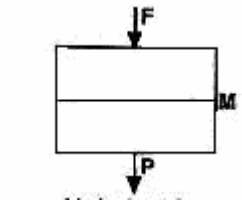


Figure 2.8 : Dead-end Flow

Table 2.1: Lists of membrane process, membrane type, driving force and applications (*Mulder*, 1996):

Membrane process	Membrane type	Driving force	Application
Microfiltration	Symmetric microporous	Hydrostatic pressure	Clarification, sterile filtration
ultrafiltration	Asymmetric microporous	Hydrostatic pressure	Separation of macromolecular solution
nanofiltration	Asymmetric microporous	Hydrostatic pressur	Separation of small organic
Reverse Osmosis (Hyperfiltration)	Asymmetric, composite with homogeneous skin	Hydrostatic pressure	Separation of microsolutes
Gas permeation	Asymmetric or composite, homogeneous or porous polymer	Hydrostatic pressure Concentration gradient	Separation of gas mixture
Dialysis	Symmetric microporous	Concentration gradient	Separation of microsolutes and salts
Pervaporation	Asymmetric, composite	Concentration gradient, vapor pressure	Separation of mixtures of volatile liquids
Membrane distillation	Microporous	Temperature	Separations of water from non-volatile solutes
Electrodialysis	Ion-exchange, homogeneous	Electrical potential	Separations of ions from water
Electrophoresis	Microfiltration membrane	Electrical potential, hydrostatic pressure	Separations of water and ions

2.4 Characterization of membrane

Membrane can be characterized by its pores size (*Jena et al*, 2002). Different membrane process used different type of membrane. The size of particles or molecules retained by the membranes also differs based on the size of the pores in each membrane process. There are two different types of characterization method for porous membranes which is (*Jena et al*, 2002):

i. Structure-related parameters: determination of pore size, pore size distribution, top layer thickness and surface porosity.
ii. permeation-related parameters : determination of the actual separation using solutes that are more or less retained by the membrane (cut-off measurements)

It is often very difficult to relate the structure-related parameters directly to the permeation-related parameters because of the pore size and the shape of the pores is not very well defined. There are techniques to determine and to distinguish the morphology and characterization of the membrane as following:

i. Scanning Electron Microscopy (SEM)
ii. Atom Force Microscopy (AFM)
iii. Pure Water Permeation(PWP)
iv. Solute Separation Permeability

2.4.1 Scanning electron microscopy (SEM)

SEM provides a very convenient and simple method for characterizing and distinguishing the morphology or the membrane porous structure. The principle of SEM can be understood using the figure as shown below (*Goldstein et al*, 1981).

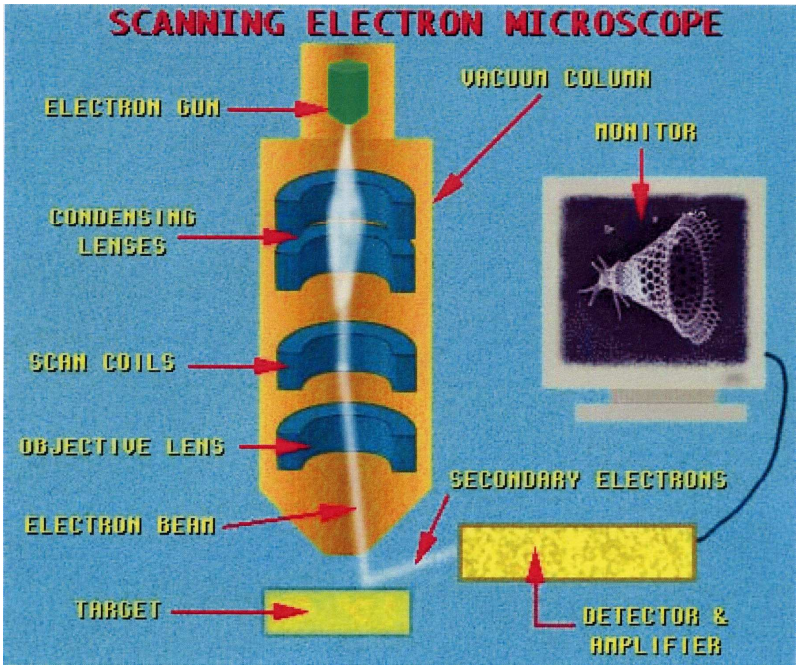


Figure 2.9: Diagram of scanning electron microscopy

A narrow beam of electrons with kinetics energies in the order of 1-25 kV hits the membrane sample (*Danilatos et al*, 1990). This event is called primary (high-energy) electrons, and those reflect are called secondary electrons. Secondary electrons (low-energy) are not reflected but liberated from atoms from the surface; they mainly determine the imaging on the screen or on the micrograph (*Goldstein et al*, 1981). When the membrane or polymer is placed in the electron beam, the sample can be burn or damaged, depending on the type of polymer and accelerating voltage employed (*Goldstein et al*, 1981). This can be avoided by coating the sample with a conducting layer, usually a thin gold layer to prevent charging up of the surface (*Danilatos et al*, 1990). The preparation technique is very important since bad preparation technique give rise to artifacts.

2.4.2 Atomic force microscopy (AFM)

Another technique to distinguish the morphology of the membrane is by using AFM or Atomic Force Microscopy. Atomic Force Microscopy (AFM) uses various forces that occur when two objects are brought within nanometres of each other (*Humphris et al*, 2005). An AFM can work either when the probe is in contact with a surface, causing a repulsive force, or when it is a few nanometres away, where the force is attractive (*F. Giessibl*, 2003). AFM is based on scanning a flexible, force-sensing cantilever across a specimen (*F. Giessibl*, 2003). Attractive and repulsive forces acting on the tiny diving-board-like arm cause deflections that can be measured with laser methods (*F. Giessibl*, 2003). The schematic diagram of AFM is as shown at Figure 5 below:

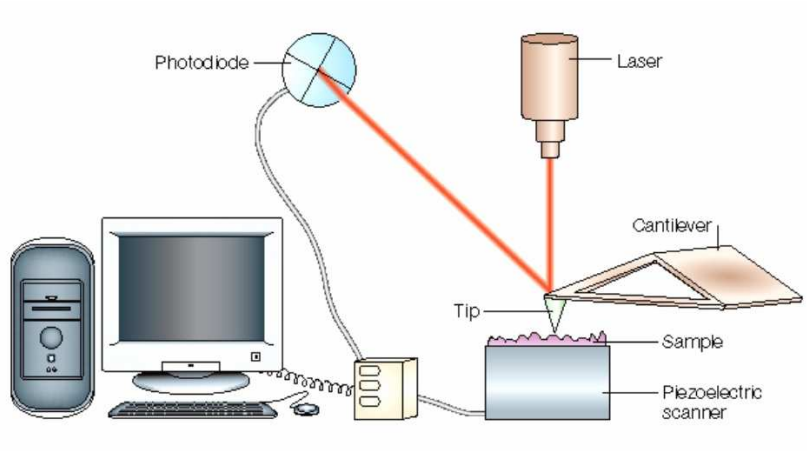


Figure 2.10 : schematic representation of the components of an atomic force microscope (AFM)

2.4.3 Pure Water Permeation(PWP)

Permeability method base on pure water permeation flux at a constant pressure obtain after the separation process can determine the size of the pore on the membrane and also the permeability of the membrane to absorb water. This can be calculated by using the following equation:

$$PWP = \frac{Q}{At} \tag{2.1}$$

Where PWP is the pure water permeation flux through the membrane, Q is the volume of the permeate (L), A for membrane surface area (m^2) and lastly t for time (*Idris et al*, 2006).

2.4.4 Solute separation permeability

Membrane fouling can have a drastic effect on the separation characteristics. Fouling means process resulting in loss of performance of a membrane due to the deposition of suspended or dissolved substances on its external surfaces, at its pore openings, or within its pores (*Koros et al, 1996*). In a solution which contains a large difference in molecular weight, the components which have a smaller molecular weight will be discharge first and the higher molecular weight will retained on the surface of the membrane. Fouling might form and it possible that the component which retained at the surface of the membrane blocks the pores. Solute separation of the membrane by percentages is calculated in order to determine the efficiency of the membrane in separating permeates and retentate. The solute separation of the membrane was given by:

$$R(\%) = \left[1 - \left(\frac{C_p}{C_f}\right)\right] x100 \tag{2.2}$$

Where C_p solute concentration in permeate stream and C_f is solute concentration in feed stream (*Idris et al, 2006*).

The flux (J) in the presence of solute for blend membrane is obtained by:

$$J = \frac{V}{At} \qquad (2.3)$$

Where V is volume of permeate (L), A for membrane surface area (m^2) and lastly t for time (*Idris et al, 2006*).

2.5 Polymer membranes

Membrane for the pressure driven processes are available in a number of different materials including polymer, ceramics, glass and metals(*Scott and Hughes*, 1997). In selecting a material from which to make a membrane destined for commercial use, it must meet:

i. The material must be available. This usually means that the bulk polymer is primarily manufactured for some purpose other than making membranes.
ii. The material must be chemically stable in a range of condition.
iii. The material must be formable. In the case of polymers this usually means that the material must be able to make a stable solution in a suitable solvent, or alternatively to be able to withstand stretching.
iv. The material may need to be approved for food or water contact for certain application.

2.5.1 Poly(vinyl alcohol)

Poly (vinyl alcohol) (synonyms, vinyl alcohol polymer, PVA, ethenol homopolymer) resin is prepared by polymerization of vinyl acetate, followed by partial hydrolysis of the resulting ester in the presence of an alkaline catalyst (*Burford et al,* 1968).

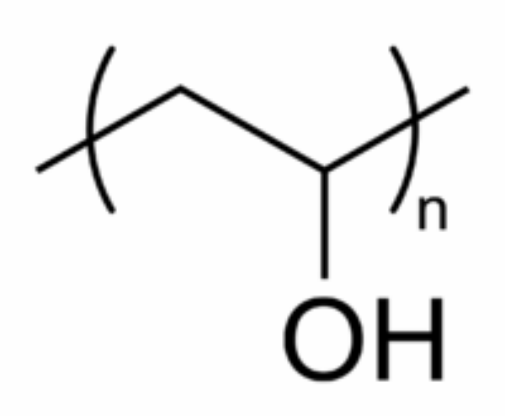


Figure 2.11: Molecular structure of polyvinyl Alcohol

After polymerization, the material undergoes controlled hydrolysis with aqueous sodium hydroxide, during which the ester groups in the vinyl acetate are replaced with hydroxyl groups. Polyvinyl alcohol is precipitated, washed and dried to form an odourless, tasteless, white or cream-coloured granular powder. The primary raw material used in the manufacture of polyvinyl alcohol is vinyl acetate monomer dissolved in methanol. The number of acetate groups in polyvinyl alcohol is determined by the degree of hydrolysis (*Burford et al,* 1968).

Polyvinyl alcohol is used as a coating, binder, sealing and surface finishing agent in food products such as dairy-based desserts, confectionery and cereal products and dietary supplement tablets, in the range of 0.2–1.8% by weight (*Burford et al,* 1968). Polyvinyl alcohol is non-toxic, water soluble, bio-compatible and biodegradable synthetic polymer has been widely used in biomedical field (*Jia et al,* 2007). Polyvinyl alcohol has been better fiber-forming and highly hydrophilic properties and its fibers

have been commercialized since the 1950s (*Jia et al*, 2007). It has high tensile strength, flexibility, as well as high oxygen and aroma barrier. However, these properties are dependent on humidity, in other words, with higher humidity more water is absorbed. The water, which acts as a plasticizer, will then reduce its tensile strength, but increase its elongation and tear strength (*Jia et al*, 2007). Polyvinyl alcohol is an atactic material where essentially all the configurational (repeating) units in its material are identical but exhibits crystallinity as the hydroxyl groups are small enough to fit into the lattice without disrupting it.

Polyvinyl alcohol has a melting point of 230°C and 180–190°C for the fully hydrolysed and partially hydrolysed grades. It decomposes rapidly above 200°C as it can undergo pyrolysis meaning it can be decomposed by heating it in the absence of oxygen or any other reagents, except possibly steam at high temperatures.

2.6 Chitosan

Chitosan(poly-β(1,4)-D-glucosamine) a cationic polysaccharide, is obtained by alkaline deacetylation of chitin, the principle exoskeletel component of crustaceans(*Yang et al*, 2004). The degree of deacetylation (%DA) can be determined by NMR spectroscopy, and the %DA in commercial chitosans is in the range 60 to 100 %. Below are the figures of Chitosan that can be obtain:

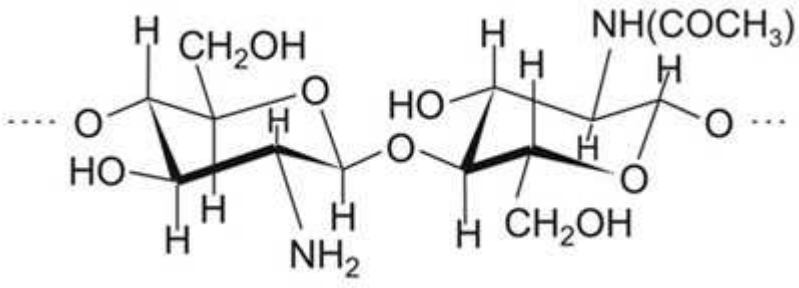


Figure 2.12 : Chemical structure of chitosan

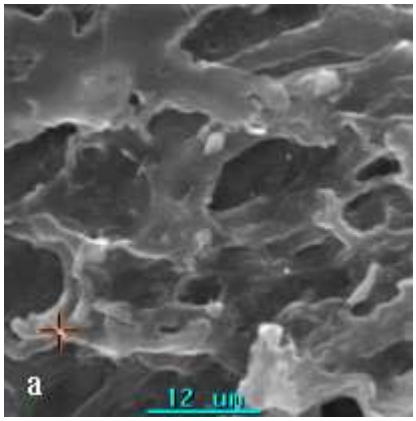


Figure 2.13 : SEM photograph on cross-section of cellulose acetate (CA)

Chitosan is presently under investigation for a wide range of therapeutic applications, such as burn and wound dressings, sutures, bone fillers, engineered tissue scaffolds, and drug and gene delivery vehicles. Chitosan also has its own advantages such as water binding capacity, fat binding capacity, bioactivity, biodegrability, nontoxicity, biocompatibility and antifungal activity (*Yang et al*, 2004).

Chitosan is a linear copolymer of glucosamine and *N*-acetyl glucosamine in a β 1-4 linkage, structurally similar to extracellular-matrix glycosaminoglycans (*Amaral et al*,2006). In chitosan, the molar fraction of N-acetylated units is known as the degree of N-acetylation (DA), which, depending on the extent of deacetylation, can range from 0 to 50%. DA is a structural parameter affecting the charge density, solubility, and propensity to enzymatic degradation, with higher DAs leading to faster biodegradation rate. Like most polysaccharides, chitosan has the ability to elicit specific cellular functions, namely as an immunoadjuvant because of the ability of *N*-acetyl glucosamine residues to attract and activate polymorphonuclear leucocytes inducing the production of cytokines and the subsequent regeneration of connective tissues(*Amaral et al*,2006)

The main interest in chitosan derives from its cationic nature in acidic solutions, which provides unique properties relative to other polysaccharides which are usually neutral or negatively charged. The cationic nature allows chitosan to be used in among

other applications waste-water treatment. Positively charged chitosans reacts with organic solids and cell surfaces which are usually negatively charged. Another useful property is that it is possible to control the solubility and the charge density by varying the mole fraction of N-acetylated units. Regarding potential applications of chitosan in water-solutions, control of the solubility of the chitosans is of primary importance. In general, the solubility of heteroglucans are influenced by the polarity and size of the monomers, distribution of the monomers along the chain, the flexibility of the chain, branching, charge density and molecular weight of the polymer.(*Dalwoo*, 1999)

CHAPTER 3

MATERIAL AND METHODOLOGY

3.1 Introduction

This chapter will briefly explains the method and procedures that was used during the whole process, starting from the preparation of samples, making the membrane, membrane testing, data collection and finally, analyzing the data and results. The flow of the testing process is as shown in Figure 3.1 below.

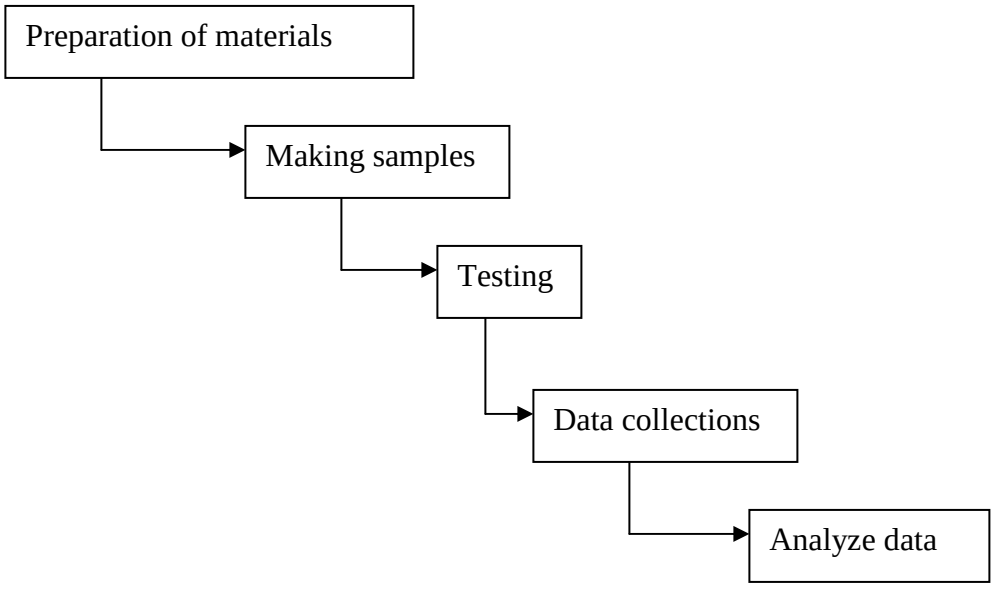


Figure 3.1 : Flow Chart of Methodology

The purpose of this study is to study the characterization and morphology of the Chitosan and PVA blended membrane by adding PEG 400 as additive. Membrane can be made in difference method and ways depends on the type of membrane, purpose and function of the membrane itself. With every method of making the membrane, the performance and morphology will also differs. Here, several methods have been selected in order to study the characterization and morphology of the membrane from selecting the material, making the samples until testing and evaluating the samples.

3.2 Materials

Chitosan powder with degree of N-dacetylation 79% was purchased from R&M Chemical. Polyvinyl alcohol was purchased from Merck Schuchardt OHG was used as matrix polymer in preparation of membrane. Polyethylene Glycol 400 (PEG 400) with molecular weight of 400 was also purchased from Merck Schuchardt OHG and is used as additive. Bovine Serum Albumins (BSA) with molecular weight of 1,000 to 35,000 Dalton and Lysozyme were used as solutes. Feed solutions were prepared using distilled water. Sodium hydroxide (NaOH) was used as a coagulant in coagulation bath. Other chemical were acetic acid where it was used as a solvent for Chitosan, and formaldehyde and sulfuric scid (H_2SO_4) as a crosslink agent.

3.3 Methodology

3.3.1 Preparation of Chitosan and Polyvinyl alcohol(PVA) Blended Membrane Dope Solution.

Dope solution was prepared by dissolving Chitosan 3 wt% in 1% acetic acid solution at ambient temperature with stirring overnight. The solution was filtered before use. 1 wt% of Polyvinyl alcohol or PVA solution were prepared by dissolving PVA in 80°C distilled water and stir for about 4 hour(*Yang et al, 2004*). Then the mixture of

Chitosan and PVA solution were stirred together with different concentration of Polyethylene Glycol 400 or PEG 400. The mixture then was stirred for 24 hours. After the mixture became homogeneous, the solution then kept into a glass bottle for about 24 hours without stirring to free the air bubbles entrapped in the dope solution, and it was finally filter to remove any insoluble particles.

Sample	Chitosan (wt%)	PVA (wt%)	PEG 400 (wt%)
Sample 1	3	1	4
Sample 2	3	1	6

Table 3.1:Ratio of concentration of Chitosan, PVA and PEG 400 as additive.

3.3.2 Membrane Casting

After the solution is prepared, the solution then cast in a petri dish at ambient temperature for 48 hours. After the membrane completely dry, the membranes were then peeled off from the petri dish. The membranes were soaked in 2.5 wt% of formaldehyde solution with 17 wt% of sulfuric acid (H_2SO_4) aqueous for crosslinking the membrane (*Yang et al, 2004*). The membranes then were soaked into 12 wt% of hyrochloric acid (HCl) aqueous at room temperature for 1 hour to removes traces of Acetic acid in the membrane. The membranes then were washed with distilled water for 24 hours. Dry the membrane in ambient temperature for storage and soaked into the distilled water for further use.

3.3.3 Bovine serum Albumin (BSA) and Lysozyme preparations.

The concentration of BSA and lysozime was determined. The absorbance was run using UV VIS-Spectrophotometer. Concentration of BSA solution in permeates were analyzed using Biuret Reagent. Then it was heated in coagulation bath at 37°C for

about 15 minutes (*Idris et al*, 2007). The colour of the Biuret reagent in BSA solution was allowed to change. The absorbance was tested using spectrophotometer at a wavelength of 595 nm against reagent blank.

3.3.4 Scanning electron Microscopy (SEM)

Chitosan and PVA blend membrane was tasted using EDX Spectrometer model EVO50 from Zeiss. The samples were cut into tiny pieces so that the beam of the SEM can see the cross section of the membrane clearly. The samples were then placed on a sample stand and allow the samples to stand in 90° for clear images. The cross section and surface morphology of Chitosan and PVA blend membrane with PEG 400 were being viewed with high voltage of SEM.

3.3.5 Atomic Force Microscopy (AFM)

The surface morphology (2D and 3D topographic images) were analyzed. The roughness analysis of mean roughness (R_a), the root mean square of data (R_z) and the mean difference in the height between the highest peak and the lowest peak (R_y) for Chitosan and PVA blend membrane with PEG 400 of different molecular weight as additives in scan area were characterized using an atomic force microscopic model Shimadzu SPM-9500J2(*Idris et al*, 2007).

3.3.6 Membrane Performance Evaluation

Pure water permeation (PWP) for the Chitosan and PVA blend membrane with PEG 400 in different concentration was calculated using this equation;

$$PWP = \frac{Q}{At} \qquad \text{(from 2.1)}$$

The measurement of water content of Chitosan and PVA membrane was measured by immersing the blended membrane in distilled water at ambient temperature for 1 hour. The weight of the blended membrane before and after immerse were determine. The absorb water content was then calculated (*Yang et al, 2004*);

$$W\% = \left(\frac{W_t - W_d}{W_t} \right) x100 \qquad (2.4)$$

The solute separation of the Chitosan and PVA blend membrane was given;

$$R(\%) = \left[1 - \left(\frac{C_p}{C_f} \right) \right] x100 \qquad \text{(from 2.2)}$$

The flux of solute for the Chitosan and PVA blend membrane is obtained by;

$$J = \frac{V}{At} \qquad \text{(from 2.3)}$$

The samples of all the membranes were evaluated and tested using all the method given. The tabulated results are the average value. Evaluating all the samples of blended membrane by calculating its performance can determine the effectiveness and strength of the membranes.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

In this chapter, all the method which already been done from the previous chapter will gives the results that needed in order to determine the characterization and performance of the chitosan blend Polyvinyl alcohol (PVA) membranes with PEG 400 as additive. All the results were obtained from scanning electron microscopy (SEM), atomic force microscopy (AFM) and membrane performance evaluations. At the end of this chapter, the effectiveness and selectivity of the membrane by testing the membrane using bovine serum albumin (BSA), lysozyme and pure water permeation (PWP) can be determine and thus, can select which samples is better for separation.

4.2 Pure Water Permeation (PWP)

chitosan blend Polyvinyl alcohol (PVA) membranes with different concentration of PEG 400 have already been tested by using Amicon Cell as the core equipment for determine the total of water permeation. Distilled water was put into the Amicon Cell and Chitosan and PVA membrane at the bottom of the equipment. Pressure was then release into the Amicon Cell and fluxes were collected via time in seconds. Below is the result obtained from the experiment.

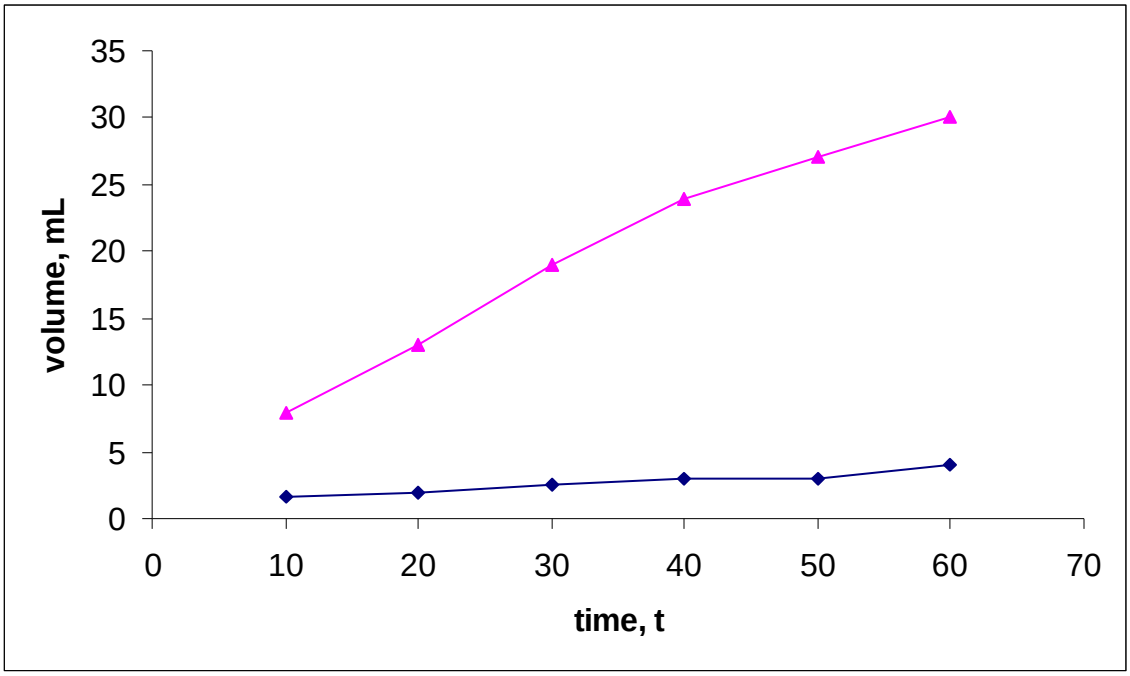


Figure 4.1: Water permeation flux graph obtained using Chitosan blend PVA membrane with different concentration of PEG 400 as additive.

The graph shows that the permeation flux increased with every increasing of time. Membrane with concentration 6 wt% of PEG 400 as indicates in triangle line shows good water permeation flux then membrane with concentration 4 wt% of PEG 400 shown in rectangular line. This is due to the hyrophilicity of the membranes which being effected by the concentration of PEG 400. PEG 400 which has hydrophilic properties makes the membrane became more suitable in absorbing water. With increasing of concentration of PEG 400, the hydrophilicity of the membrane also increased.

To calculate pure water permeation or PWP, equation 2.1 was used and the graph is shown at figure 4.2;

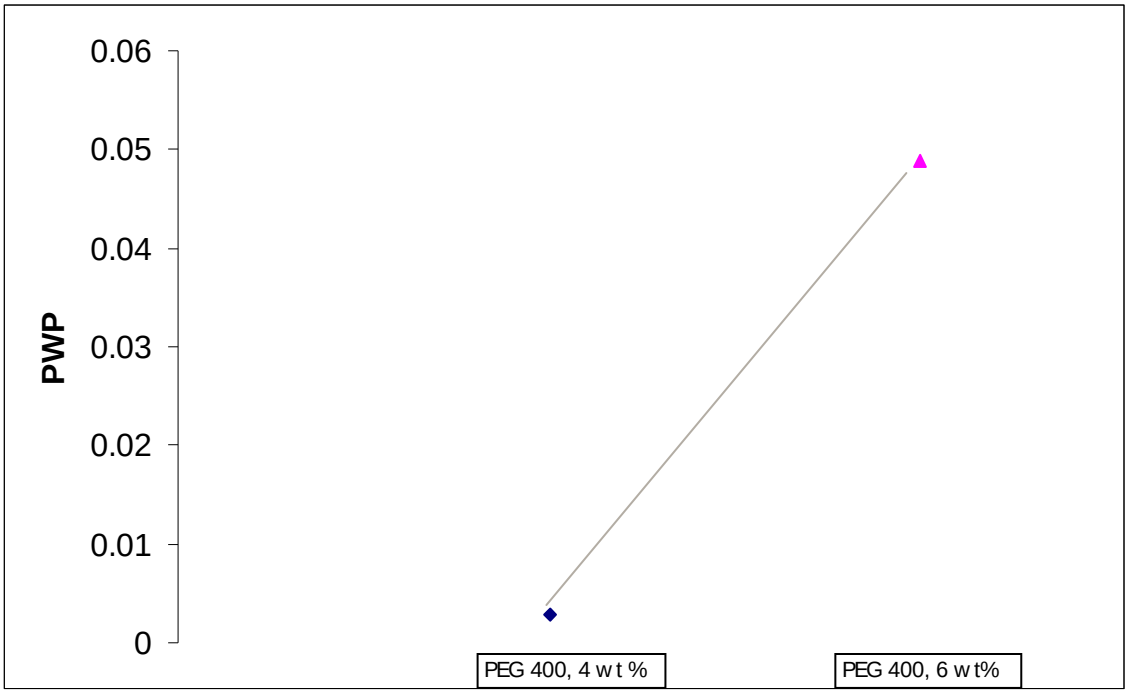


Figure 4.2: Pure water permeation (PWP) graph obtained using Chitosan blend PVA membrane with different concentration of PEG 400 as additive.

From the graph above, the pure water permeation (PWP) were increased as the concentration of the PEG 400 also increased from 4 wt% to 6 wt%. Its clearly shows that the PWP is significantly effected by PEG 400 added in the dope solution in preparing Chitosan and PVA blend membrane. It is observed that higher concentration of PEG 400 in casting solution increased PWP rate. As mention previously, it was due to the hydrophilicity of the membrane. Other reason for the increasing of water permeation and PWP rate is because of the crooslinking in the membrane. Crosslinking between formaldehyde and membrane were done in order to lower the crystallinity of the PVA in the membrane and thus, increased the affinity of the membrane toward water. This also result in increasing the selectivity of membrane toward water and it makes Chitosan blend PVA membrane with different concentration of PEG 400 have greater interaction with water.

4.3 Solute separation permeability

Membrane characterizations were done using Bovine Serum Albumin (BSA) and lysozyme. These protein were flowed across the membrane by using pressure and the permeate flux from the separation process were determine by its optical density before and after the separation process. Below were the optical density of BSA and lysozyme before the processes were begun.

Table 4.1: Optical density of Bovine Serum Albumin (BSA)

Concentrtion (ug/ml)	Absorbance (595 nm)
125	0.1519
250	0.3001
500	0.5524
750	0.7856
1000	0.9289
1250	1.1978
1500	1.4532
1750	1.6554
2000	1.7983

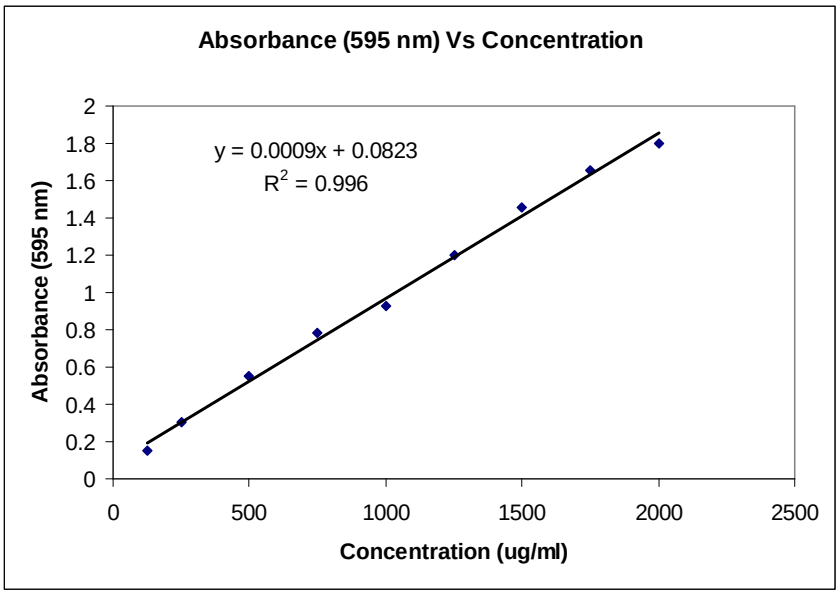


Figure 4.3 : Standard curve of Bovine Serum Albumin (BSA)

Table 4.2: Optical density of lysozyme

Concentration	1st	2nd	3rd	Average
0	0	0	0	0
200	0.232	0.235	0.234	0.23366667
500	0.501	0.503	0.503	0.50233333
1000	0.789	0.788	0.789	0.78866667
1500	1.127	1.126	1.126	1.12633333
2000	1.434	1.433	1.435	1.434

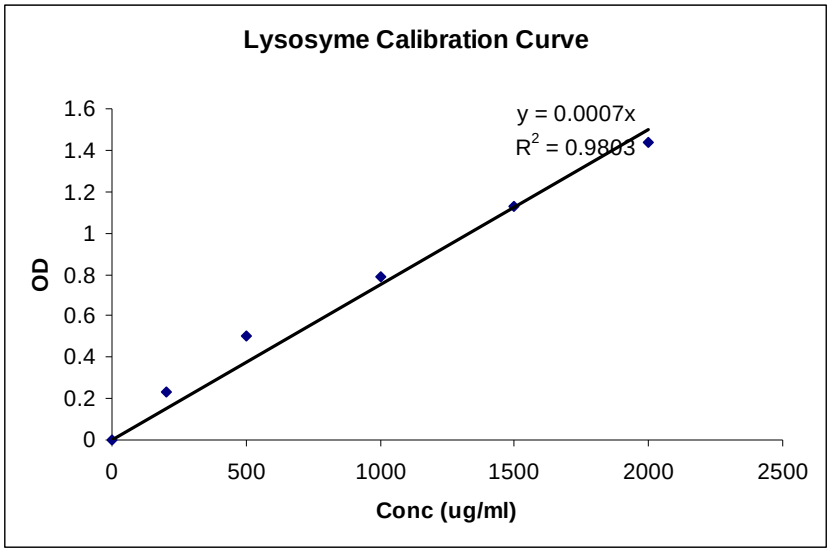


Figure 4.4: Standard curve of Lysozyme

By using standard curve, the concentration of the proteins can be determined after the separation experiment. The concentration before and after the separation processes can identify weather the membrane can or cannot separate solute from water and also to see the efficiency and selectivity of the membrane itself.

Below was the result after the solution contains proteins solute and water in concentration 2000 µg/mL was flowed across the membrane by using pressure in dead-end flow using Amicon Cell.

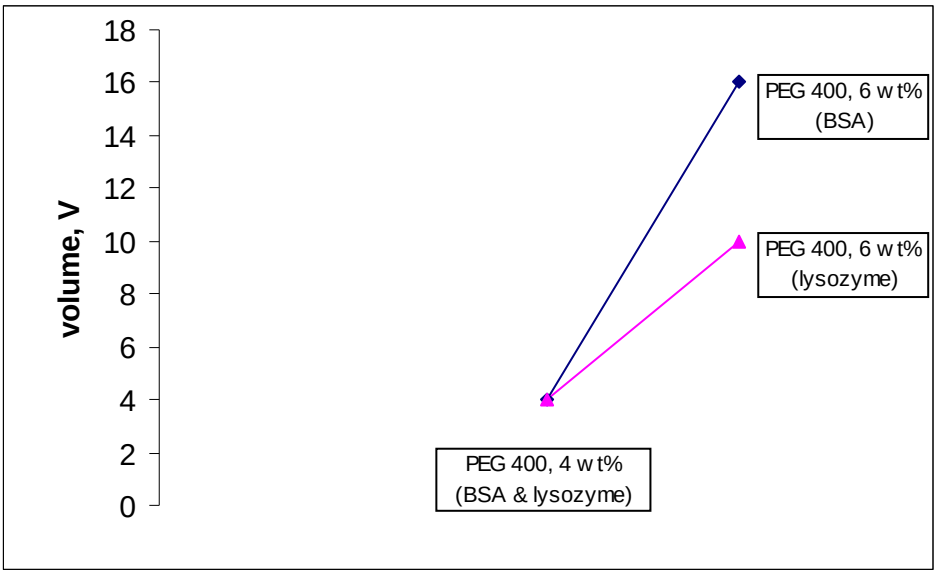


Figure 4.5: Volume of permeate flux after separation process across the Chitosan blend PVA membrane with different concentration of PEG 400.

From figure 4.5, triangle line indicates the increasing of lysozyme volume from 4 mL to 10 mL. While rectangular shows the increasing of BSA volume from 4 mL to 16 mL.

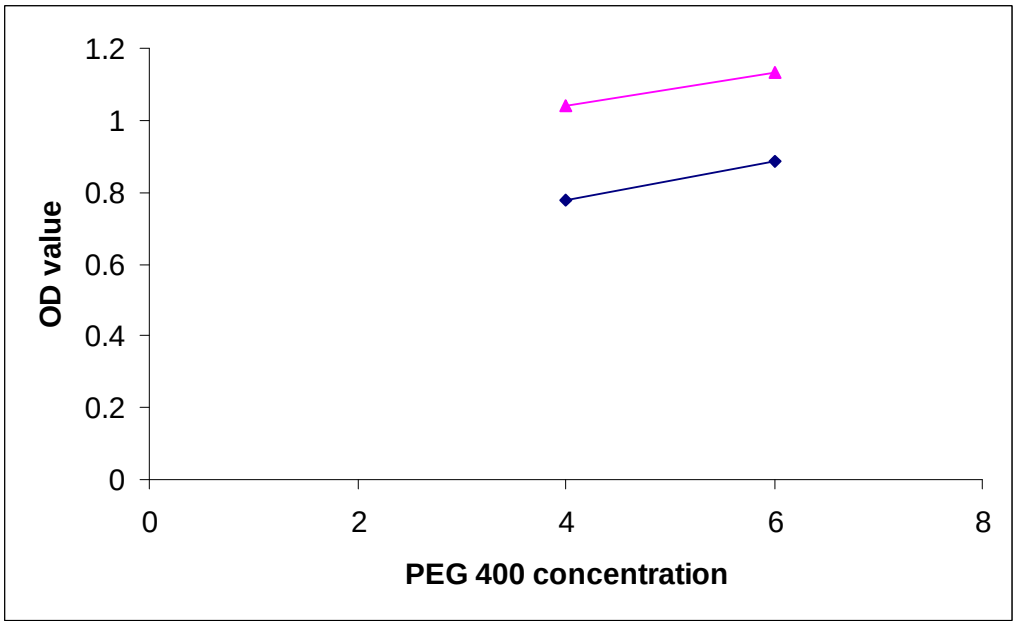


Figure 4.6: Optical density of solutes after separation process across the Chitosan blend PVA membrane with different concentration of PEG 400.

Based on the figure 4.6 above, triangle line indicates the increment of optical density for BSA using membrane containing PEG 400 in 4 wt% and 6 wt% concentrations from 1.042 to 1.1314. As for rectangular line, it shows the increment of optical density for lysozyme using membrane containing PEG 400 in 4 wt% and 6 wt% concentrations from 0.780 to 0.884.

sample	PEG 400 (4 wt%), C_p (µg/mL)	Peg 400 (6 wt%) C_p (µg/mL)
BSA	960	1020
lysozyme	980	1350

Table 4.3: Concentrations of solutes after separation process across the Chitosan blend PVA membrane with different concentration of PEG 400.

After comparing the value from the result from figure 4.6 above with the standard curve of the proteins, the concentration that can be gain from the permeation flux is 960 µg/mL for BSA and 980 µg/mL for lysozyme in concentration of 4 wt% of PEG 400. For concentration of 6 wt% of PEG 400, BSA permeate concentration was 1020 µg/mL and 1350 µg/mL for Lysozyme. Concentration of lysozyme is higher than concentration of BSA due to the molecular weight of both proteins. BSA has molecular weight of 66,430 Daltons meanwhile lysozyme has molecular weight of 14,400 Daltons. The graph of differences in concentrations solutes is shown at figure 4.7 below. One of the purposes of adding PEG 400 is to alter or changes the size of the pores and increased the porosity of the membrane. With increasing of PEG 400 in the dope solution, the pores size of the membrane also increased. But here, BSA which has high molecular weight has difficulty to cross and absorb into the membrane and tend to retain at the surface of the membrane. After a while, fouling occurred at the surface of the

membrane and as well blocks the pores. This explain why the permeate flux concentration is lower than lysozyme. For lysozyme, because of its smaller size compared to BSA, it can cross and absorb more easily through the membrane. Concentration of solute proteins at permeate still can be considered high even though there have different value of solutes concentration before and after the separation processes. Other factor that influences the separation between solute proteins and water was due to the crosslinking of formaldehyde with membrane where it decreases the crystallinity of PVA but also decreases the amine group that contains in Chitosan chemical structures which cause the absorption rate of the membrane became lower. Thus, proteins cannot bond themselves at the surface of the membrane.

Figure for percentages of solute separation obtained based on equation 2.2 is shown at figure 4.8;

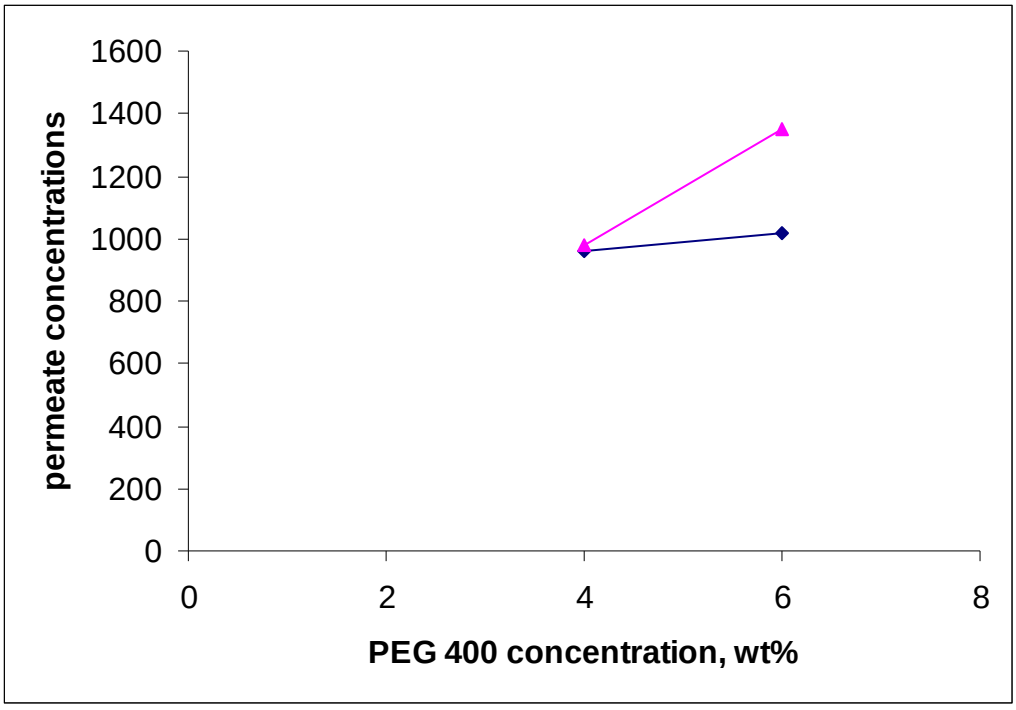


Figure 4.7: Concentration of permeate after separations process using Chitosan blend PVA membrane with different concentration of PEG 400

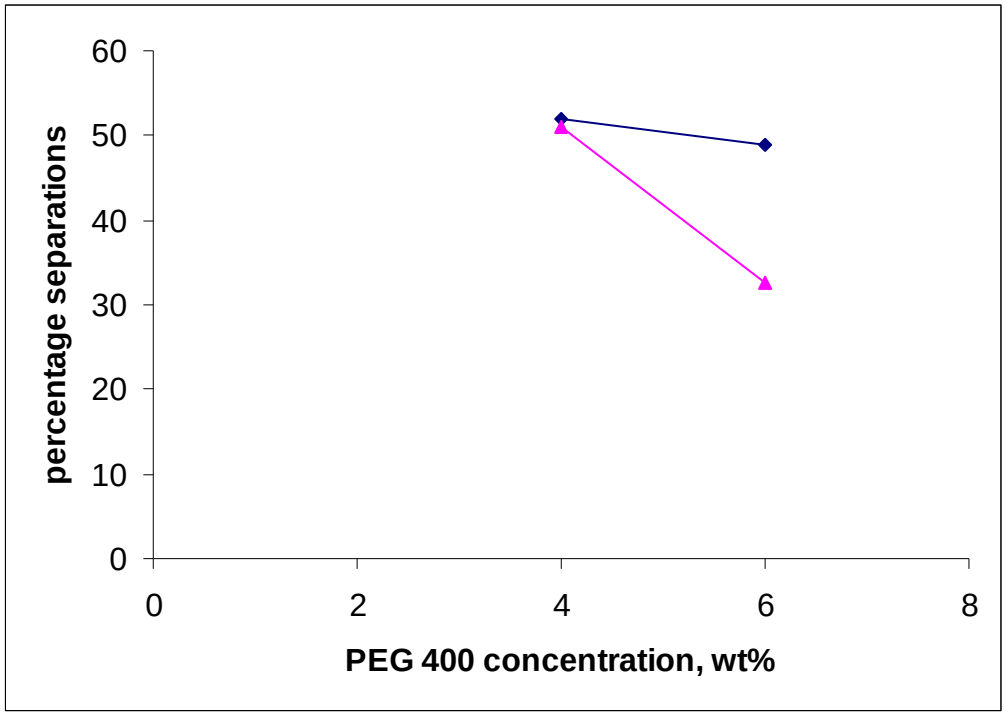


Figure 4.8: percentage of solute separation of permeate after separations process using Chitosan blend PVA membrane with different concentration of PEG 400

For figure 4.8 above, triangle line shows the decreasing of percentage solute separation in membrane containing PEG 400 concentrations in 4 wt% to 6 wt% from 52% to 49% of separation for BSA. Meanwhile, rectangular line indicates the decreasing of percentage solute separation in membrane containing PEG 400 concentrations in 4 wt% to 6 wt% from 51% to 32.5% of separation for lysozyme. The percentage separation from 4 wt% concentration of PEG 400 is higher then 6 wt% of PEG 400. Due to smaller pores size of the membrane, the selectivity and separation factor increased even though the permeation flux decreased. This makes the membrane with 4 wt% concentration of PEG 400 has its advantages in separating solute even though the permeation flux is lower.

The flux (J) in the presence of solute for blend membrane is obtained from equation 2.3 and flux rate graph is shown in figure 4.9 below:

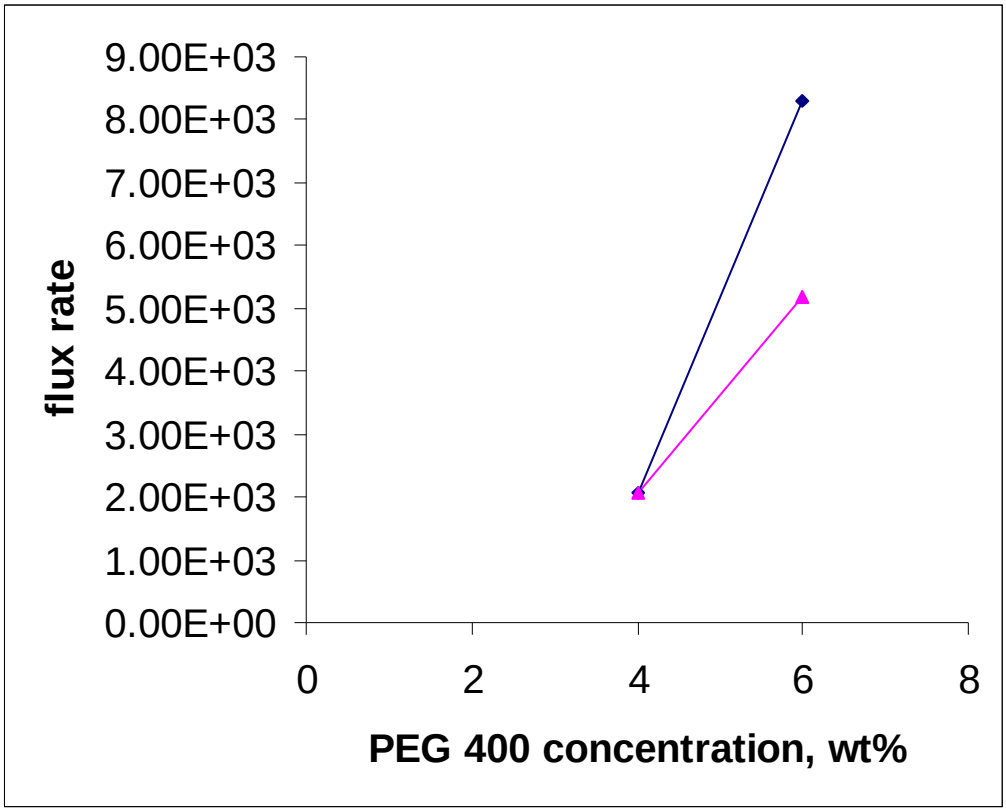


Figure 4.9: Flux rate graph obtained after separation processes using Chitosan blend PVA membrane with different concentration of PGE 400

From the graph above, triangle line indicates the increment of flux rate using 4 wt% and 6 wt% of concentration of PEG 400 for BSA. While rectangular line indicates the increment of flux rate using 4 wt% and 6 wt% of concentration of PEG 400 for lysozyme. This makes the membrane having 6 wt% of PEG 400 has good advantages in obtaining higher flux rate. With differences concentrations of PEG 400 in membrane, the differences of pores size also implied. This means, 4 wt% of PEG 400 in membrane have good solute separation but lower results in flux rate while for 6 wt% of PEG 400 in membrane makes the flux rate to increase but also results in poor solute separations.

4.4 Effect of additive on membrane morphology using Scanning Electron Microscopy (SEM)

The SEM images of Chitosan and PVA blend membrane with different concentration of PEG 400 as additive were shown in Figure 4.1 (a) and (b). The morphology of the blend membrane changed as the crosslinking between formaldehyde and membrane occurred and as the formation of membrane structure by adding PEG 400 as additive.

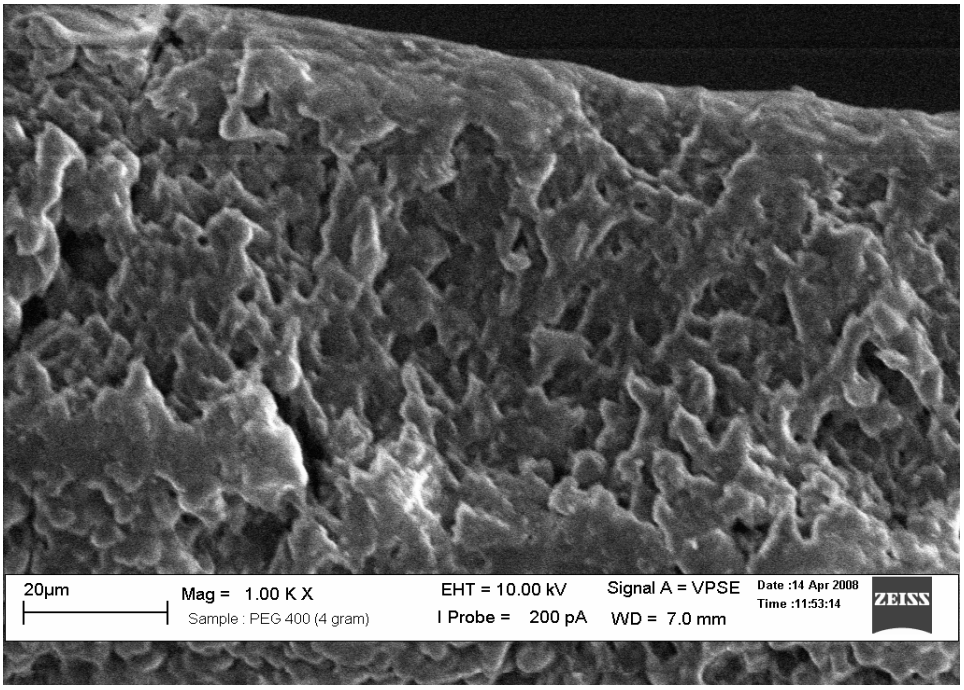


Figure 4.10(a) : SEM photograph of the cross section of Chitosan blend PVA membrane with concentration of PEG 400 in 4 wt% as additive.

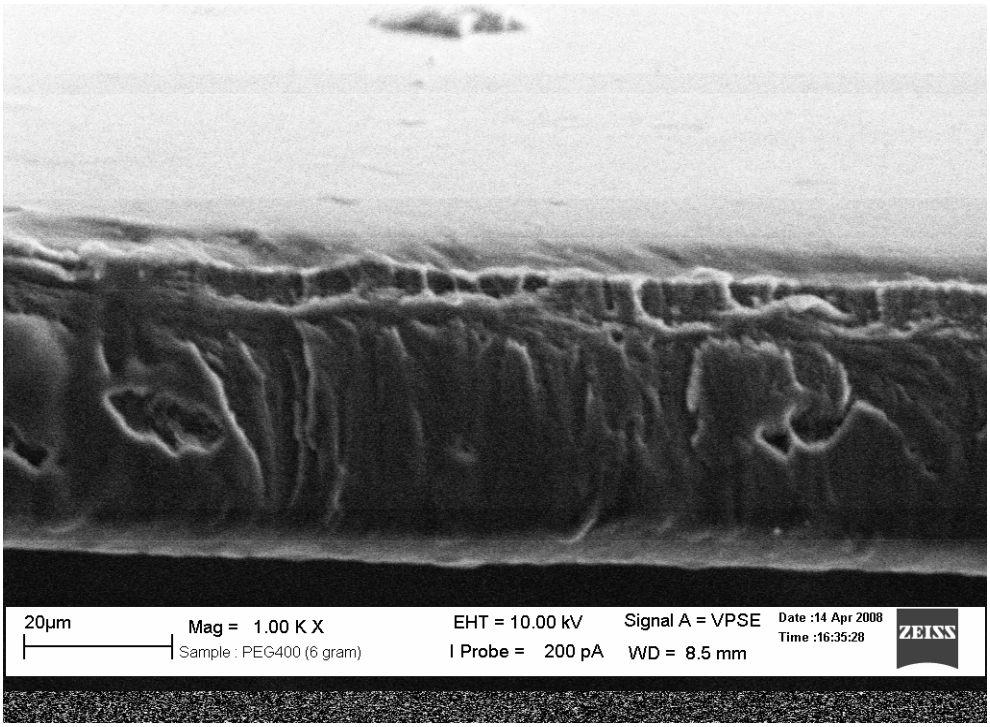


Figure 4.10(b): SEM photograph of the cross section of Chitosan blend PVA membrane with concentration of PEG 400 in 6 wt% as additive.

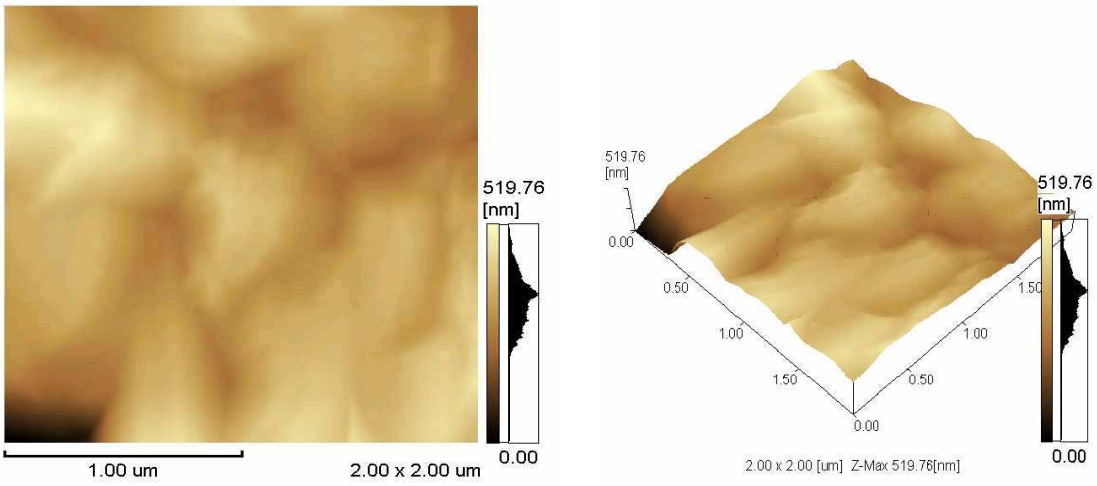
From the figure 4.10(a), the SEM images shows that the membrane is rough and no pores. The membrane porosity is very low but has small pores due to the concentration of low PEG 400 as additive. Small but lots of microvoid can also be seen. This explains why the solutes permeation rate or flux obtained were really low. Pure water permeation is strongly depends on the top surface sub layer of the membranes. This probably the cause for low pure water permeation rate for Chitosan and PVA blend membrane containing 4 wt% of PEG 400. The image also shows that the crosslinking between PVA and Chitosan in not complete. The crosslinking between both PVA and chitosan is expected to have small and clear microvoid with numerous number of pores due to adding chitosan in to the dope solution. From the image above, it looks like a melting membrane with lots of small and difference size of nodules.

From figure 4.10 (b), the SEM images shows that the membrane least rough and no pores. Pores across the membrane cannot be seen clearly even though the

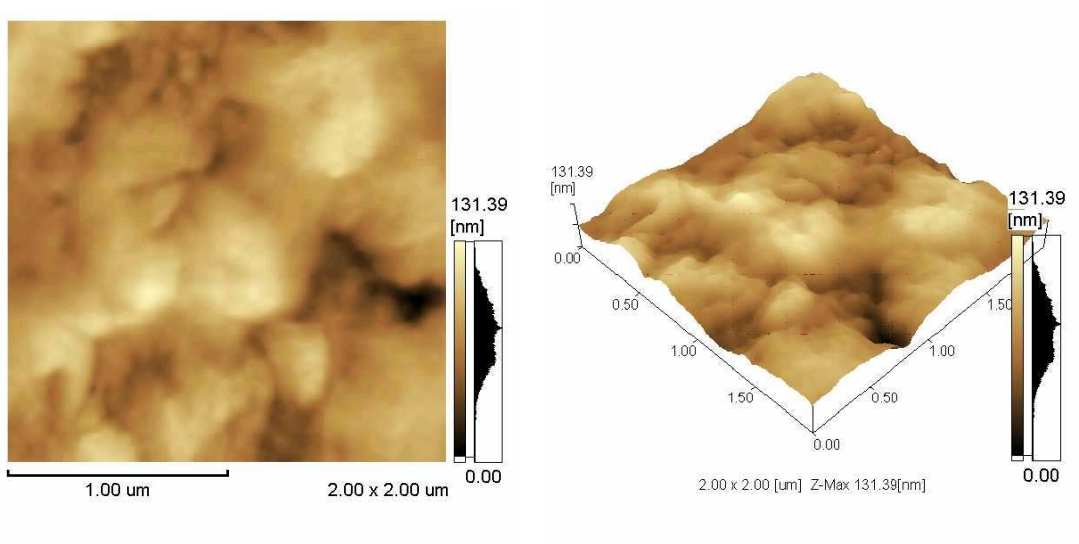
concentration of PEG 400 used in the dope solution is higher. Microviod also cannot be distinguished. The crosslinking between PVA and chitosan might as well ineffective where pores and microvoid became homogeneous. The crosslinking between both PVA and chitosan in 6 wt% of PEG 400 is expected to have small and clear microvoid with numerous number of large pores due to adding chitosan and high concentration of additive in to the dope solution. From the image above, the membrane also looks like a melting membrane but with no small nodules between the surface and the bottom of the membrane

4.5 Atomic Force Microscopy (AFM)

In this study, AFM has been used to characterize different Chitosan blend PVA membranes with different concentration of PEG 400. Images below shows the results obtained by using AFM.



(a)



(b)

Figure 4.11: AFM topography images of Chitosan blend PVA membranes with (a) 4 wt% concentration of PEG 400 and (b) 6 wt% concentration of PEG 400

Topography images at figure 4.11(a) shows the 2D and 3D AFM images of Chitosan and PVA blend membrane with 4 wt% concentartion of PEG 400 at a scan area of 2 µm x 2 µm where the nodules are large merged forming nodules. This results in higher separation and selectivity. This explains the low pure water permeation and solute separation in the membrane. The roughness parameter of Chitosan and PVA blend membrane decreased with addition of 4 wt% concentration of PEG 400 as additive. Low concentration of PEG 400 probably forms a very smooth surface layer and less roughness as seen at the roughness parameter values. The results for roughness parameter with different concentration of PEG 400 can be seen at table 4.9.

Topography images at figure 4.11(b) shows the 2D and 3D images of chitosan blend PVA membrane with 6 wt% concentration of PEG 400 at a scan area of 2 µm x 2 µm where the nodules are less merged and increasing in roughness area. The increasing of roughness as the concentration of additive also increased exhibits high flux but poor

selectivity. This explains the high pure water permeation and solutes separation in the membrane. Thus, the concentration of additive in the dope solution affects the morphology of the skin surface of chitosan blend PVA membrane with different concentration of PEG 400.

Sample	**Ra(nm)**	**Ry(nm)**	**Rz(nm)**	**Rms(nm)**
PEG 400 (4 wt%)	15.776	132.065	71.898	19.305
PEG 400 (6 wt%)	51.492	514.720	220.664	67.115

Table 4.4: Roughness parameter of AFM topography images of Chitosan blend PVA membrane with different concentration of PEG 400

The results for roughness parameter, Ra, Ry, Rz, and Rms for chitosan blend PVA membranes with differences concentration of PEG 400 as additive are shown at table 4.4. The roughness parameter increased with increment of concentrations of PEG 400 while the roughness parameter for lower concentrations of PEG 400 is decreased. This lower concentration of PEG 400 creates a very smooth surface of the membrane and low roughness parameter values. But as the concentrations of PEG 400 became higher, the roughness parameter also increased. It appears that the higher the concentration of PEG 400, it tend to have less merged and less tightly packed nodules but the surface became rougher. This also indicates that the high surface membrane roughness contributes to high flux and membrane with smooth surface membrane exhibits low flux.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Difference concentrations of PEG 400 in chitosan blend PVA membranes gives difference results. Two concentrations of PEG 400 that was used in membrane experiment were 4 wt% and 6 wt% of concentrations. Blending PEG 400 with Chitosan and PVA gives advantages in the membrane mechanical strength and enhancement in its pores size and hydrophilicity.

Pure water permeation (PWP) resulting from the testing of two difference concentration of PEG 400 in two membranes indicates that the higher the concentration of PEG 400, the higher the pure water permeation of the permeate which is. While membrane containing PEG 400 4 wt% only have low pure water permeation. It shows that the hydrophilicity of the membrane from 6 wt% of PEG 400 is higher then the hydrophilicity from membrane containing 4 wt% of PEG 400. 4 wt% of PEG 400 contains in the chitosan blend PVA membranes have higher percentages of solutes separation due to its small size of pores. Means that the selectivity of this membrane is higher then the selectivity of 6 wt% PEG 400 contains in chitosan blend PVA membranes. Another thing that affects the selectivity of the membrane is the crosslinking method between formaldehyde and the membrane. Crosslinking cause the cristallinity of PVA to decrease but it also decreased the effect of absorption rate for the membranes. So, the solute cannot bond themselves at the surface of the membrane.

Two differences morphology of membrane can be seen by using Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM). In SEM, the 4 wt% of PEG 400 gives homogenous, and symmetric type of membrane where the pores is small and microvoids cannot be distinguish and recognizes. This can be seen from the cross-section of the membrane. In 6 wt% of PEG 400, cross-section of the membrane reveals that it's also a homogenous membrane with microvoid and the pores cannot be seen clearly. The purpose of AFM is to identify the surface morphology of the membranes. Here, 4 wt% of PEG 400 in membrane gives the membrane a merged and large nodules with smooth surface. The surface membrane of 6 wt% of PEG 400 contains in the membrane shows that the surface became rougher with lots of small nodules. This explains the effect of surface membrane permeation flux. The rougher the surface of the membrane, the higher the permeation flux tends to be.

5.2 Recommendations

The test conducts for the membranes was quite successful because its indicates that the lower the concentrations of the PEG 400, the higher the selectivity and percentages of solute separation can be. However, to study the characterization of the membrane better, these are few steps that could be taken to improve the result and data analyzing process.

a. To ensure that the results are more interesting, the membrane with PEG 400 in polyvinyl alcohol chitosan blend PVA membranes should be fabricated.
b. For the SEM analysis, the cross sectional area 'cutting' should not use knife or a pair scissors, Instead, the membrane should be soaked into liquid nitrogen for a day until its fully hard and after that break it into small pieces.
c. For the membrane performances analysis, the initial concentration of BSA and lysozyme should be lower. This can prevent the effect of fouling and can contribute to good solute separation.

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APPENDIX A

TABLE OF CALCULATED VALUE

Table 4.1: water permeation flux obtained using Chitosan blend PVA membrane with different concentration of PEG 400 as additive.

Time, s	PEG 400 (4wt%), mL	PEG 400 (6wt%), mL
10	1.7	8
20	2	13
30	2.5	19
40	3	24
50	3	27
60	4	30

Table 4.2: pure water permeation (PWP) obtained using Chitosan blend PVA membrane with different concentration of PEG 400 as additive.

Time, s	PEG 400 (4wt%), mL	PWP, $\frac{mL}{cm^2 s}$	PEG 400 (6wt%), mL	PWP, $\frac{mL}{cm^2 s}$
10	1.7	0.005284	8	0.024868
20	2	0.003108	13	0.020205
30	2.5	0.00259	19	0.019687
40	3	0.002331	24	0.018651
50	3	0.001865	27	0.016786
60	4	0.002072	30	0.015542

Take diameter, D = 6.4 cm

Area, A = $\frac{\pi D^2}{4}$

= 32.1699 cm^2

Table 4.5: Volumes of permeate fluxe after separation process across the Chitosan blend PVA membrane with different concentration of PEG 400.

Time, s	PEG 400 (4 wt%)		PEG 400 (6 wt%)	
	BSA, mL	Lysozyme, mL	BSA, mL	Lysozyme, mL
60	4	4	16	10

Table 4.6: Optical density of solutes after separation processes across the Chitosan blend PVA membrane with different concentration of PEG 400.

parameter	PEG 400 (4 wt%)		PEG 400 (6 wt%)	
	BSA	Lysozyme	BSA	Lysozyme
Optical density,	1.042	0.780	1.1314	0.884

Table 4.7 : Percentage of solute separation obtain after separation processes using Chitosan blend PVA membrane with different concentration of PGE 400

sample	PEG 400 (4 wt%), C_p (µg/mL)	R %	Peg 400 (6 wt%) C_p (µg/mL)	R %
BSA	960	52	1020	49
lysozyme	980	51	1350	32.5

Solute concentration at feed stream, C_f = 2000 µg/mL

Table 4.8: Flux rate obtained after separation processes using Chitosan blend PVA membrane with different concentration of PGE 400

sample	PEG 400 (4 wt%), mL	J, $\frac{mL}{cm^2 s}$	Peg 400 (6 wt%) mL	J, $\frac{mL}{cm^2 s}$
BSA	4	2.072×10^3	16	8.289×10^3
lysozyme	4	2.072×10^3	10	5.181×10^3

Take diameter, D = 6.4 cm

Area, A = $\frac{\pi D^2}{4}$

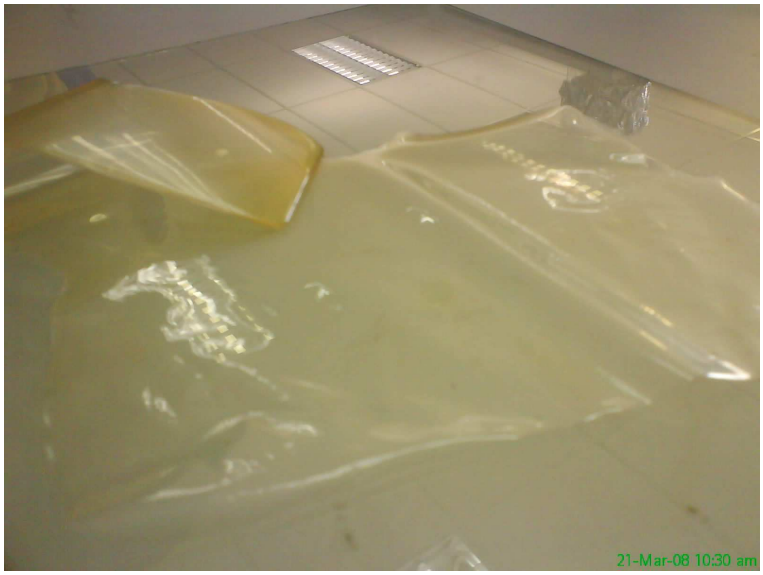
= 32.1699 cm^2

t = 60 s

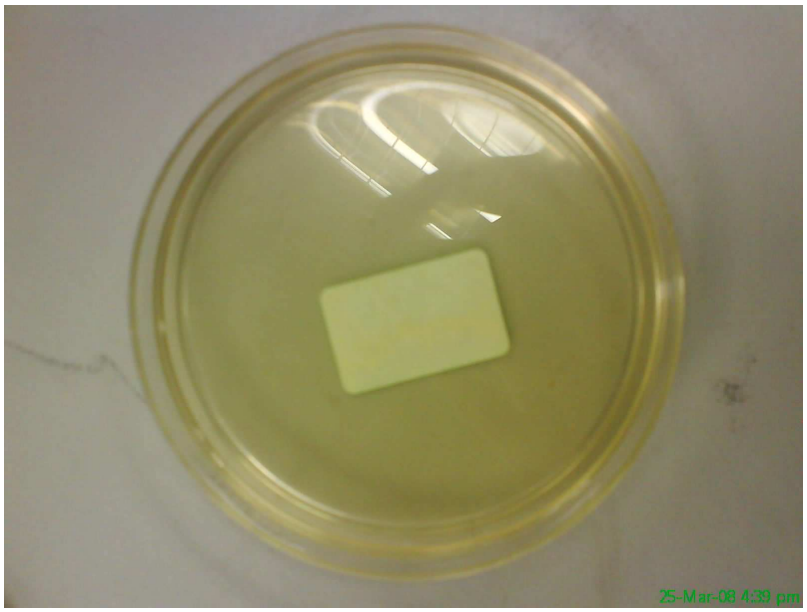
APPENDIX B

Membrane in coagulation bath

Dope solutions



Membrane that already being peeled off



Casting membrane in a petri dish

APPENDIX C : GHANTT CHART FOR PSM I

TASK	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14	W15	W16
FIRST MEETING WITH THE SUPERVISOR																
DEFINING PROJECT INFORMATION tittles, objectives, scopes, problem statements, etc																
LITERATURE REVIEWS																
PREPARE FIRST DRAFT FOR LITERATURE REVIEWS AND SUBMIT																
METHODOLOGY 1. Define meterial apparatus 2. Define method used for the research 3. Studies on properties of the material																
PREPARE FULL DRAFT OF REPORT AND SUBMIT																
PREPARATION OF PRESENTATION preperation of slide																
PRESENTATION OF PSM 1																
CORRECTION AND PREPARATION OF FINAL DRAFT																
SUBMISSION OF FINAL DRAFT REPORT																
CORRECTION AND PREPARATION FOR PSM II																
PREPARATION FOR PSM II																

GANTT CHART FOR PSM II

TASK	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14
PREPARATION OF SAMPLE														
START ON EXPERIMENT AND TESTING 1. Dope solution 2. casting method 3. scanning electron microscopy (SEM) 4. atomic force microscopy (AFM)														
PERFORM ANALYSIS AND COLLECTED DATA 1. analyze result 2. graph plotting 3. discussion 4. final conclusion 5. verify result, discussion and conclusion with supervisor														
COMPLETING FINAL REPORT														
PRESENTATION ON PSM II														
SUBMIT FULL REPORT														