SYNTHESIS AND CHARACTERIZATION OF SAGO/POLYVINYL ALCOHOL BLEND PERVAPORATION MEMBRANE FOR ETHYL ACETATE RECOVERY

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TO MY FATHER

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ABSTRACT

Pervaporation is a membrane separation technology with high selectivity, efficiency and energy saving benefits that make it the method of choice for separation of mixtures. The application for pervaporation includes removal of dilute organic compounds from aqueous solution and dehydration of organics such as dehydration of ethyl acetate-water mixture. The best successful application that has been used for pervaporation is dehydration of organic liquid from water using hydrophilic polymer membrane. In this work, material used for membrane separation was sago and polyvinyl alcohol. However, during the separation process, excessive affinity of water towards hydrophilic polymer membrane led to an increase in the swelling of the membrane. To control the degree of swelling the membranes were cross-linked to improve the intrinsic properties of hydrophilic polymer membranes. Sago starch was used as based polymer to prepare membranes with polyvinyl alcohol (PVA) with various morphologies such as homogenous, composite and blended ration of sago and PVA. Sago/PVA membranes were cross-linked using three different approaches: firstly, using glutaraldehyde, secondly using thermal treatment (80 °C) and thirdly by using both glutaraldehyde and thermal treatment. The effects of various cross-linking methods on the intrinsic properties of hydrophilic polymer membrane were investigated. Before applying the cross-linking to sago/PVA membranes for separation of ethyl acetate-water mixture, a physicochemical characterization was carried out using Fourier transform infrared spectroscopy, differential scanning calorimeter (DSC), thermogravimetric analysis (TGA), atomic force microscopy and swelling experiments. The investigation on the effect of cross-linking on the sago/PVA membranes showed an increase in surface hydrophobicity from contact angle measurements. DSC measurements showed an increase in melting temperature of the polymer membranes after cross-linking. In addition, TGA showed an increase in the stability of the polymer membranes after cross-linking. The effects of operating condition such as feed temperature and feed concentration on the permeation flux and separation factor were also investigated. For the pervaporation of ethyl acetate-water mixture, a decrease in flux and an increase in the separation factor were observed with chemical and combination of chemical and thermal cross-linking. Finally, central composite designs (CCD) of response surface methodology was applied to analyse pervaporation performance of thermal cross-linked membrane. Regression models were developed for permeation flux and separation factor as a function of feed temperature, feed concentration and permeate pressure.

ABSTRAK

Penelapsejatan adalah teknologi pemisahan membran dengan pemilihan yang tinggi, kecekapan dan tenaga manfaat penjimatan tenaga yang menjadikannya kaedah pilihan untuk pemisahan campuran. Aplikasi penelapsejatan termasuk penyingkiran sebatian organik cair daripada larutan akueus dan dehidrasi bahan organik seperti dehidrasi campuran etil asetat-air. Aplikasi terbaik yang digunakan untuk penelapsejatan adalah dehidrasi cecair organik dehidrasi daripada air menggunakan membran polimer hidrofilik. Dalam kajian ini, bahan yang digunakan sebagai pemisahan membran adalah sagu dan polivinil alkohol. Walau bagaimanapun, semasa proses pemisahan, afiniti lebihan air ke arah membran polimer hidrofilik membawa kepada peningkatan dalam pembengkakan membran. Untuk mengawal tahap pembengkakan, membran dirangkai silang untuk meningkatkan sifat-sifat intrinsik membran polimer hidrofilik. Kanji sagu telah digunakan sebagai polimer asas bersama polivinil alkohol untuk menyediakan membran dengan pelbagai morfologi seperti homogen, komposit dan campuran nisbah sagu dan PVA. Membran sagu/PVA dirangkaisilang dengan menggunakan tiga pendekatan yang berbeza: pertama, menggunakan glutaraldehid, kedua menggunakan rawatan haba (80 °C) dan ketiga dengan menggunakan kedua-dua glutaraldehid dan rawatan haba. Kesan pelbagai kaedah silang kepada sifat-sifat intrinsik membran polimer hidrofilik telah disiasat. Sebelum aplikasi rangkai silang membran sagu/ PVA untuk pemisahan campuran etil asetat-air, pencirian fizikokimia telah dijalankan menggunakan spektroskopi inframerah transformasi Fourier, kalorimeter pengimbasan pembezaan (DSC), analisis termo-gravimetrik (TGA), mikroskopi daya atom dan eksperimen pembengkakan. Penyiasatan ke atas kesan rangkai silang membran sagu/ PVA menunjukkan peningkatan dalam permukaan hidrofobisiti daripada ukuran sudut bersentuhan. Pengukuran DSC menunjukkan peningkatan dalam suhu lebur membran polimer selepas rangkai silang. Di samping itu, TGA menunjukkan peningkatan dalam kestabilan membran polimer selepas dirangkai silang. Kesan daripada keadaan operasi seperti suhu makanan dan kepekatan suapan kepada faktor fluks penyerapan dan pemisahan juga telah disiasat. Untuk penelapsejatan campuran etil asetat air, penurunan fluks dan peningkatan dalam faktor pemisahan telah diperhatikan dengan bahan kimia dan gabungan kimia dan haba rangkai silang. Akhir sekali, kaedah gerak balas permukaan yang telah melalui reka bentuk komposit pusat (CCD) digunakan untuk menganalisis prestasi penelapsejatan membran yang dirangkai silang dengan haba. Model-model regresi telah dibangunkan untuk fluks penyerapan dan faktor pemisahan sebagai fungsi suhu suapan, kepekatan suapan dan resapan tekanan.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

DMAc	-	N,N-dimetilacetamide
EA	-	Ethyl acetate
ME	-	Microfiltration
PS	-	Polysulfone
PTFE	-	Poly(tetrafluoroethylene)
PTMS	-	poly[1- (trimethylsilyl)- 1-propyne]
PV	-	Pervaporation
PVA	-	Polyvinyl alcohol
PVDF	-	Polyvinylidene difluoride
RO	-	Reverse osmosis
S	-	Sago
UF	-	Ultrafiltration

LIST OF SYMBOLS

α	-	Membrane selectivity
c_i	-	Concentration of component which has lower volatility
c_j	-	Concentration of component which has higher volatility
X_i, X_j	-	feed mole/ weight fraction of species i and j
y_i, y_j	-	permeate mole/ weight fraction of species i and
A	-	Effective membrane area
J	-	Permeation flux
T	-	Absolute temperature
W	-	Weight of permeate
Δt	-	Permeation time
θ	-	advancing contact angle

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Research Background

Pervaporation is a membrane separation process used to separate mixture of dissolved solvents. In recent years there has been increased interest in the use of pervaporation membrane separation techniques for the selective separation of organic liquid mixtures, because of its high separation efficiency and flux rates coupled with potential savings in energy costs and environmentally friendly (Zhu *et al.*, 2005). Two applications of pervaporation have been commercialized to date. The first application is the separation of the dilute dissolved organics such as trichloroethylene and phenol from the wastewater stream. The second and most important application is water removal from aqueous alcohol solutions such as removal water from ethyl acetate (Shao and Huang, 2007). Conversion of biomass into an energy source by fermentation processes to yield chemicals and fuels like bioethanol, to be used as fossil energy resources has been receiving increasing attention in recent years. Ethyl acetate is an organic compound with formula produced from liquid phase esterification of ethanol and acetic acid (Yuan *et al.*, 2011). Ethyl acetate is extensively used in many chemical industry processes like solvent of essence, pharmacy, printing ink and paint (Parvez *et al.*, 2012).

Sago starch can be developed to be a membrane for separation of ethyl acetate from aqueous solution. Sago starch is an important biopolymer and has been used in various applications such as in food, textile and paper. In recent years, production by fermentation of biomass has greatly increased. Sago is present in South East Asia and it could be produced from sago palm, and it is known as rumbia. Sago palm is an important resource especially to the people in rural areas because it has various uses especially in the production of starch either as sago flour or sago pearl (Rishabha 2010). In recent times, interest in the production of sago palm starch has improved extensively. The sago palm is felled, the trunk is split lengthwise and the pith is separated, the pith is crushed to release the starch, washed and strained to remove the starch from the fibrous residue (Abdorreza *et al.,* 2012).

The sago palm is a crop par excellent for sustainable agriculture. It is interesting to note that sago palms are economical acceptable, environmentally friendly, and promotes a socially stable agroforestry system. It is an extremely hardy plant, thriving in swampy, acidic peat soils, submerged and saline soils where few other crops survive, growing more slowly in peat soil than in mineral soil. The palm is immune to floods drought, fire, and strong winds. The large fibrous root system traps silt loads and removes pollutants. Starch is found to accumulate in the trunk of the sago palm until the flowering stage with maximum starch content occurring just before the onset of the palm flowers (Singhal *et al.,* 2007).

Application of sago starch (i.e., thickeners, sizing and coating papers, sizing textile, adhesive formulations, fluid loss additive in drilling mud and other applications) always involves gelatinization of starch that breaks the inter-chains hydrogen bonding such that the rheological properties of starch paste can be utilized effectively. The non-pith parts of the sago palm trunk can be utilized in a variety of ways: as an excellent building material for local and urban houses, sheds, or other buildings; as a resource for cornposting (biofertiliser); as a resource for gasification and energy production; and as an animal feed (Singhal *et al.,* 2007).

The choice of a particular membrane material is dictated by the kind of application such as dehydration of alcohol/water and filtration of waste products in pharmaceutical industries, and the operating condition like temperature, concentration and thickness. In pervaporation dehydration of alcohol/water, the highly hydrophilic polymer such as polyvinyl alcohol is preferred due to its affinity for water. Hydroxy terminated polybutadiene (HTPB-PU) membrane has been used for the recovery of ethyl acetate from water, but the separation factor decreased significantly from 575 to 320 with increase in the operating temperature from 25 to 65 oC; this is due to the weak resistance to the high temperature of physical cross-linking (Bai et al., 2008).

Membrane technology is considered as an efficient and economic separation process in the chemical industry. We can divide the process into three stages according to the mechanism of the separation; the first stage is the sieving mechanism, ultrafiltration, and microfiltration, the second stage is the electrochemical mechanism and electro-dialysis, and the third stage is the solubility mechanism, and pervaporation (Baker, 2004b).

Recently, the extractive distillation and azeotropic distillation have been completely investigated, and was used for the separation and concentration of ethyl acetate. Yet, the two processes are still facing hardship from high operating costs and low concentration of the products because entrainers are required, while pervaporation has a good advantage because it has a low cost, easy operation and no entrainers are required (Zhang *et al.*, 2009b).

Currently, pervaporation, characterized by high separation efficiency, is one of the best alternative processes for the separation of volatile organic materials (such as ethyl acetate) (Konakom *et al.*, 2011) from dilute aqueous solutions. A mixture containing solution of ethyl acetate-water can be made to diffuse from the inside to the outside of non-porous membranes by using a vacuum to the outside of the membrane. The driving force of the pressure differential combined with a membrane selective for ethanol makes ethanol concentration possible (Hasanoğlu *et al.*, 2005).

In pervaporation, a variety of membrane materials have been developed to cater for mainly two types of industrial liquid separations: organic-water and organic-organics separations. For water selective membranes, the most important factor responsible for the separation is the specific interaction between water and the polymer.

Pervaporation has a high potential for separations where the more conventional techniques, such as distillation, are not possible to be realized or too expensive. The pervaporation, for separation of several components from a liquid mixture cannot be only determined by changes in their vapor pressure but can be determined by their permeation rate through the membrane. The actual driving force for the permeation of the different components through the membrane is the difference between the two phases separated by the membrane. Concentration polarization in pervaporation is usually controlled by decreasing the laminar boundary layer's thickness through hydrodynamic measures. This situation can lead to problems if the flux is high and thin film composite membranes are used for the separation process. The laminar boundary layer at the permeate side is always as thick as the porous substructure and severe concentration polarization as well as capillary condensation may occur on the permeate side of the membrane (Nagy, 2012). Table 1.1 shows the list of the most important membrane process, the major field of application and the driving force for the preferentially permeating component.

Table 1.1 Membrane processes (Purchas and Sutherland, 2001)

Membrane process	Separation potential for	Driving force realized	Preferably permeating component
Reverse osmosis	Aqueous low molecular mass solutions, aqueous organic solutions	Pressure difference (≤ 100 bar)	Solvent
Ultrafiltration	Macromolecular solutions, emulsions	Pressure difference (≤ 10 bar)	Solvent
Microfiltration (Cross-flow)	Suspensions, emulsions	Pressure difference (≤ 5 bar)	Continuous phase
Gas permeation	Gas mixtures, water vapour-gas mixtures	Pressure difference (≤ 80 bar)	Preferably permeating component
Pervaporation	Organic mixtures, aqueous-organic mixtures	Permeate side: Ratio of partial pressure to saturation pressure	Preferably permeating component
Liquid membrane technique	Aqueous low molecular mass solutions, Aqueous-organic solutions	Concentration difference	Solute (ions)
Osmosis	Aqueous-organic solutions	Concentration difference	Solvent
Dialysis	Aqueous-organic solutions	Concentration difference	Solute (ions)
Electrodialysis	Aqueous-organic solutions	Concentration difference	Solute (ions)

Pervaporation is a membrane separation process used to separate mixtures of dissolved solvents. In recent years there has been an increased interest in the use of pervaporation membrane separation techniques for the selective separation of organic liquid mixtures; this is due to its high separation efficiency and flux rates coupled with potential savings in energy costs. In pervaporation, volatile organic components are removed from a liquid feed mixture through a semipermeable membrane into a gas phase.

In pervaporation the chemical potential gradient is usually induced by either applying a vacuum on the permeate side of a membrane or by using a sweep gas to remove the permeating component and by applying a temperature difference between the liquid feed mixture and permeate gas phase. Most of the pervaporation membranes are composites formed by solution-coating of the selective layer onto a micro-porous support (Nagy, 2012).

1.2 Problem Statement

Sago and polyvinyl alcohol are highly hydrophilic material. Sago based membrane should be very selective to water; potentially effective for separation dehydration of aqueous solution. In general, the hydrophilic containing polymers have high solubility parameters and show relatively large water solubility. However, the introduction of hydrophilic groups sometimes swells the membranes significantly under aqueous mixture due to its plasticization action that results in poor selectivity. Malaysia is one of the largest producers of sago starch which covers around 7% of Sarawak total area. The production capacity of the sago palm varies from 10-25 tons/ha in dry starch. With the growing concern about the renewable energy especially the conversion of biomass to the biofuel. Potential industrial applications include the recovery of ethanol from fermentation process and the esterification of ethanol and acetic acid to produce the ethyl acetate which is attracting increasing attention due to its low toxicity. Ethyl acetate is an important solvent for antibiotics, paint, printing ink; solvent of essence and it's also use in the manufacture of various drugs. Removal of ethyl acetate from ethanol and water mixture or from isooctane mixture is difficult because of the proximity of boiling point. Currently the industrial methods for the recovery of ethyl acetate from water depends on the extractive distillation, and this process contained several practical problems, such as technology complexity and high energy consumption, thus an eco-friendly and concise separation process is being demanded. Sago starch is a highly hydrophilic material and an important biopolymer, it has been widely used in various industrial applications. Highly hydrophilic, sago based membranes can be specially effective for the recovery of azeotropic mixtures such as ethyl acetate - water solution. The separation dehydration of ethyl acetate -water mixtures is an example for such application where sago based membranes is expected to be effective to remove the water content and purify the ethyl acetate. The efficiency of the pervaporation process depends mainly on the intrinsic properties of the polymers used to prepare the membrane.

Since the sago is highly hydrophilic polymer and polyvinyl alcohol too, there is need for the decrease of the degree of swelling; thus, cross-linking will be important. Cross-linking were affects the physicochemical properties of a membrane, the diffusion and sorption process will be affected by the membrane surface. In order to achieve the effects and the improvement of membrane performance on dehydration of ethyl acetate/water, it will be interesting to study these properties such as (SEM, AFM and FTIR).

1.3 Objectives

The main objectives of this work are to develop sago membrane for dehydration of ethyl acetate/water and to study the effect of some operating parameters, namely volumetric flow rate, feed temperature, permeate-side pressure, thickness and degree of swilling on flux and ethyl acetate selectivity during the pervaporation separation process using sago membrane. Specific objectives of this study for achieving the above purpose are to:

i. Develop sago based membranes for the dehydration of ethyl acetate -water mixtures at different operating conditions.
ii. Investigate the effect of crosslinking on the physiochemical properties of sago/PVA blend membrane before and after crosslinking.
iii. Study the effect of cross-linking on the composite membranes consisting of sago/PVA- poly sulfone for pervaporation of ethyl acetate-water mixture.

iv. Study the effect of sago blended ration on the separation of ethyl acetate-water mixtures.
v. Study and optimize the effect of operating conditions on pervaporation performance using a statistical design (RSM) of experimental approach.

1.4 Scope of the Thesis

This thesis is including the studies of various hydrophilic sago/PVA membranes, their characterization techniques and use for pervaporation of ethyl acetate- water mixture. Characterizing of the membrane is necessary in order to determine the physical and chemical properties of the thin film. The sago-based membranes were characterized using Scanning Electron Microscopy and Fourier Transform Infrared Spectroscopy (FTIR). The driving force for transport across the membrane is the chemical potential gradient and the physical structure of the membrane determines the flux. The difference between the individual penetrant components determines the membrane selectivity. Cross-linking modification was used for improvement of the membrane performance. The research consists of two parts. The first part is including the development, characterization and separation of ethyl acetate water mixture using various membrane of sago/PVA. The second part will focus on the optimization of pervaporation process variables. The scope of each chapter is listed as follows:

Chapter 1: presents an overview of the thesis, including a brief introduction to the pervaporation membrane technology and pervaporation process and its history and overview to membrane materials. The objectives and scope of the thesis are also given in this chapter.

Chapter 2: provided the overview of sago starch and some aspects of pervaporation characteristics, different type of membrane materials, membrane modification techniques, factors affecting pervaporation and pervaporation of ethyl acetate-water mixture.

Chapter 3: presented material and method of the thesis that used for preparing of the homogenous membranes, composite membrane and blended membranes.

Chapter 4: discusses the effects of various cross-linking on the physicochemical properties of sago/PVA membrane and on the pervaporation of ethyl acetate-water mixture. The liquid sorption and pervaporation were investigated for the ethyl acetate-water mixture using sago/PVA membrane.

Chapter 5: study the effect of cross-linked sago/PVA - polysulfone composite membrane for pervaporation of ethyl acetate-water mixture. The results were compared with homogenous sago/PVA membrane using the pervaporation separation index (PSI).

Chapter 6: of this study focuses on the development of sago/PVA membrane and the effect of sago blended ration on the separation performance. The effect of feed temperature, concentration and sago blended ratio on the separation factor and permeation flux was investigated.

Chapter 7 presents a study on the optimization of the operating conditions on the separation factor and the permeation flux by Central Composite Rotatable Design (CCRD) for pervaporation of ethyl acetate-water mixture; in order to find the optimum operating conditions of feed temperature feed concentration and permeate pressure.

The final chapter is the conclusions of the studies conducted in the thesis and recommendations for future work.

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