PERVAPORATION SEPARATION OF METHYL *TERT*-BUTYL ETHER /METHANOL MIXTURES USING POLYION COMPLEX CHITOSAN MEMBRANES

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To my beloved parents and family members

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ABSTRACT

The main objective of this research was to develop polyion complex composite (PIC) membranes consisting of chitosan/sodium alginate (SA) for the pervaporation separation of methyl *tert*-butyl ether (MTBE)/methanol mixtures. The PIC membranes were fabricated by casting homogeneous chitosan/SA solution onto polysulfone (PS) which was used as a porous support. The chitosan and sodium alginate solutions were prepared separately by using dilute acetic acid as the common solvent. The homogeneous chitosan/SA solution was prepared by mixing both the chitosan and SA solutions for 24 hours. The morphological structure of the membranes was studied using Scanning Electron Microscopy (SEM). The morphology study showed that the PIC membranes consisted of two layers. The top layer was dense which indicated that the two polymers were very homogenously intermixed and in the state of the polyion complex. The membranes were used for the separation of MTBE/methanol mixture via pervaporation. The separation performance of the chitosan/SA membrane was compared with that of composite chitosan membrane. The sodium alginate content incorporated in the PIC membrane enhanced the membrane performance in the pervaporation separation of MTBE/methanol mixtures. The overall pervaporation performances for the membranes were determined in term of pervaporation separation index (PSI). The PIC membranes showed higher PSI than that of the composite membrane especially at high feed MTBE concentrations. The optimum concentration of SA in membranes was found to be 1.5 wt. %. At this concentration of SA, the degree of swelling was 1.02 % with 42.5 wt. % MTBE sorbed in membrane while PSI increased from 946.9 g/m^2.h (composite chitosan membrane) to 3211.8 g/m^2.h. It was also found that a rise in operating temperature increased the permeation flux and separation factor.

ABSTRAK

Objektif utama kajian ini adalah untuk menghasilkan membran komposit kompleks poli ion (PIC) yang terdiri daripada kitosan/natrium alginat untuk proses pemisahan pervaporasi bagi campuran metil *tert*-butil eter (MTBE)/ metanol. Membran PIC dihasilkan dengan membuat lapisan larutan kitosan/natrium alginat ke atas membran polisulfon sebagai penyokong poros. Kitosan dan natrium alginat dilarutkan ke dalam larutan akues asid asetik secara berasingan. Larutan homogen kitosan/ natrium alginat dihasilkan daripada campuran larutan kitosan dan natrium alginat selama 24 jam. Morfologi membran tersebut dikaji dengan menggunakan Mikroskop Pengamatan Elektron (SEM). Morfologi bagi membran PIC menunjukan terdapat dua lapisan di mana lapisan atas adalah padat membuktikan campuran homogen dua polimer membentuk membran poli ion. Membran yang dihasilkan digunakan untuk pemisahan pervaporasi campuran MTBE/metanol. Prestasi pemisahan membran kitosan/natrium alginat dibandingkan dengan membran komposit kitosan. Kehadiran natrium alginat dalam membran PIC meningkatkan prestasi membran bagi pemisahan MTBE/metanol. Prestasi keseluruhan pervaporasi membran yang dihasilkan ditentukan menggunakan indeks pemisahan pervaporasi (PSI). Membran PIC menunjukkan PSI yang lebih tinggi berbanding dengan membran komposit kitosan terutama dalam campuran yang mempunyai kepkatan MTBE yang tinggi. Kepekatan optimum bagi SA adalah 1.5 wt.%. Pada kepekatan natrium alginat ini, darjah pengembangan adalah 1.02 % di mana 42.5 wt. % MTBE diserap oleh membran dan PSI meningkat dari 946.9 g/m^2.h (membran komposit kitosan) kepada 3211.8 g/m^2.h. Kajian ini juga menunjukkan peningkatan suhu operasi meningkatkan kadar penelapan dan faktor pemisahan.

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LIST OF SYMBOLS

J_t	-	Total flux
A	-	Membrane area
W	-	Weight of permeate
Δt	-	Permeation time
α	-	Separation factor
x_j	-	Concentration of the component i in the feed
x_j	-	Concentration of the component j in the feed
y_i	-	Concentration of the component i in the permeate side
yj	-	Concentration of the component j in the permeate side
β	-	Enrichment factor
J	-	Permeation flux
E_p	-	Activation energy
T	-	Absolute temperature
R	-	Gas constant
DS	-	Degree of swelling
w_f	-	Final (swollen) weight of the membrane
w_j	-	Initial (dry) weight of the membrane
C_{MTBE}	-	The weight fraction of permeate MTBE component in the membranes at equilibrium sorption
$C_{methanol}$	-	The weight fraction of permeate methanol component in the membranes at equilibrium sorption
X_{MTBE}	-	The weight fraction of feed MTBE component in the membranes at equilibrium sorption

$X_{methanol}$	-	The weight fraction of feed methanol component in the membranes at equilibrium sorption
$\alpha_{\text{sorption } i/j}$	-	Sorption separation factor component i and j
PSI	-	Pervaporation separation index

LIST OF ABBREVIATIONS

MTBE	-	Methyl *tert*-butyl ether
ETBE	-	Ethyl-*tert* butyl ether
DMC	-	Dimethyl carbonate
TAME	-	*tert*-amyl methyl ether
PAN	-	Polyacrylonitrile
PIC	-	Polyion complex composite
PS	-	Polysulfone
SA	-	Sodium alginate
DMAc	-	N,N-dimetilacetamide
EPDM	-	Ethylene propylene diene monomer
PVDF	-	Polyvinylidene difluoride
PTFE	-	Poly(tetrafluoroethylene)
PVA	-	Polyvinyl alcohol
CS	-	Chitosan
NR	-	Natural rubber
ENR	-	Epoxidized natural rubber
SBR	-	Styrene–butadiene rubber
PPOP	-	Poly[bis(phenoxy)phosphazene]
PU	-	Polyurethane
GFT	-	Gesellschaft fur Trenntechnik

LIST OF GLOSSARIES

Membrane	-	Membran
Composite	-	Komposit
Complex	-	Kompleks
Polyion	-	Poli ion
Chitosan	-	Kitosan
Sodium alginate	-	Natrium alginat
Polysulfone	-	Polisulfon
Methanol	-	Metanol
Methyl *tert*-butyl ether	-	Metil *tert*-butil eter
Pervaporation	-	Pervaporasi
Morphologies	-	Morfologi
Permeation	-	Penelapan
Sorption	-	Erapan
Swelling	-	Pengembangan

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CHAPTER 1

INTRODUCTION

1.1 Background

Separation processes are very important in a variety of industries for the removal of contaminants from raw materials, recovery and purification. Distillation and liquid-liquid extraction are two of the most common and traditional technologies used for separation. However, distillation generally is an energy intensive process, while liquid-liquid extraction suffers from major drawback of enhanced downstream processing due to the presence of an additional solvent. Thus, alternative technologies may be required to meet the desired separation objective. Membrane separation process enjoys numerous industrial applications with the major advantages such as appreciable energy savings, environmentally benign, clean technology with operational ease, produces high quality products, and also has greater flexibility in designing systems. The most important membrane qualities are high selectivity, high permeability, mechanical stability, temperature stability and chemical resistance (Rautenbach and Albrecht, 1989). 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 (Rautenbach and Albrecht, 1989).

Table 1.1: Membrane processes (Purchas, 1996)

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 one of the membrane separation processes in which a multicomponent liquid is passed across a membrane that preferentially permeates one or more of the components (Baker, 2000). The term pervaporation is derived from the words permeation and evaporation (Bowen, 2003), which are the primary mechanisms in this process. The basic pervaporation system has a membrane module, a feed delivery system, and a permeate condensation/recovery system (Peng, 2004). Pervaporation differs from other process in that the membrane constitutes a barrier between a liquid in the liquid phase and permeate in the vapour phase (Huang and Rhim, 1983). A pervaporation membrane is usually a synthetic polymer film, and components of a liquid feed first dissolve in the membrane and then diffuse across a concentration gradient. A vacuum is usually maintained on the downstream side, removing all molecules migrating to this stream (Shao, 2003).

The main advantage of pervaporation is that it uses much less energy than other phase-change separations such as distillation. Pervaporation systems do not have emission problems or require expensive regeneration steps. They can operate continuously without consuming sorbents, can be used to recycle/re-use solvents, and cost less to operate than many other applications (Bowen, 2003; Peng, 2004). Unlike distillation, where separation is based on the boiling point differences of the components, pervaporation does not require such high temperatures and can be run at room temperature. Membrane based pervaporation technique is an economical separation method compared to conventional processes for specific separations involving azeotropic and close boiling mixtures due to its high separation factor and flux rates (Dhanuja et al., 2005). Azeotropic mixtures are an obvious target application for a pervaporation process. Usually the pervaporation process is separated into three steps:

i. Sorption of the permeable component into the separating layer of the membrane.
ii. Diffusive transport of the substance across the membrane
iii. Desorption of the substance at the permeate side of the membrane.

In this process, liquid mixture are feed under pressure to a non-porous membrane, where components pass through the membrane by solution-diffusion and evaporate at the permeate side of the membrane. The requirements can be satisfied by the so-called composite membrane consisting of a highly permeable support layer either polyacrylonitrile (PAN) or polysulfone (PS) coated with a thin and highly selective separation layer (Haack et al., 2001). It current usage is well known in dehydration of the organic solvents and mixtures and the removal of organics from aqueous stream.

Pervaporation is a potential industry method for the separation of liquid mixtures. For this purpose, good membrane material should have high flux, high separation efficiency, and long-term stability to maintain its original permselectivity under operating condition. Since a trade-off between the flux and separation factor exists, much effort has been made to achieve high fluxes and separation factors simultaneously (Huang et al., 1999). There are two typical kinds of pervaporation membranes, one is water-permselective and second is alcohol-permselective membranes (Ge et al., 2000).

Membrane-based pervaporation process has been actively studied for separation of aqueous/organic mixtures and more recently, organic/organic mixtures. In the chemical and petrochemical industries the separation of organic/organic mixtures is the most important process (Schell et al., 1989). The first plant for organic/organic separation by pervaporation was reported by Air Products for the removal of methanol from methyl-*tert*-butyl ether (MTBE) in the production of octane enhancer for fuel bends (Chen et al., 1989).

1.2 Problem Statement

Methyl *tert*-butyl ether (MTBE) has found wide applications as oxygenate for gasoline to meet the clean Act requirements (Kim et al, 2000). To reduce air pollution, it is recommended to use lead-free or low leaded gasoline. Therefore, new effort has been made to substitute the *tert*-ethyl lead by some ethers and alcohol, such as methyl *tert*-butyl ether (MTBE), ethyl *tert*-butyl ether (ETBE) etc., wherein MTBE is widely tested to be a good substitute (Cao et al., 2000). The application of MTBE has been expanding rapidly in recent years. MTBE is primarily produced on an industrial scale by reacting isobutene with excess methanol and it can be expressed as;

$$(CH_3)_2C{=}CH_2 + CH_3OH \text{ -----> } (CH_3)_3\ C\text{--}O\text{--}CH_3 \qquad (1.1)$$

In the process of MTBE synthesis, the product contains residual methanol. The unreacted methanol is subsequently distilled off and recovered. The separation of methanol from MTBE is an organic–organic separation whose economic importance has increased with the industrial production of octane enhancers (Gozzelino and Malucelli, 2004). Separation of MTBE/methanol mixtures is an important unit operation. It is very difficult to separate, because methanol forms minimum-boiling azeotropes with MTBE product at a composition of 14.3 wt. % methanol at 760 mm Hg (Kim et al., 2000). A significant part of the process costs is dedicated to the separation of the reaction mixtures because of the azeotropic nature of the MTBE/methanol mixtures. Removal of methanol is important due to azeotropic nature of the mixtures.

The traditional separation method is washing MTBE with water to remove methanol (Shi et al., 2004). The azeotropic mixture is taken to a botanizer to produce a MTBE bottom product and a binary methanol/C_4 azeotrope overhead (Kim et al., 2000). The stream is then washed with water to remove the excess methanol and the water/methanol mixture is distilled to recover the methanol for recycling. Since distillation is needed to separate methanol from water, energy consumption is very high. Pervaporation process using membrane has been considered as an alternative separation technique to replace the conventional separation.

The mixture of MTBE with methanol is a representative of a polar/non-polar solvent system, and methanol is more hydrophilic than MTBE due to dipole moment of hydroxyl group and it is supposed that the interaction to polar molecule of methanol should be larger than that of MTBE, which results in larger affinity of methanol in polar or hydrophilic polymer materials (Kim et al., 2000).

In the present study, it was demonstrated that chitosan membranes are capable of separating MTBE/methanol mixtures by pervaporation. Chitosan is a partially deacetylated polymer of chitin, which is found in a wide range of natural source like crab, lobster and shrimp shells. Chitosan is widely used in membrane applications based on its high hydrophilicity, good film-forming character, functional groups that can easily modified apart from its good mechanical and excellent chemical-resistant properties (Wang et al., 1996). Recently, many researchers have been directed to chitosan as a pervaporation membrane material (Kittur et al., 2005). Chitosan can be used for homogenous membranes or the skin layer of composite membranes (Ge et al., 2000).

Chitosan has an amino and hydroxyl group that can be used as a chemical reactions and salt formation (Devi et al., 2005). The alginate inside the chitosan which is one of the polysaccharides extracted has been found to be a material that can used to give a great performance as a pervaporation membrane material (Shi et al., 1996). The hydrophilic groups are important in preferential water sorption and diffusion through chitosan membrane. However the larger free volume between the molecular chains of pure chitosan membrane causes the unsatisfactory separation performance in terms of the total permeation flux, separation factor and mechanical strength. The performance of chitosan is stated to be better by blending it with other polymer. Based on the previous research of MTBE/methanol separation, chitosan and sodium alginate are typical polar materials used as membrane materials for the separation of MTBE/methanol mixtures.

Sodium alginate and chitosan are ionic polymer of polysaccharides, which are not only very hydrophilic, but also rigid and bulky from the point of structural view. Blending of the polymers resulted in the spontaneous formation of polyion complex membranes (Kim et al, 2000). The polyion complex formation happens by ionic interaction between counter ion groups. The polyion complex membranes made from anionic and cationic have been reported to have an excellent selectivity and permeability with good stability (Kanti et al., 2004).

However, most of the dense membranes are found selective to water permeation, and only a few are selective to permeation of organic compounds. Thus, asymmetric and composite membrane structures have been introduced into the membrane. Chitosan/sodium alginate blend and polysulfone are good material for preparing composite membranes. Composite membrane can enhance the structural stability of chitosan/sodium alginate blend polyion complex membrane over a wide range of temperature and feed concentrations and it can control the permselectivity of the composite membrane. Kanti et al., 2004 studied the chitosan/sodium alginate blend polyion complex membrane for the dehydration of ethanol. They found that the polyion membrane improved the pervaporation properties. The purpose of this study is to prepare the chitosan/sodium alginate blend polyion complex membrane for pervaporation separation of MTBE/methanol mixtures.

Most of the pervaporation techniques in literature deal only with a small range of feed concentrations. The parameter of pervaporation is studied to predict separation behaviour over a wide range of feed concentration and separation temperature. The sodium alginate is the polar material and selects methanol as permeate. But the high concentration of methanol will affect the selective diffusivity of the membrane. Therefore, the film should be maintained to avoid the hydrophilic groups from swelling the membranes in aqueous mixture. The percentage sodium alginate content affects the permeation flux and separation factor. This study also focuses to determine an optimum concentration of sodium alginate for polyion membranes.

1.3 Objective and Scopes of Work

The objective of this research is to modify via polyion complex sodium alginate/chitosan blend membranes to improve the overall performance of the composite membranes in pervaporation separation of MTBE/methanol. In order to achieve the mentioned objective, the following need to be accomplished;

i. Development of pure chitosan composite based membrane.
ii. Development of composite type chitosan/sodium alginate polyion complex membranes. Different concentrations of sodium alginate (0.5-2.0 wt. %) were used to modify the composite chitosan membrane.
iii. Characterization of membrane morphology using Nikon Microscope and PHILIPS XL-40 Scanning Electron Microscopy (SEM).
iv. Sorption test and desorption test were used to measure the percentage of swelling and the sorbed solution composition.
v. Separation efficiency of modified membranes was determined using pervaporation process for separation of MTBE /methanol mixtures.

These experiments were conducted using different feed compositions of MTBE /methanol mixture (30-70 wt. %) and separation temperatures at 30^{o}C, 35^{o}C, 45^{o}C and 55^{o}C while permeate pressure was maintained at 0.07 bar to determine the optimum separation condition for the developed membranes.

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