ENHANCEMENT OF ANTIFOULING PROPERTIES USING RICE HUSK SILICA PARTICLES IN POLYSULFONE MEMBRANE AND OPTIMIZATION OF ITS OPERATING CONDITION

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ABSTRACT

This study investigates the effects of rice husk silica (RHS) as additive in the polysulfone membrane to enhance antifouling properties in membrane separation process. The flat sheet PSf/RHS membrane was prepared via phase inversion technique. The characterization and performance test were conducted on PSf ultrafiltration membrane prepared from a different additive concentration. The thermal stability of prepared membrane was observed by using thermogravimetric analysis (TGA). The cross section area and particles distribution of additive were carried out by using the scanning electron microscope (SEM) while the surface morphology was investigated via field emission scanning electron microscope (FESEM). The surface roughness and hydrophilicity were also determined by using Atomic force microscopy (AFM) and contact angle measurement respectively. Meanwhile, the performance was evaluated in term of pure water flux (PWF), rejection and antifouling properties. The optimized of normalized flux ($J_f/J_o$) at different parameter filtration condition (pH, ionic strength and transmembrane-pressure) was carried out by using the response surface methodology (RSM). From the analysis of SEM, FESEM and AFM, results showed that the microstructure of the membrane especially at top layer and sub layer obviously changed with the incorporation of RHS. The results also demonstrated that the mean pore size was decreased and hydrophilicity was increased as increased RHS particles in PSf membrane. The performance of the membrane was analyzed by using distilled water for permeation test and humic acid for the rejection test. The results also showed that the hydrophilic PSf/RHS membrane has significantly improved the permeation and rejection performance after the addition of RHS. The results showed that the addition of 4 wt. % RHS give the highest flux at 300.50 L/m².hour (LMH). The highest rejection was found at 3 wt. % of RHS membrane with value 98% for UV$_{254}$ and 96% for TOC. The optimal value of $J_f/J_o$ was found at 0.62 with the parameter condition pH: 6.10, ionic strength: 0.05 mol/L and transmembrane-pressure: 2.67 bars. Optimize of RSM analysis also proved that the error of model is less than 0.05% which indicates that the model is significant.
ABSTRAK

Kajian ini menyiasat kesan silika sekam padi (RHS) sebagai bahan tambah dalam membran polisulfon untuk ciri-ciri anti-kekotoran di dalam proses pemisahan membran. Lembaran rata daripada membran PSf/ RHS telah disediakan melalui teknik fasa balikan. Pencirian dan ujian prestasi telah dijalankan ke atas penapisan ultra membran yang disediakan daripada kandungan RHS yang berbeza. Kestabilan terma membran dikaji dengan menggunakan analisis Termogravimetri (TGA). Kajian ke atas keratan rentas dan taburan zarah pada membran telah dijalankan dengan menggunakan mikroskop imbasan elektron (SEM) manakala morfologi permukaan telah disiasat melalui bidang pelepasan mikroskop imbasan elektron (FESEM). Kekasaran permukaan dan sifat hidrofilik juga ditentukan melalui daya Atom mikroskopi (AFM) dan ukuran sudut sentuh. Sementara itu, prestasi aliran air tulen (PWF), penolakan dan pencirian anti-kekotoran juga dijalankan. Proses mengoptimumkan flus normal \( \left( \frac{J_f}{J_o} \right) \) pada keadaan penapisan yang berbeza parameter (pH, kekuatan ionik dan membran tekanan membran) telah dijalankan dengan menggunakan kaedah gerak balas permukaan. Keputusan yang ditunjukkan oleh SEM, FESEM dan AFM bahawa mikrostruktur membran terutama di lapisan atas dan lapisan sub telah berubah. Keputusan kajian juga menunjukkan bahawa saiz purata liang menurun dan peningkatan sifat hidrofilik meningkat disebabkan peningkatan kuantiti RHS dalam membran PSf. Prestasi membran dianalisa dengan menggunakan air suling untuk ujian penyerapan dan asid humik untuk ujian penolakan. Keputusan juga menunjukkan bahawa membran prestasi penyerapan dan penolakan PSf/RHS hidrofilik telah meningkat selepas penambahan RHS. Hasil kajian menunjukkan bahawa dengan tambahan 4 wt. % daripada RHS memberi flus tertinggi iaitu 300.50 L/m².jam (LMH). Penolakan tertinggi ditemui dengan 3 wt. % daripada RHS memben dengan nilai 98% untuk UV\(_{254}\) dan 96% untuk TOC . Nilai optimum \( \frac{J_f}{J_o} \) ditemui pada nilai 0.62 dengan keadaan parameter pH: 6.1, kekuatan ionik 0.05 mol/L dan tekanan membran: 2.67 bar. Analisa optimum dengan menggunakan RSM juga membuktikan bahawa ralat model adalah kurang daripada 0.05% dan menunjukkan bahawa model adalah boleh diterima pakai.
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<tr>
<td>AFM</td>
<td>Atomic Force Microscopy</td>
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<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>A</td>
<td>Membrane Area (m²)</td>
</tr>
<tr>
<td>Cₚ</td>
<td>Solute Concentration in permeate stream</td>
</tr>
<tr>
<td>C₇</td>
<td>Solute Concentration in Feed</td>
</tr>
<tr>
<td>CA</td>
<td>Cellulose Acetate</td>
</tr>
<tr>
<td>FTIR</td>
<td>Fourier transform-infrared</td>
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<tr>
<td>FESEM</td>
<td>Field Emission Scanning electron microscopy</td>
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<td>HA</td>
<td>Humic Acid</td>
</tr>
<tr>
<td>Jₘₕ</td>
<td>Total of pure water flux</td>
</tr>
<tr>
<td>Jₚ</td>
<td>Total of filtration</td>
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<td>J₇/J₀</td>
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<td>LMH</td>
<td>L/m². h</td>
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<td>MD</td>
<td>Membrane Distillation</td>
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<td>MF</td>
<td>Microfiltration</td>
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<td>NF</td>
<td>Nanofiltration</td>
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<td>NMP</td>
<td>N-methyl-2-pyrrolidone</td>
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<td>PEG</td>
<td>Polyethylene glycol</td>
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<td>PWF</td>
<td>Pure Water Flux</td>
</tr>
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<td>PSf</td>
<td>Polysulfone</td>
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<tr>
<td>Q</td>
<td>Permeate volume (L)</td>
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<tr>
<td>R</td>
<td>Rejection of feed components</td>
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<td>Rₙ</td>
<td>Total resistance</td>
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<td>Symbol</td>
<td>Description</td>
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<tr>
<td>$R_m$</td>
<td>Membrane resistance</td>
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<tr>
<td>$R_a$</td>
<td>Resistance due to absorption</td>
</tr>
<tr>
<td>$R_c$</td>
<td>Resistance due to cake layer</td>
</tr>
<tr>
<td>$R_{cp}$</td>
<td>Resistance due to cake polarization</td>
</tr>
<tr>
<td>RHS</td>
<td>Rice husk silica</td>
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<td>RSM</td>
<td>Response surface methodology</td>
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<td>RO</td>
<td>Reverse osmosis</td>
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<tr>
<td>SEM</td>
<td>Scanning electron microscope</td>
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<tr>
<td>SiO$_2$</td>
<td>Silica Oxide</td>
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<tr>
<td>UF</td>
<td>Ultrafiltration</td>
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<tr>
<td>Wt.%</td>
<td>Weight Percentage</td>
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<td>XRD</td>
<td>X-ray diffraction</td>
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CHAPTER 1

INTRODUCTION

1.1 Introduction

In recent years, water pollution has become a serious issue that need to be discussed and attract attention from all over the world. Based on World Health Organization society, the worst situation that facing by most people in the world is the lack of access to get clean water. As the world population is going to be increased to 1.1 billion by year 2015, the contamination problem from all sources of clean water i.e. river water, rain water, sea, lake are with unwanted material such as bacteria, floating debris and dust is becoming a serious problem that need to be handled (Tebbutt et al., 1998). Thus, many researches have been conducted recently to treat the water medium via different approaches such as activated carbon, activated alumina, aeration, ion exchange, neutralizing filter and membrane. Based on extensive study that reported in source articles, membrane technology offer much easiest way.

Basically, a membrane is divided into several types of separation such as microfiltration, ultrafiltration, nanofiltration and reverse osmosis. Ultrafiltration can be used to remove contaminants from the polluted water with the intense regulatory activity and scarce high quality source water. These stages of separation can be considered as a very promising process for drinking water production due to its pore sizes range that is between 2 - 100 nm. Ultrafiltration is able to remove viruses, bacteria, colloids, and larger particulate matter from suspensions (Dorgan, 1992). According to Haijun et al., (2009) polysulfone (PSf) is a very popular polymer and widely used in the fabrication of ultrafiltration membrane due to its good mechanical, thermal and chemical stability. However, because of its hydrophobic nature, PSf membranes are susceptible to membrane fouling by the adsorption of proteins and other biomolecules from the feed stream (Yan, Xiang, and S. Xianda, 2006). This
fouling mechanism causes the flux decline by concentration polarization where a membrane undergoes plugging or coating by some element in the stream being treated, in such a way its output or flux is reduced. In general, when fouling occurs in separation process it can affect the performance of membrane such as flux permeation, water permeability, surface porosity and morphological (Mansourizadeh and Ismail, 2010).

Most previous studies have shown that incorporation of additive into membrane formulation can play an important role in preventing the fouling problem which may cause improper morphology structure including its pore and skin layer (Vatanpour et al., 2011; Nghiem et al., 2008). The incorporation of different material or additive have contribute to significant effect to membrane performance by reducing fouling and increasing rejection. The incorporation of additives in membrane formation is expected to add value to membrane properties to make porous, increase hydrophilicity, induced antibacterial properties and enhanced membrane performances (Saljoughi et al., 2010; B.Torrestinana-Sancheza et al., 1998; Basri et al., 2010). The incorporation of most inorganic fillers such as polymeric additive, silica, aluminum, zeolite, and titanium dioxide to dope solutions is able to produce membranes with higher porosity and hydrophilicity (Ma et al., 2011; Idris et al., 2011; Jian et al., 2012 and Arthanareeswaran et al., 2007).

The potential of organic fibers from natural sources (non-hazardous element) for example rice husk (which has high silica content) was being considered due to its biodegradable properties and green technology. In fact, some inorganic additives are able to suppress the formation of macrovoids, enhance pore formation and improve pore interconnectivity and hydrophilicity of the membranes. The inorganic additive also can increase membrane permeability and control membrane surface properties (Chakrabarty et al., 2008). As reported by Yan et al., the additive of aluminum oxide (inorganic) used as additive in membrane dope formulation has improved the antifouling properties (Yan et al., 2007). By improving membrane hydrophilicity it can reduce the membrane fouling to some extent.

The addition of additive in membrane formulation is expected to change the characteristic such as pore sizes, pore distribution, physical properties and mechanical characteristic. As reported in many papers, silica can suppress the formation of amphiphilic component and macrovoids, enhance the pore formation, improve pore interconnectivity and hydrophilicity for the membrane (He et al., 2002;
Qu et al., 2010; Hero et al., 2006; Huang et al., 2012 and Arthanareeswaran et al., 2007). The incorporation of potential organic fibres from natural sources (non-hazardous element) i.e. rice husk was considered as its contain high silica compound. Besides that it also has biodegradable properties and offers green technology. Hopefully, the incorporation of this additive will improve the antifouling mechanism of the membrane and membrane performance at the same time reduces the costs.

1.2 Problem Statement

Most applications in membrane separation process i.e. chemical, food, petroleum, mining etc. having a critical problem with fouling mechanism. Membrane fouling is characterized in general as a reduction of permeate flux through the membrane, as a result of increased flow resistance due to pore blocking, concentration polarization, cake formation and absorption. Membrane fouling gives a negative impact on filtration performance as it decreases the permeate flux or increases the transmembrane-pressure (TMP). The effect of this fouling mechanism on the decrease of flux depends on factor such as membrane pore size, solute loading and pore size distribution, membrane material and operating conditions. In addition, the efficiency of membrane separation process is highly dependent on fouling effect of natural organic matter (NOM) that present in surface water. Humic substance is a major component in NOM and generally categorized into humic acid (HA) (Combe et al., 1999). Al-Amoudi reported that pH and ionic strength affect the molecular size distribution of HA (Al-Amoudi, 2010). This NOM can change the molecule to be large, flexible and linear shape at high and low pH (Al-Amoudi, 2010). In addition, the high ionic strength and concentration in HA water also accelerate the fouling formation on membrane (Wang et al., 2001). Thus it is necessary to study the anti-fouling mechanism of membrane in membrane separation process. The good and potential additive which can enhance the antifouling performance will be determined. The addition of suitable additive is wisely needed in order to modify membrane structure and properties to overcome this fouling phenomena. The modification of suitable additive in membrane formulation will be carried out and the effect of membrane properties and structure will be investigated. Silica from rice husk is highly potential as additive that induced hydrophilicity properties can enhance the antifouling agent.
1.3 Research Objectives

The objective of this work were:

i. To prepare ultrafiltration composite membrane with silica from rice husk as an additive at different weight percentage.

ii. To study the effect of rice husk silica (RHS) as additive towards polysulfone (PSf) membrane characteristic and performance.

iii. To determine the fouling properties of prepared membrane.

iv. To optimize normalized flux of PSf/RHS membrane at three difference parameter filtration such as pH, ionic strength and transmembrane-pressure by using response surface modeling (RSM).

1.4 Research Scope

In order to achieve the above mentioned objectives, the following scopes of study were drawn.

i. Preparing the silica from rice husk at burning temperature 600 °C as an additive and characterize in term of XRD pattern, particles content and size of particles.

ii. Fabricating composite membrane by preparing dope solutions from PSf as polymer material, N-Methyl-2-pyrrolidone (NMP) as solvent, PEG 400 as pore forming agent and RHS as additive at different concentration (0 – 6 wt. %) via phase inversion technique.

iii. Characterizing the prepared membrane in terms of its cross section area, surface morphology, surface roughness, particles distribution, hydrophilicity, porosity, mean pore sizes and tensile strength.

iv. Measuring performance of prepared the membrane via pure water flux (PWF) and rejection of humic acid (HA) solution by using ultrafiltration permeation testing unit.

iv. Evaluating fouling performance filtration of HA by using the ultrafiltration permeation testing unit in term of membrane resistance, normalized flux, and flux recovery after membrane chemical and physical cleaning.
v. Optimizing of PSf/RHS membrane performance on final normalized flux at three difference operating condition (pH, Ionic strength and transmembrane-pressure by using Response Surface Methodology (RSM).

1.5 Research significance

Due to the nature of current polysulfone membranes that has hydrophobic properties and tends to absorb foulants at top and inside the pore structure, therefore the enhancement of membrane to increase antifouling properties is crucially needed. In this study, the use of silica that possesses strong hydrophilicity had proven can increase membrane structure and performance. In fact, the synthesisization of silica from agro waste material which is rice husk not only can minimize the pollution but also introduce green materials to membrane technology. Furthermore, the use of RSM also provides the variable response in getting the optimized range of filtration operation which benefit to membrane technology.
CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will discuss on previous related works on the membrane separation process. This section will explain the effect of fouling towards membrane performance. Further explanation on membrane modification and fabrication will also be highlighted.

2.2 Membrane Separation Process

Membrane separation can be defined as the filtration of two or more components from a fluid stream based on size difference (Munir, 2000). According to Mulder the definition of membrane is a selective barrier between two phases (Marcel Mulder, 1991). The term ‘selective’ being inherent to a membrane or a membrane process. Membrane separation describes the ability of a membrane to control the permeation rate of particles and molecules passing through the membrane. The membrane has the ability to transport one component more readily than the other because of differences in physical or chemical properties between the membrane and the permeating components. The movements of those components across the membrane need a driving force. Figure 2.1 shows the schematic diagram of membrane separation concept. The diagram demonstrated that phase 1 is that usually considered as feed or upstream side while phase 2 is considered as permeates or downstream side (Marcel Mulder, 1991). The solute bigger particle has tendency to foulant and blocked the inner pore, meanwhile the smaller particle will pass through the membrane which is called permeates. The concentration bigger particle of blocked by the membrane calculated to get rejection data.
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