UNIVERSITI PUTRA MALAYSIA

EVALUATION OF CHARACTERISTICS AND PERFORMANCE OF FABRICATED POLYSULFONE/CELLULOSE ACETATE ULTRAFILTRATION MEMBRANE

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By

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June 2018

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Ultrafiltration is a separation technique, which is specifically for biomolecular separation and it has been widely used in food and pharmaceutical industries. The performance and characteristics of fabricated ultrafiltration membrane have gained some concern in producing a new membrane with robust characteristics. Therefore, the selection of membrane material is very important to improve the membrane performance. In this study, the objectives involve development and formulations of polysulfone/cellulose acetate (PSf/CA) ultrafiltration membranes and evaluating the flux performance and the characteristics of the fabricated PSf/CA ultrafiltration membrane. This study involved three different formulations of polymer ratio, polymer concentration, and additives compositions. The polymer ratio varied into three different ratio of PSf/CA compositions, which were 90/10, 80/20, and 70/30. Meanwhile, the formulations of polymer concentration were compared with different concentrations of 15 wt. %, 17.5 wt. % and 20 wt. % and further modification was carried out for selected membrane by the additive compositions. The additives involved were polyvinylpyrrolidone (PVP), pluronic (Plu) and PVP/Plu. All the fabricated membranes were evaluated for compaction, pure water flux, hydraulic resistance, and fouling characteristic. Further, these membranes were characterized for chemical structure identification by fourier transform infrared (FTIR), morphology by scanning electron microscopy (SEM) and mechanical properties test. From overall evaluation and characterization, PSf/CA-20 membrane obtained the most uniform flux permeation and good membrane morphology but low flux recovery. However, further modification with additives composition showed that PSf/CA-PVP/Plu obtained a good flux permeation, flux recovery and satisfied polyphenol rejection. The exposure of PSf/CA-PVP/Plu membrane to the different hydraulic cleaning methods resulted in similar range of flux recovery ratio (FRR %) between 67 % to 79 % compared to the membrane with single additive; PVP or Plu. Hence, PSf/CA-PVP/Plu membrane is the more preferred membrane, which can be further applied to the

related industries due to the improved morphology, which are advantages to the flux permeation and fouling resistance.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENILAIAN TENTANG CIRI-CIRI DAN PRESTASI MEMBRAN ULTRATURASAN POLISULFON/SELULOSA ASETAT YANG DIREKA

Oleh

WAN AISYAH FADILAH BINTI WAE ABDULKADIR USIN

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Ultraturasan adalah teknik pemisahan yang khusus untuk pemisahan biomolekul dan telah digunakan secara meluas dalam industri makanan dan farmaseutikal. Prestasi dan ciri-ciri membran ultraturasan yang direka telah menimbulkan kebimbangan dalam menghasilkan membran baru dengan ciri-ciri yang mantap. Oleh itu, pemilihan bahan membran sangat penting untuk meningkatkan prestasi membran. Objektif kajian ini adalah untuk menghasilkan rumusan membran ultraturasan polisulfon/ selulosa asetat (PSf/CA) dan menilai prestasi aliran dan ciri-ciri membran ultraturasan PSf/CA yang direka. Kajian ini melibatkan tiga nisbah polimer, kepekatan polimer, dan komposisi bahan tambah yang berbeza. Tiga nisbah polimer yang berbeza untuk komposisi PSf/CA ialah 90/10, 80/20 dan 70/30. Sementara itu, rumusan untuk kepekatan polimer telah dibandingkan dengan tiga kepekatan yang berbeza iaitu 15 wt. %, 17.5 wt. % dan 20 wt. % dan pengubahsuaian selanjutnya telah dilakukan dengan penambahan bahan tambah bagi membran yang terpilih. Bahan tambah yang terlibat ialah polivinilpirolidon (PVP), pluronik (Plu) dan kombinasi PVP/Plu. Semua membran yang direka telah dinilai untuk pemadatan, aliran air tulen, rintangan hidraulik, dan ciri-ciri mendakan. Selanjutnya, membran-membran ini telah dikenalpasti struktur kimia melalui fourier mengubah inframerah (FTIR), morfologi melalui pengimbasan mikroskop elektron (SEM) dan menguji sifat mekanikalnya. Dari keseluruhan keputusan ujikaji, membran PSf/CA-20 telah memperolehi aliran peresapan yang baik, pemulihan aliran dan penolakan polifenol yang memuaskan. Membran PSf/CA-PVP/Plu telah menunjukan nisbah pemulihan aliran (FRR%) yang baik dimana nilai FRR% adalah antara 67 % hingga 79 % apabila didedahkan kepada dua pembersihan hidraulik yang berbeza berbanding dengan membran yang mempunyai satu bahan tambah; PVP atau Plu. Oleh itu, PSf/CA-PVP/Plu membran adalah membran yang sesuai untuk digunakan dalam industri yang berkaitan. Ini kerana peningkatan dalam morfologi membran ini mampu memberi kebaikan untuk aliran peresapan dan rintangan bagi mendakan.

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I would like to end this segment with an encouraging thought by Hellen Keller:

“Character cannot be developed in ease and quiet. Only through experience of trial and suffering can the soul be strengthened, ambition inspired, and success achieved.”

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

MF	Microfiltration
UF	Ultrafiltration
NF	Nanofiltration
RO	Reverse Osmosis
PES	Polyethersulfone
PSf	Polysulfone
CA	Cellulose Acetate
PVP	Polyvinylpyrrolidone
PEG	Polyethylene glycol
Plu	Pluronic
PWP	Pure water permeation
R_m	Hydraulic Resistance
FRR	Flux Recovery Ratio
TMP	Transmembrane Pressure

CHAPTER 1

INTRODUCTION

1.1 Background Study

Membrane technology is one of the new invention in treating wastewater in Malaysia. This technology is applied in various industries such as food, pharmaceutical, as well as in palm oil industry to reduce the high production of waste. The fabrication of the membrane can meet the industry expectation that lead to many advantages for Malaysia industry. Thus, this economically reduces the cost. Nowadays, the shortage of natural sources has supported the Malaysia's experts to investigate the enhancement of wastewater disposal treatment in producing drinking water from unexpected sources. The production of a high purity and quality of water for various purposes was declared by former researcher to use different types of membrane processes *e.g*. ultrafiltration with reverse osmosis membrane (Nicolaisen, 2002) in a water treatment.

In membrane technology field, there are four well-known water separations processes; microfiltration (MF), ultafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) (Baker, 2004). The difference between these classifications is depending on the formation of pore size inside the membranes and the specification of material selection. Besides, their uses have been successfully proven in various industries such as reverse osmosis in desalination technology (Lee et al., 2011) and ultrafiltration as a tool for concentration and separation in food processing industry (Mohammad et al., 2012). Besides, Baker stated that, the ultrafiltration membranes were used to filter the dissolved macromolecules in a solutions (Baker, 2004) and it reclaimed to be one of an appropriate separation process that reduces the cost of the treatment. The ultrafiltration membranes have range of pores that are suitable for biomolecules separation. Thus, for this type of separation, it is very useful for application of food industry.

In order to produce a robust ultrafiltration membrane, the selection of material for membrane preparation is an important factor in polymer blending. This will assist the formation of new characteristics of fabricated membrane. Besides, polymer is the main material involved in the membrane fabrication, which determines the membrane characteristic. Most of previous researchers tended to used hydrophobic polymer with modification of some additives to improve the limitation of main polymer, e.g. polyethersulfone (PES) with pluronic F127 as additive for ultrafiltration separation (Zhao et al., 2008) and ultrafiltration polyvinylidene fluoride (PVDF) membranes were modified with amphiphilic polymer and linear hydrophilic polymer for comparison study (Zhao et al., 2008). Hence, this shows that the addition of hydrophobic and hydrophilic polymer has enhanced the performance of the fabricated membranes.

1.2 Problem Statement

The technical innovation in producing new polymeric membranes with high demand on the filtration properties with lower cost has introduced a membrane treatment process in many different industries such as pharmaceutical, textile and wastewater treatment (Razzaghi et al., 2014). Moreover, reuse wastewater from membrane process has been widely used to overcome the shortage of water resources and many requirements for clean water (Huang et al., 2012). Therefore, due to the economic competitive of the existing separation technologies including aggressive environments challenges for membranes, many applications seek for more applicable and strong membrane materials which give better properties on permeability and selectivity. In order to fabricate and to produce a novel and compatible membrane as demanded, the selection of membrane materials is a crucial research area to be investigated because the efficiency of membrane is highly depends on the formulation. Therefore, the limitations of single polymer properties have been overcome by polymer blending technique.

Cellulose acetate (CA) is a potential hydrophilic organic polymer for membrane fabrication, which can be explored in the polysulfone (PSf) polymer blend technique. CA is an adaptable material and has attracted much attention due to its outstanding performance such as good toughness, high biocompatibility and relatively low cost (Han et al., 2013). The presence of acidic and carbonyl functional groups on its structure has facilitated the enhancement of PSf membrane performance by improving the hydrophilic characteristic of the blend membrane. Idris and Ahmad (2011) claimed that the combination of CA and polyethersulfone (PES) has found to reduce remarkably the harmful components consist in wastewater of agro-industry in Malaysia. This was due to the high concentration of total polymer and compatible ratio of both polymers that facilitated the high separation of solute and at the same time improved the flux permeation of the blend membrane. The hydrophilic characteristic of CA had improved the hydrophobic characteristic of PES and the high mechanical strength of PES enhanced the mechanical properties of the blend CA/PES membrane. Even though, PES polymer has similar chemical and thermal limits to PSf, it still can obtain a markedly different performance during flux permeation process (Ali, 2013). In view of this, an attempt has been made to study the effect of using different polymer ratio and concentration of CA in casting solution with further improvement by additives composition in term of flux permeation, characteristics and fouling resistance of PSf based membranes.

The formation of asymmetric UF membrane is highly influenced by the other components such as solvent, non-solvent and additive other than polymers and these components have proved to affect the characteristics of membrane. The presence of additive in the polymer matrix plays an important role in adjusting the properties of membrane. In general, additives encourage the formation and interconnectivity of pore, induce sponge-like pore to suppress the macrovoid formation and improve hydrophilicity (Ali, 2013; Rahimpour & Madaeni, 2007). Usually, a hydrophilic additive is blend together in casting solution to form a hydrophilic membrane. However, there is a study which investigates using two hydrophilic additives; polyvinylpyrrolidone (PVP) and polyethylene glycol (PEG) in order to improve the hydrophilicity and porosity of the blend membrane (Arthanareeswaran & Kumar, 2010). The investigation on the concentration and the effect between single and combined additives provides a potential platform for developing a better performance membrane. The two factors show a significant influence in the membrane fabrication process by changing the molecular structure during formation of the better performance membrane.

In this study, PSf is chosen as the main polymer for PSf/CA blend ultrafiltration membranes. The effects of different polymer ratio and concentration of PSf/CA composition were studied in term of flux permeation, morphology, mechanical strength, and fouling characteristic of PSf/CA blend membrane. The results of PSf/CA polymer ratio were compared with the pure PSf membrane. Then, the best polymer ratio of PSf/CA membrane was compared with other concentration of similar polymer ratio. Therefore, the best polymer ratio and concentration of PSf/CA was selected for investigation on the role of different type of additives; PVP (hydrophilic), and pluronic F127 (Plu, amphiphilic) in the polymer matrix. PSf/CA membranes with additives were prepared by varying the additive compositions of PVP, Plu and PVP/Plu, respectively. The effect of these compositions was investigated to find the best performance of PSf/CA blend membrane with additive. Besides, the hydrophilic and amphiphilic additives had decided to be used in order to observe their interaction in the polymer matrix as the amphiphilic additive could encourage the stability of membrane hydrophilicity. Next, the PSf/CA membranes with additives were further evaluated on the flux recovery ratio and the selective removal of polyphenol in the commercial apple juice. This was carried out in order to identify the ability of the selected membranes to be used for several ultrafiltration usage after the permeation of real industry product and the selectivity of the fabricated membranes.

1.3 Objectives

i. To develop formulations of the polysulfone/cellulose acetate (PSf/CA) ultrafiltration (UF) membranes.
ii. To evaluate the flux performance and the characteristics of the fabricated PSf/CA ultrafiltration membranes

1.4 Scope of Research

This research focuses on the development of newly formulated ultrafiltration membrane of PSf/CA composition that relates to the evaluation of flux performance and the characteristics of new membranes.
The scopes of this study are as follows:

i. Formulating the polymer solution of asymmetric PSf/CA ultrafiltration membranes.
ii. Casting the PSf/CA polymer solution with water / pre-treatment with ethanol and n-hexane and drying at room temperature.
iii. Fabricating PSf/CA ultrafiltration (UF) membranes based on molecularly phase inversion process containing different polymer composition of PSf/CA.
iv. Analysing the influence of different polymer ratio and concentration, and types of additives on PSf/CA membranes performance.
v. Characterizing the morphologies of fabricated ultrafiltration membranes.
vi. Determining the efficiency of the improvement of UF fabricated membrane.

The scope of research for the first objective is related to the formulation and fabrication of the PSf/CA ultrafiltration membrane in order to form a thin film membrane. For the second objective, the effects of selected materials are investigated in term of compaction test, pure water permeation (PWP), hydraulic resistance, and fouling characteristics. The characteristics of these membranes are further studied by chemical structure identification, morphology analysis and mechanical properties test.

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