THE EFFECT OF COATING CONDITION AND AQUEOUS PHASE COMPOSITION ON THE PERFORMANCE OF THIN FILM COMPOSITE REVERSE OSMOSIS FLAT SHEET MEMBRANES

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ABSTRACT

Thin film composite (TFC) layers are formed using interfacial polymerization reaction between an aqueous phase and organic phase on membrane supports. In the preparation of thin film composite membrane, there are many interfacial reaction factors that influence the membrane performance, amongst them are dipping time, curing temperature and composition of aqueous phase. Thus in this project these factors were studied in two stages. In the preliminary stage, the effect of dipping time and curing temperature on the performance of TFC membrane were evaluated using two different kinds of polymer membrane supports, cellulose acetate and polysulfone. Initial results revealed that dipping time does not affect the performance of TFC membranes especially rejection rate but curing temperature have significant influence. Curing temperature ranging from 40°C to 100°C was used during the interfacial reaction process. Cellulose acetate TFC membranes cured at 60°C exhibited highest rejection rate of 76% whilst polysulfone TFC membranes cured at 80°C showed not only highest rejection rate of 80% but also excellent flux rates. Since polysulfone TFC membranes showed superior performance compared to cellulose acetate, it is chosen for the second stage of the experiment. In this stage, a systematic experimental design based on the response surface methodology was used to identify the significant interfacial reaction factors which influence the membrane performance. The factors considered were the composition of aqueous phase that includes the ratio of *m*-phenyldiamine to hydroquinone as monomer, percent of tetrabutylammonium bromide as a catalyst and percent of sodium hydroxide as an acid acceptor. Rejection and flux rates were the response variables investigated. The experimental results indicate that the proposed mathematical model suggested could adequately describe the performance indicators within the limits of the factors that are being investigated.

ABSTRAK

Membran komposit filem nipis dihasilkan melalui tindak balas pempolimeran antara muka di antara fasa akuas dengan fasa organik di atas permukaan membran penyokong. Dalam penyediaan membran komposit filem nipis, terdapat beberapa faktor yang mempengaruhi antaranya masa pencelupan dalam fasa akuas dan suhu rawatan. Oleh itu melalui penyelidikan ini, kesan faktor-faktor tersebut dikaji melalui dua peringkat. Melalui peringkat pertama, kesan masa pencelupan dalam fasa akuas dan suhu rawatan diuji di atas dua jenis membran penyokong iaitu polisulfona dan selulosa asetat. Keputusan awal menunjukkan masa pencelupan dalam fasa akuas tidak mempengaruhi pekali penyingkiran secara siknifikan tetapi mempengaruhi kadar fluks. Suhu rawatan didapati mempengaruhi prestasi membran pada keseluruhannya Suhu rawatan di antara 40°C hingga 100°C diaplikasikan dalam penyediaan membran komposit filem nipis. Pekali penyingkiran untuk membran komposit selulosa asetat pada suhu 60°C menunjukkan keputusan tertinggi iaitu 76% manakala membran komposit polisulfona pada suhu rawatan 80°C bukan sahaja mencapai pekali penyingkiran yang lebih baik iaitu 80% malahan menunjukkan kadar fluks yang tinggi. Memandangkan prestasi keseluruhan membran polisulfona didapati lebih baik, lalu ia diaplikasikan pada peringkat seterusnya. Pada peringkat kedua, rekabentuk eksperimen dipilih berdasarkan kaedah tindak balas permukaan bagi mengenalpasti faktor dalam tindak balas antara muka yang sangat mempengaruhi prestasi membran. Faktor yang dipilih ialah nisbah *m*-phenildiamina terhadap hidrokuinon sebagai monomer, peratus tetrabutilammonium bromida sebagai katalis dan peratus natrium hidroksida sebagai asid penerima. Pekali penyingkiran dan kadar fluks dipilih sebagai reaksi variasi. Keputusan eksperimen menunjukkan model matematik yang dicadangkan cukup untuk menjadi penunjuk prestasi daripada keseluruhan faktor yang dikaji.

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LIST OF SYMBOLS

A	-	Pure water permeability constant (g-mol/cm^{2}sPa)
c_{Bm}	-	Concentration of water in membrane (mol/m^3)
c_1, c_2, c_3		Molar density of feed solution, concentrated boundary solution and product solution respectively (g-mol/m^3)
D_{BM}	-	Diffusion coefficient of water in the membrane (m^2/s)
$\frac{D_{AM}}{K\delta}$	-	Solute transport parameter (m/s)
FR		Flux rate (m^3/m^{2}day)
f'		True value of solute separation by membrane pore
G	-	Gas constant (8.314J/mol K)
J_v	-	Water flux (mol/m^{2}s)
J_A	-	Solute flux (mol/m^{2}s)
J_B	-	Solvent water flux (mol/m^{2}s)
k	-	Mass transfer coefficient on the high pressure side of membrane (m/s)
ℓ_p	-	Hydrodynamic permeability coefficient (m/skPa)
M_W	-	Molecular weight of water (kg/kmol)
P	-	Pressure (Pa)
P_A	-	Reflection coefficient solute (m^2/s)
P_B	-	Reflection coefficient water (m^2/skPa)
PWP	-	Pure water permeability through effective area of membrane surface (m^3/m^{2}day)
R	-	Fraction of solute separation (%)
S	-	Effective membrane area (cm^2)

T	-	Absolute temperature (Kelvin)
v_B	-	Molar volume of water (m^3/mol)
X_{A1}, X_{A2}, X_{A3}		Mole fraction of feed solution, concentrated boundary solution and product solution respectively (g-mol/cm^3)
ΔP	-	Pressure different across membrane (Pa)
Δx	-	Membrane thickness (m)
$\Delta \pi$	-	Osmotic pressure different across the membrane (Pa)

Greek letters

σ	-	Reflection coefficient
ω	-	Solute permeability coefficient (kmol/m^2skPa)
π	-	Osmotic pressure of solution (Pa)
$b(\rho)$	-	Dimensionless friction function
ϕ		Potential in the interfacial force field
$\phi(\rho)$	-	Dimensionless potential function
β_2	-	Dimensionless operating pressure
$\alpha(\rho)$	-	Dimensionless solution velocity profile in the pore
β_1	-	Dimensionless solution viscosity

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CHAPTER 1

INTRODUCTION

1.1 Overview

Membrane technology is still evolving and finding more and more applications in a broad range of fields and the development of membranes will strongly influence separation process in the future. Rapid growth in membrane technology development is primarily based on consciousness on the potential of this technology. The technology contributes to the solution of some of the most crucial problem nowadays. It has been widely used in many applications like industrial wastewater treatment, desalination of sea and brackish water and liquid food treatment.

In membrane separations, each membrane has the ability to transport one component more readily than the other because of differences in physical and chemical properties between the membrane and the permeating components. Furthermore, some components can freely permeate through the membrane, while others will be retained. The stream containing the components that permeate through the membrane is called permeate and the stream containing the retained components is called retentate. The transport of permeate across the membrane is achieved by the application of either mechanical, chemical, electrical or thermal works (Scott, 1998).

Reverse osmosis is a well-developed industrial membrane separation processes. This process is well established and the market is served by a number of experienced companies. The separation process for reverse osmosis (RO) is not restricted to aqueous based solution, but it can essentially separate all solute species both inorganic and organic from solution. It involved the application of mechanical pressure without using any other energy like heat. Thus, RO is energy-saving separation process and indirectly reduce the cost of operation. The use of RO encompasses a variety of industries especially in the desalination to produce potable water.

Desalination of sea or brackish water entails forcing salt solution through a permselective membrane at pressure, which is sufficiently high to overcome the osmotic forces and tends to drive water in the opposite direction. These membranes must allow water to permeate at high rate but must reject permeation of the salt molecules to a high degree.

A breakthrough of RO membranes to industrial applications begun in 1960 with the invention of the first integrally skinned asymmetric cellulose acetate hyperfiltration membrane by Loeb and Sourirajan (Kesting, 1985). This membrane consist of a very dense top layer or skin with thickness of 0.1 to 0.5µm supported by a porous sub layer with a thickness of about 50 to 150µm. These membranes combine the high selectivity of a dense membrane with the high permeation of a very thin membrane.

In 1970's, the first commercial composite reverse osmosis (RO) membrane was developed. The membrane consists of a very thin dense top layer, which is supported by a porous sub layer of a different material, which is quite different from the asymmetric cellulose acetate membrane where it is developed with two layers of the same material. The advantage of the so-called thin film composite (TFC) membrane is that each layer can be optimized independently to obtain optimal membrane performance with respect to selectivity, permeation rate and chemical and thermal stability.

1.2 Background of the Problem

Thin film composite (TFC) membrane was developed by a combination of two or sometimes three layers that were made of totally different material, structure and function. A thin dense active layer consists of a very thin film supported by microporous support reinforced onto polyester fabric. In laboratory studies, the microporous layer need not actually be directly coated onto a fabric base, as it is proved to be difficult and inconvenient when done by hand (Peterson, 1993). Rather the microporous film can be casted on a glass plate because the fabric base is responsible for the formation of membrane defects as well as additional membrane pores (Berg and Smolders, 1990).

Microporous layer are commonly synthesized using phase inversion to develop an asymmetric membrane. It comprises almost the entire thickness and provides the required mechanical strength. Polysulfone are commonly used as membrane polymer because of their high performance, tough and has high temperature and chlorine resistant characteristic. Polysulfone is a hydrophobic polymer thus hydrophilic polymer such as polyvinylpyrrolidone and n-2-methylpyrrolidone are added as solvents.

Several techniques can be used to apply an ultrathin active layer upon a support like dip coating, spray coating, spin coating, interfacial polymerization, plasma polymerization and grafting, but the interfacial polymerization concept has been predominated as the optimal system (Peterson, 1993). Thin film composite structure membranes fabricated via interfacial polymerization meet this demand. The two phases that are involved in interfacial polymerization process includes aqueous phase and organic phase to form the active layer having semi permeability. The material as well as polymer molecular structure of the polymer in both of the two phases influenced the permeation properties of membrane performance (Arthur, 1989).

The successful interfacially membrane formed by Cadotte at Northstar Research in 1978 consists of a combination of aromatic amines with *aromatic* acyl chloride (Peterson, 1993). Best result was obtained by the reaction of *m*-phenylenediamine as aqueous phase and trimesoyl chloride as organic phase. The TFC polyamide membrane that is composed of a fully aromatic network structure was dominated as an outstanding recipe to produce good rejection rate and at the same time acceptable water permeability.

Polyamides have excellent intrinsic separation characteristics for reverse osmosis, however their chlorine tolerance is relatively low (Kawaguchi and Tamura, 1984; Tran *et al.*, 1989). Chlorine was widely added to water as a disinfectant and bactericide. A membrane is considered chlorine resistant, when it can withstand exposure for several years in a biocidal concentration of 1mg/l chlorine (Rajinder, 1994). Many attempts have been made to improve the chlorine resistant of composite membrane by changing molecular structure of the monomers used for the polymerization.

Blais (1977) has reported the correlation between chemical structure and membrane performance of polyamides. Few studies have been done to investigate the correlation between chemical structure of polyamides and their oxidation resistance (Kawaguchi and Tamura, 1984). Glater *et al.* (1983) have recently reported the sensitivity of polyamide to halogen disinfections by monitoring the decay of the membrane performance. A more extensive study of model-compound chlorine sensitivity was reported by Lowell *et al*. (Glater *et al.*, 1994). He found that ester linkages were generally chlorine resistant, in agreement with Jayarani and Kulkarni (2000) when they developed composite membrane with the incorporation of hydroquinone as an ester linkage. The membrane named as composite polyesteramides showed higher chlorine tolerance compared to commercial composite polyamide. Thus, based on these references, this study is aimed to develop TFC polyesteramide membrane by interfacial polymerization of aromatic amines in the presence of aromatic diol as ester linkages with *aromatic* acyl chloride on a microporous support.

Available TFC reverse osmosis membranes in the market, are highly effective in their intended application for desalination and industrial process water due to continuing searching for new and improved polymers for RO membrane materials (Jian and Ming, 1987; Ibbora *et al.*, 1996). To date, most of the research done was to improve thin film composite membrane performance by changing the structure of membrane monomer or the coating conditions. A few researchers studied the effect of polyamide molecular structure on the performance of reverse osmosis membrane (Cadotte, 1981; Hirose *et al.,* 1997). Roh *et al.* (2002) observed the influence of rupture strength of thin film structure, whilst Arthur (1989) investigated the structure and properties relationship of the thin film composite membrane.

The relationship between separation properties and coating condition is of particular importance for the development of new TFC membrane (Kim *et al.*, 2000; Rao *et al*., 2003). The exact coating condition is important for attaining the desired stability of thin film composite membranes. This stability is important to give high water permeability and rejection rate. Thus, the major emphasis now seem to be focused on optimizing the membrane coating conditions and also to study how these conditions effect the structural changes on membrane formation of TFC membrane so as to enhance separation properties.

Most of the research work done previously had studied the performance of TFC membrane using polysulfone as a porous support. Literature search seem to suggest that there has been no study using cellulose acetate as a porous support for the TFC membranes. Thus, this study investigates the possibility of using cellulose acetate as a porous support and compares its performance with polysulfone microporous support membrane. Cellulose acetate was preferably used as a microporous support because of its low cost and low tolerant to chlorine reaction. In cellulose acetate, spaces in water swollen polymer matrix were the primary provider of continuous flow channel that contribute to the separation of salt and small organic molecules (Khulbe *et al*., 2002).

The growth of the interfacially polymerized film was influence by aqueous phase composition (Bartels and Kreuz, 1987; Bartels, 1989). Until now, not much has been said regarding the effect of the composition of aqueous phase such as the effect of the catalyst and acid acceptor on the performance of membrane. The catalyst is one of the factors affecting the reverse osmosis performance of the membrane (Wang, 1988a). The highest permeation rate was obtained in the presence of catalyst in the membrane recipe.

The aqueous phase consists of alkaline amine solution, particularly when caustic is used as an acid acceptor. Acid acceptors are commonly added in aqueous phase as a neutralizer for hydrogen halide generated during the course of the reaction. A study of acid acceptor showed that base strength of the acid acceptor affected the degree of concurrent hydrolysis (Cadotte, 1979). A small amount of acid acceptor is enough in the preparation of TFC membrane.

Most of the previous research on membrane performance usually use one-factor-at-a-time experimental approach which can be time consuming and exorbitant in cost (Haaland, 1989). The conventional practice consisting in varying one variable at time does not allow evaluation of the combined effects of all the factors involved in the process and constitutes a time consuming methodology (Cochran and Cox, 1992). Recently, statistically approach was increasingly used for optimization steps in membrane process. Ismail and Lai (2004) developed the defect free asymmetric polysulfone membranes for gas separation using response surface methodology. Ani *et al.* (2002) has used the Taguchi method which is a statistical design to determine the significant factors affecting the spinning process and the optimal spinning parameter. Chau *et al.* (1995) had studied phase inversion factors influencing polysulfone ultrafiltration hollow fiber membranes fabrication in a systematic manner using the orthogonal array method, whilst Pesek and Koros (1994) studied the influence of four factors in production of gas membranes using the complete 2^k factorial methods.

Thus in this study an attempt is made to investigate the composition effect of the aqueous phase used on the interfacial polymerization of TFC reverse osmosis membrane performance using RSM. This research continues the quest for producing practical thin composite membrane with high rejection and flux rates. Emphasis is however placed towards studying the effect of the concentration of monomer, catalyst and acid acceptor in view of the fact that only a considerable amount of research had been done in this area. Using the RSM method, parameter interaction and optimum composition can also be determined. This work has demonstrated the use of a central composite design (CCD), which is the most popular class of RSM design. The rejection and flux rates were the response variables investigated.

Membrane morphology is very much related to the membrane performance. Recent advances in microscopy have led to attempts to correlate surface characteristics to the performance of membrane. Scanning electron microscopy (SEM) and atomic force microscopy (AFM) can provide direct characterization of membrane morphology with the aid of image analysis. The scanning electron microscopy (SEM) is a powerful tool to investigate the morphology of membranes. SEM not only views the cross-section of the membrane, but also shows the surface of the top layer and bottom layer of the membrane. Recently, atomic force microscopy (AFM) became popular and many AFM pictures of reverse osmosis membrane surface had been taken in attempts to relate morphology to membrane performance. Hirose *et al.* (1996) found a relationship between the flux of reverse osmosis membranes and their roughness parameters measured by AFM. According to their experiments, an increase in surface roughness resulted in a higher water permeation flux. This theory was confirmed by consequent research (Gao and Chen, 1998). Thus, in this study, an attempt is also made to correlate membrane morphology and membrane performance using SEM and AFM.

1.3 Objective of the Study

The objectives of the research are to investigate the effect of coating conditions such as dipping time and curing temperature on the TFC membranes performance using both cellulose acetate and polysulfone as the microporous support. The performances of these membranes were then compared. Consequently, the effect of aqueous phase composition such as monomer ratio, concentration of acid acceptor and catalyst is studied on using RSM central composite design (α=2) in order to identify the significant factors and to develop a mathematical model thus enabling the prediction of responses. Finally, the correlation of membrane morphology with fabrication conditions was studied to extend knowledge for producing high rejection and flux rate thin film composite membrane.

1.4 Scope of the Study

In order to obtain the objectives listed above, the scope of the study are identified as follows:

i. The active layer of TFC membranes is prepared using interfacial polymerization method, while the asymmetric microporous membranes were prepared using phase inversion methods. The microporous material comprises of two polymers, polysulfone or cellulose acetate. Various dipping and curing condition were used so as to determine their effects on the performance of TFC membranes.

ii. Membrane permeation study was evaluated in terms of rejection rate and flux rate. Transport properties were also determined using transport parameter equation by Kimura-Sourirajan analysis, while membrane morphology was characterized using scanning electron microscopy (SEM).

iii. Influencing factors such as ratio of monomer, percent of catalyst and percent of acid acceptor were studied using RSM, using the membrane with the favorable performance based on initial finding i) and ii). A total of 20 experiments were carried out and tested in a dead end permeation cell to obtain the flux and rejection rate, which are the response variables. This response variables obtained was evaluated and analyzed using response surface methodology so as to determine the significant factors. Based on the significant factors identified, the relationship of each factor with the response variable was determined so as to predict the mathematical model.

iv. Finally, atomic force microscopy was used to correlate the relationship between membrane structure and membrane performance.

1.5 Outline of the Thesis

The thesis is basically divided into five chapters. An overview, background of the problem, research objective and scope of this research are presented in Chapter 1. A comprehensive literature review had been carried out prior to any experimental work. Literature review was conducted in providing state of the art background to the research project and these were discussed in detail in Chapter 2. Chapter 3 provides preliminary studies for coating condition and comparison between cellulose acetate and polysulfone as porous support. In this chapter, membrane performance in term of rejection rate, flux rate, transport properties and membrane morphology was identified and discussed. Chapter 4 presents the application of response surface methodology in describing significant factor affecting TFC production, clarify the interaction between parameters and proposed mathematical models for predicting TFC membrane performance. The membrane morphology was also characterized. Finally, Chapter 5 discusses the overall objective of this research and concludes the outcome of research project. Some recommendation for future studies also discussed. The schematic diagram summarizing the overall methodology experimental is shown in Figure 1.1.

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