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Characterization of Polysulfone Membrane with Variation of Ethanol Concentrations in Coagulation Bath for Ultrafiltration Membrane

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Abstract: The aim of this research is to study influence of ethanol concentrations in coagulation bath to charecteristic of physical properties and polysulfone membrane performance. Ethanol concentrations that used is 0; 40; 50; 60 ; 70; 80; 90 %. Process of making for polysulfone ultrafiltration membrane is prepared with phase inversion technique. Polysulfone membrane is made from polysulfone solved in N,N-dimetilacetamida (DMAc) and additive poly(ethylene glikol) (PEG) 600 in proportion 18 %: 77 %: 5 %. The result showed that increasing ethanol concentrations causes the increasing of density and increase concentrations from 0 - 50 % causes increasing *swelling* degree, but decreasing *swelling* degree from concentrations 60 - 90 %. However influence of ethanol concentration in coagulation bath to membrane performance is increasing of water flux, permeability coefficient, and rejection coefficient to dextran. It based on of result that polysulfone membrane with ethanol concentrations 80 dan 90 % included membrane classification ultrafiltration by rejection coefficient value above 90 %.

Keywords: Polysulfone, ethanol, hydrophilicity, permeability, rejection.

INTRODUCTION

One of the membrane classifications based on pore size and pressure is the ultrafiltration (UF) membrane. Ultrafiltration membranes have pore sizes ranging from 0.1 µm to 1 nm [1]. The working principle of ultrafiltration membranes is generally used to retain colloids and macromolecules but to pass salt and water particles [2]. Polysulfone is one of the ultrafiltration membrane materials. The use of polysulfone is because it has thermal stability and has resistance to extreme pH [3]. Conversely, polysulfone tends to be hydrophobic so that its permeability for aqueous solution systems is not very good [4]. One way that can be used to overcome this deficiency is by varying the concentration of ethanol in the coagulation bath to increase the hydrophilicity of the membrane and to increase the permeability of the membrane.

Making ultrafiltration membranes can be done by several techniques, including phase inversion. The phase inversion method is the method most often used, because the manufacturing process is easy and can be used for all types of polymers [5].

One of the parameters that influence the formation of membrane structures using the phase inversion technique is the composition of the coagulation bath, which in this study uses ethanol. Parameters that determine the quality of ultrafiltration membranes include physical properties (density and degree of swelling) and membrane performance (flux and permeselectivity). Good quality membranes are expected to have physical properties such as density that is close to the density of the original material, flux, high permselectivity and high hydrophilicity (He C, et al, 2007).

EXPERIMENTAL

The materials used in this study were distilled water, filter paper, polysulfone pa (pure analysis) (BM= 35,000 D, ρ= 1.24 g/cm^3) from Sigma-Aldrich, DMAc solution (Dimethyl Acetamide) (ρ = 0.94 g/cm^3) from Merck, Polyethylene glycol 600 (PEG600) (BM= 570 g/mol, ρ = 1.13Kg/L) from Merck, dextran from Sigma Aldrich (BM 35 - 45 kDa and 100 - 200 kDa), phenol 5 %, sulfuric acid (ρ = 1.84 g/cm^3, purify 96%, pa).

Preparation of Polysulfone Membrane

3.6 grams of polysulfone added 14.48 mL of DMAc, 1.1 mL of PEG600 in an Erlenmayer glass covered with aluminum foil. The mixture was left for several hours (until homogeneous) accompanied by stirring until the dissolution process was complete. The resulting solution is called a dope solution. The dope solution is poured onto the surface of the glass plate which has been covered with tape on the edges, then pressed evenly over the entire surface of the glass using a glass cylinder to form a polysulfone film. Furthermore, the films were put into a coagulation bath containing ethanol with various concentrations of 0 (as a control); 40; 50; 60; 70; 80; 90 % *v/v* for a few moments so that solidification occurs.

Characterize of the membrane

Density

The density value of the membrane can be obtained by calculating the ratio of the mass of the film (g) to the volume of the film (cm^3).

Swelling

Testing the degree of swelling is done by weighing the weight of the membrane before and after immersion in water. The value of the degree of inflation is obtained by using the following equation (Zhong, 2008):

$$Swelling = \frac{m_t - m_a}{m_a}$$

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RESULT AND DISCUSSION

The polysulfone membrane formulation is a dope solution consisting of polysulfone polymer, DMAc solvent (DimethylAcetamide) and Poly(ethylene glycol)600 (PEG600) additive. With a composition of 18 %, 77 % and 5 %. The dissolution process takes ± 10 hours to obtain a homogeneous dope solution. The dope solution was then printed on a glass plate and then put into a coagulation bath containing ethanol from a concentration of 0 - 90%. During the coagulation process, solidification occurs to form a solid matrix, namely the membrane. Polysulfone membrane formulation is a dope solution consisting of polysulfone polymer, DMAc solvent (DimethylAcetamide) and PEG600 additive. With a composition of 18 %, 77 % and 5 %. The dissolution process takes ± 10 hours to obtain a homogeneous dope solution. The dope solution was then printed on a glass plate and then put into a coagulation bath containing ethanol from a concentration of 0 - 90 %. During the coagulation process, solidification occurs to form a solid matrix, namely the membrane.

Effect of Ethanol Concentration on Density

The effect of ethanol concentration on the density of each polysulfone membrane is shown in Figure 1. The graph of polysulfone membrane density on ethanol concentration (Figure 1) shows the maximum value for the density of polysulfone membranes with 90 % ethanol concentration, which is 0.5019 g/cm^3, while at 0 ethanol concentration % indicates the lowest density value, namely 0.3497 g/cm^3. The trend of the membrane density graph shows that the increase in density value is directly proportional to the increase in ethanol concentration. This happens because when the membrane film is inserted into the coagulation bath a solidification process occurs, the membrane film which is still in liquid form moves to fill the empty pores or bundles due to DMAc diffusion into ethanol resulting in tighter pores compared to the 0 % ethanol coagulation bath (water).

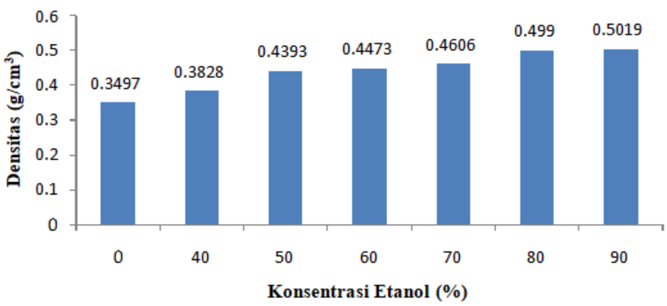

Figure 1. Effect of Ethanol Concentration on Density

Effect of Ethanol Concentration on Degree of Swelling

When compared with the density chart, the opposite trend occurs in the swelling degree change graph due to the influence of ethanol concentration. Figure 2 shows that the maximum value occurs in the membrane with 50 % ethanol concentration, which is 28.662. This is probably due to the large pore size and the higher hydrophilicity of the membrane resulting in more water being adsorbed into the membrane, while the ethanol concentration of 60 % to 90 % tends to be lower.

The concentration of ethanol affects the degree of swelling of the membrane because when the membrane film is put into the coagulant bath, the solvent will leave the polymer solution so that the polymer solution moves to fill the empty space used by the solvent more and more so that the resulting pores become tighter. This resulted in a decrease in the ability of the PSf membrane to absorb water.

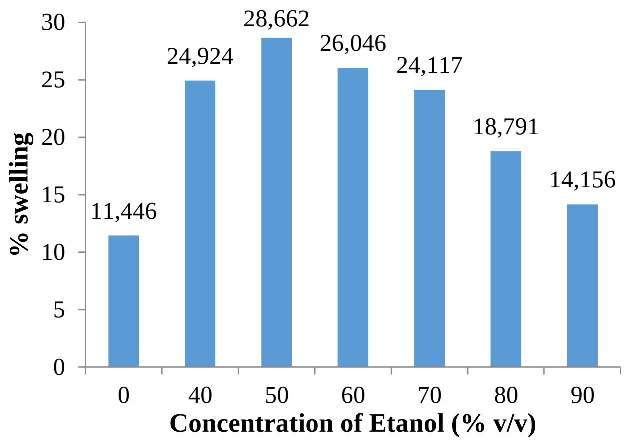

Figure 2. Effect of etanol concentration on derajat swelling

Performance of Polysulfone Membrane

Compaction of Polysulfone Membrane

Before testing the flux of a solution, compaction is first carried out on the membrane to be tested. This test is to obtain a constant water flux value at a given operational pressure of 2 bar. The results shown in Figure 3 show that there is a decrease in flux until the time the flux value is constant. The decrease in water flux occurs due to mechanical deformation of the membrane matrix due to the applied pressure.

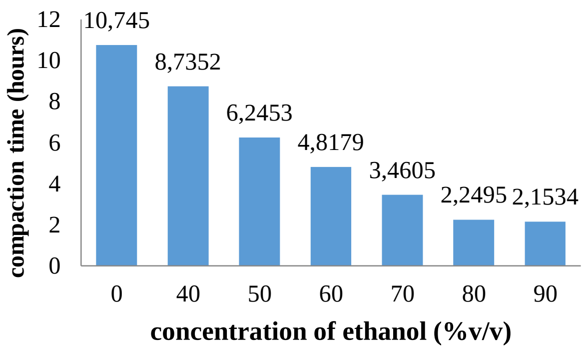

Figure 3. Graph of the effect of ethanol concentration on the compaction time of polysulfone membranes

Effect of Ethanol Concentration on Water Flux

Water flux or permeation rate is one of the parameters that determine membrane performance. The test results show that the water flux curve at a pressure of 2 bar tends to increase (figure 4). Where the highest flux value is at 90 % ethanol with a flux value of 1.3037 L/m^2 hours and the lowest at 0 % ethanol is 0.2093 L/m^2 hours. The higher the ethanol concentration, the higher the resulting flux value. Because with increasing ethanol concentration the pore size of the membrane gets smaller, this can be seen from the effect of the resulting membrane density value. As well as with the increasing concentration of ethanol the hydrophilicity of the membrane is getting higher, this can be seen from the increasing amount of feed solution (water) that is passed through the membrane.

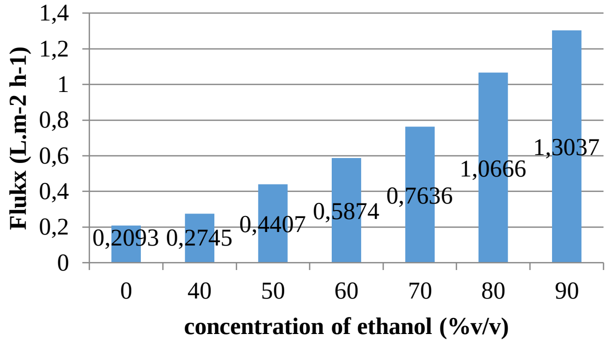

Figure 4. Effect etanol concentration on flux

Meanwhile, Radiman (2002) found that the immersion medium using 80 % ethanol had a water flux value of 645.69 L/m^2 hour. There are differences in the flux values obtained due to differences in the composition of the polysulfone membrane constituents.

Effect of Ethanol Concentration on Permeability Coefficient

Variations in ethanol concentration were also studied for their effect on membrane permeability. Permeability is often expressed as the membrane permeability coefficient. The permeability of the membrane can be obtained by measuring the value of the water flux with respect to pressure variations.

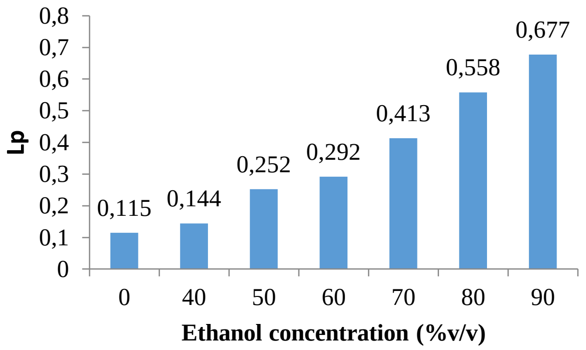

Figure 5. Effect of Ethanol Concentration on Permeability Coefficient (Lp)

Figure 5 shows the flux values on various types of membranes with variations in ethanol concentration with variations in pressure. Based on these results it can be seen that the lowest membrane permeability coefficient was polysulfone membrane with 0 % ethanol concentration, namely 0.115 and the highest with 90 % ethanol concentration, namely 0.667. This indicates that membrane permeability is related to the size and number of pores in a membrane. The bigger and less porous the membrane, the flux value will decrease. The lower flux, the permeability coefficient will also decrease. Vice versa, the smaller and more porous the membrane, the flux value will increase and the permeability coefficient will also be higher. This phenomenon is caused by the more pores formed with a larger size in the membrane, the faster the feed flow can pass through the membrane.

The Effect of Ethanol Concentration on the Rejection Coefficient of Polysulfone Membrane

The rejection coefficient is a measure of membrane selectivity. Based on the rejection coefficient results in Figure 6, it can be seen that dextran with a molecular weight of 35 - 45 kDa has a smaller rejection value than dextran 100 - 200 kDa. This is because the molecular weight of 35 - 45 kDa dextran is lower than that of 100 - 200 kDa dextran, so that the membrane rejection of 100 - 200 kDa dextran is greater than that of 35 - 45 kDa dextran.

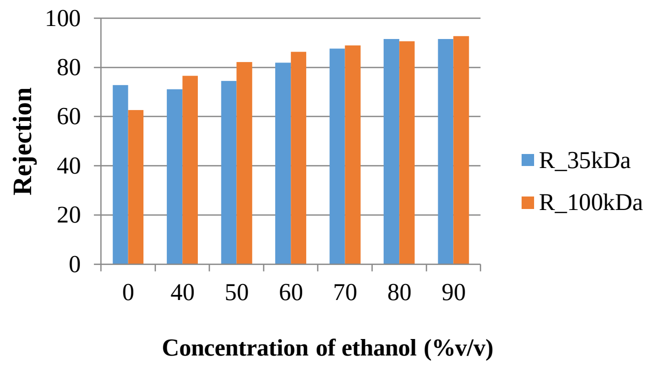

Figure 6. The Effect of Ethanol Concentration on the Rejection Coefficient of Polysulfone Membrane

Figure 6 shows that the polysulfone membrane with 90 % ethanol concentration has the highest rejection coefficient value, while the polysulfone membrane with 0 % ethanol concentration has the lowest rejection coefficient. Where more high concentration ethanol, then the rejection coefficient as well tend to be more increase. This phenomenon occurs because with increasing concentration of ethanol, so that the membrane pores that are formed are tighter and more molecules are retained by the membrane.

In this study polysulfone membranes are included in the classification ultrafiltration membrane contained in the polysulfone membrane with ethanol concentration 80 % v/v and 90 % v/v with a rejection value above 90 %. This is because the polysulfone membrane with ethanol concentration of 80 % v/v has a rejection value of 91.58 % for dextran with a molecular weight of 35 - 45 kDa and 90.67 % for dextran with a molecular weight of 100 - 200 kDa. Whereas the polysulfone membrane with an ethanol concentration of 90 % v/v had a rejection value of 91.49 % for dextran with a molecular weight of 35 - 45kDa and 92.67 % for dextran with a molecular weight of 100 - 200 kDa.

CONCLUSION

Increasing the concentration of ethanol from 0 % to 90 % causes an increase in the density value of polysulfone membranes. Increasing the concentration from 0 % - 50 % causes an increase in the degree of swelling, but decreases the degree of swelling from a concentration of 60 % - 90 % to water. Increasing the concentration of ethanol from 0 % up to 90 % increasing the water flux and permeability coefficient of polysulfone membranes as well as increasing the rejection value.

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