Characteristic and Performance Tests of Membrane PES in Biodiesel Purification by using Ultrafiltration Process

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Abstract

Polyethersulfone (PES) membranes were prepared via phase inversion method. The effect of polymer concentration on the morphology of the fabricated membranes were studied. Furthermore, the effect of trans-membrane pressure (TMP)was also studied on the filtration performance of biodiesel purification. The morphology of fabricated PES membrane was analyzed by using Scanning Electron Microscopy (SEM) indicated that the PES membranes had skin layer on the membrane surface and pores layer on the bottom surface. The permeability coefficient (Lp) of PES membranes were about 21-40 L/m^2.h. The Molecular Weight Cut Off (MWCO) test shows that the rejection of dextran solution with the molecular weight 18,8Kda was above 90%. The water contact angle of PES membranes was measured to know the hydrophilicity of PES membrane. The water contact angle was about 73^{o}confirmed that the PES membrane was hydrophobic. The increasing ofTrans-membrane Pressure (TMP) had a significant effect on the permeability of membrane which isthe permeability of biodiesel increases by increasing the TMP. The maximum of flux was obtained about 110 ml/sec.

Key words: Polyethersulfone (PES) membrane, Trans-membrane pressure (TMP), ultrafiltration, permeability

Introduction

Finding the alternative energy is a crucial thing to the world recently. Due to the need of fuel energy increases along with the rate of population growth. The increasing global price of crude oil is also attributed to this phenomenon. Biodiesel is one of the alternative resolved for the problem. Biodiesel is generated by reacting fatty acid or oil with an alcohol to produce methyl ester (crude biodiesel) and glycerol (a byproduct). Obtained results from this transesterification still require further purification. A series of processes including wet washing, dry washing and membrane technology has been applied in refinement of biodiesel. Wet washing generates a fairly high purity of biodiesel but the process forms waste as an emulsion of oil and water [Canakci, 2001, Berrios., et al., 2008, and Alves., et al, 2013]. It is reported that every one liter of biodiesel will generate one liter of wastewater [Karaosmanoğlu., et al., 1996]. Dry washing process also produces a high quality biodiesel. However, there are a number of disadvantages such as difficulties in obtaining information on resins used, and it could possibly cause contamination with biodiesel [Bertram., et.al, 2005, Berrios., et al., 2005, Berrios., et al., 2008, and Faccini, et al., 2011]. Membrane technology noted to be a promising alternative purification method. Some advantages of this process are it is so much simpler, it does not involve chemicals and it produces zero waste.

Research on biodiesel purification using membrane technology has been exploiting reactor membrane and ceramic membrane as the medias [Gomes., et al., 2010 and Atadashi., et al., 2011]. In this work, polyethersulfone (PES) membrane was prepared via phase inversion technique with different of polymer concentration. The morphology and hydrophilicity property of fabricated membrane observed by Scanning Electron Microscopy (SEM) and water contact angle meter, respectively. In addition, water permeability and molecular weight cut off (MWCO) tests were carried out to investigate the rejection ability of membrane against dextran solution in various molecular weights of 19500 Da, 39000 Da, and 188000 Da. The ultrafiltration performance of fabricated membrane was analyze by measure the permeability of biodiesel solution.

Experimental

Materials

Polyethersulfone (PES) polymer was used as main material in membrane preparation. N-Metil Pyrrolidone (NMP) is as the solvent. Palm oil and ethanol are as biodiesel base materials, and NaOH as a catalyst. Dextran with molecular weight of 19500 Da, 39000 Da, and 188000 Da were used as particle model for solute rejection investigation to determine Molecular Weight Cutoff (MWCO). Other supporting materials used were N-hexane, acetone and deionized wáter

Membrane Preparation and Characterization

Polymer solution was prepare by dissolving of PES in NMP. The concentration of PES were variate as 10, 12.5, 15 and 17.5 wt%. The homogenous polymer solutions were obtained by stirring for 24 hours. This resulted transparent solution was called dope. Debubling was performed in order to clean the bubbles off dope which formed during stirring. The dope then rested in refrigerator for 24 hours. Following that, the homogenous and bubbles-free dope was taken out and left until it corresponded to room temperature. The dope then casted onto glass plates and flattened to the entire surface of the glass by using aplicator. A thin layer formed was left at room temperature for 15 seconds to allow the realease of the solvent. The casting membranes and the glass was then immersed into coagulation bath which contained water as precipitant media (prepicitation process). The process was carried on until the membrane layer detached from the glass plate. During precipitation process, the temperature of coagulation bath should be maintained constant at 27^0C. The membrane was washed with deionnized water and then kept in the water for testing. The morphology of the membrane was observed by Scanning Electron Microscopy (SEM). The hydrophilicity property was analyzed using water contact angle meter.

Water Permeability Test

Prior to being appliedon the process, wáter permeability of a membrane firstly needed to be determined by means of passing pure wáter through the membrane in concord ant to its operating pressure. The resulted permeability coefficient specifies whether the fabricated membrane belongs to the range of ultrafiltration group or not. Membrane was installed on the ultrafiltration module, the module then was assembled to pressurize gas tube with pressure gauge to determine how big the pressure of feed is that it will go through the membrane. For initial characterization, pure water was fed onto the membrane. The test started off by flowing air through compressed gas of 1 bar as a driving force. The permeate that passed through the membrane then collected in erlenmeyer flask and its volume was measured until constant. Following that, the flux was calculated for the 1 bar initial trial. The same treatment was repeated for other samples of different pressures.

Molecular Weight Cut Off

Membrane was installed onto the membrane module, absorbance of dextran was measured before it was flowed into the membrane. 10ml of dextran with concentration of 0.5 gr/l and molecular weight variations of 195000 Da, 39000 Da and 188000 Da was fed into the feed tank. The feed was stirred at certain speed and the compressed gas was streamed into the tank until it reached the desired pressure and it was left for some time to allow the outcome of permeate. The permeate was then accommodated in a graduated cylinder. Data collecting was discontinued after the appropriate volumetric rate of permeate retrieved. Absorbance of dextran which passed through the membrane was also gauged. The same procedure was conducted for other samples of different pressure.

Permeability Test On Glycerol Removal from Biodiesel

Production of Biodiesel

A total of 500 grams of palm oil incorporated into the three-neck flask equipped condenser. The oil was heated up to 65ºC, and then 1.5 grams of methanol was slowly added in. The solution was heated for an hour. After the transesterification reaction, the glycerol was separated from biodiesel by decantation.

Biodiesel Purification (Glycerol removal from crude biodiesel)

Purification of biodiesel in this study was focused on separating glycerol from biodiesel. A total of 500 ml of biodiesel was streamed into membrane unit using compressed gas at operating pressure of 1, 1.25, 1.5, and 1.75 bar. The permeate that passed through the membrane was accumulated in erlenmeyer flask and its volume was measured until constant. The flux for each variation of pressure was then calculated. The same procedure was applied on other membranes of different concentrations.

Results and Discussion

Water Permeability Coefficient

Water permeability is the amount of water passed through the membrane based on the membrane surface area and the applied pressure in period of filtration time. Permeability coefficient (Lp) is one of the parameter of membrane characteristic. Permeability coefficient shows the ease of water (distilled water) flow through membrane. Lp value was obtained from slope of flux to the operational pressure (Mulder, 1996). Figure 1 show the water permeability coefficient of membranes with different concentration of polymer. From Figure 1, the water permeability coefficient were in the range of 21.05 to 40.34 L/m^2.h.bar. According to Mulder (1996), ultrafiltration membrane has permeability coefficient of 10-50 L/m^2.h.bar. Based on the results, it can be seenthat the fabricated polyethersulfone membrane in various concentrations decrease under ultrafiltration category.

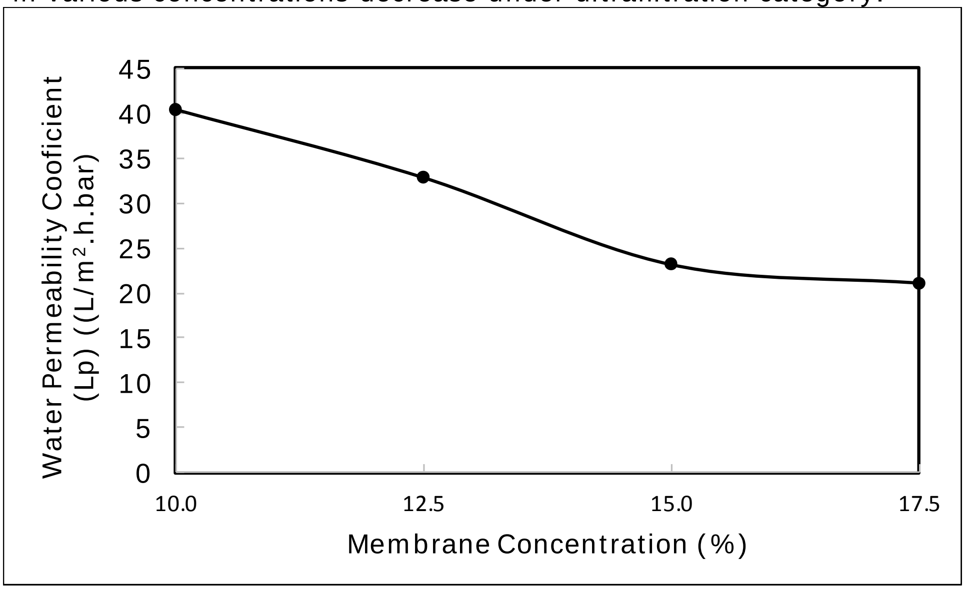

Figure 1. Water Permeability Coefficient as function of Membrane Concentration

Molecular Weight Cut Off (MWCO)

Molecular Weight Cut Off (MWCO) is one of membrane's characteristics. MWCO is defined as the molecular weight value of solutes that can be retained by the membrane with a rejection more than 90% (Radiman and Eka, 2007). Molecular Weight Cut Off (MWCO) was investigated by using dextran solution with molecular weight of 19500 Da, 39000 Da, and 188000 Da. MWCO was obtained by plotting molecular weights of dextran over rejection of PES membrane as displayed in Figure 2. The figure portrays that the bigger the molecular weight of dextran, the higher the rejection achieved for membrane of all various concentrations. The similar tendency can also be seen on the increase of concentration of the polymer membranes. The picture reveals that polymer with concentration of 15 and 17.5% both exhibit rejection above 90%.

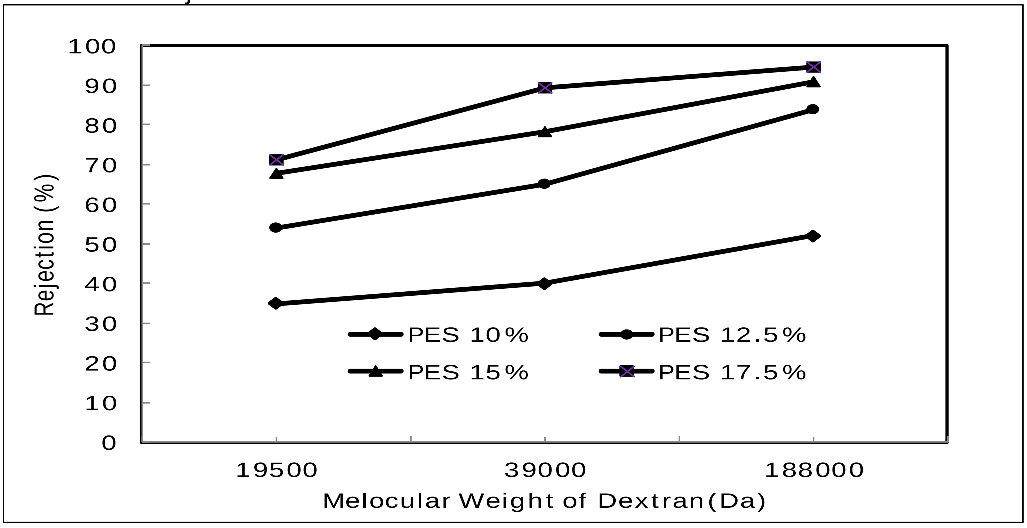

Figure 2. Rejection of Membrane as Function of Molecular Weight of Dextran

Membrane Morfology

The results of membrane morphology structures were displayed in Figure 3. It is seen that the surface of fabricated PES membrane had porous structures. In the membrane separation process, the surface layer of a membrane plays a role as a media separator. Selectivity or rejection established higher on the membrane with tighter surface porous, in the same circumstance, its flux and permeability also found greater. Cross-section image of SEM shows that the prepared membrane was an asymmetric, composed of two layers. The top dense layer functions as a separator medium meanwhile the bottom porous layer serves as the support of the membrane.

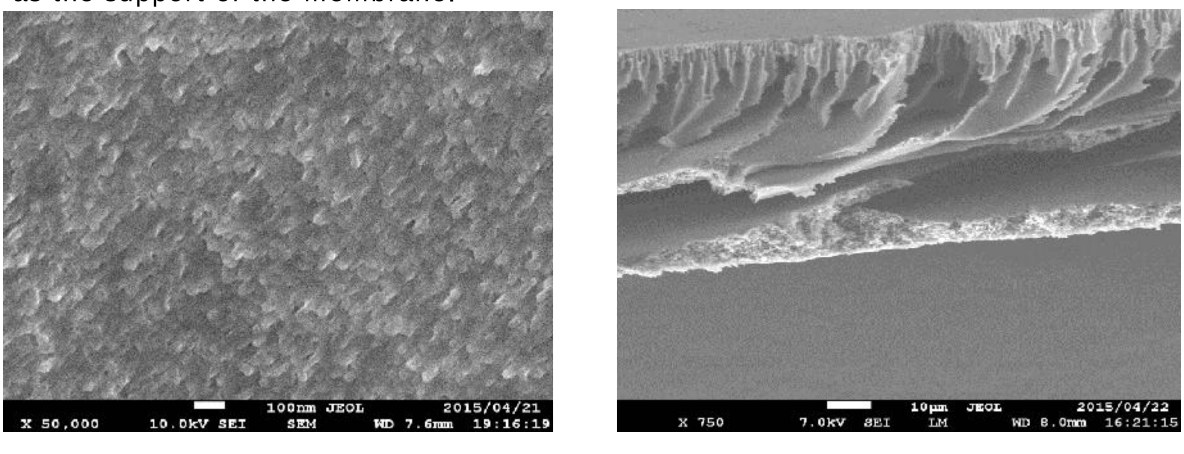

Figure 3. SEM images of PES 15 % membrane. Left : Membrane Surface, Right: Cross-section

Membrane Hydrophilicity

PES membranes have excellent resistance to chemicals, applicable at a wide range of temperature and has an appreciable mechanical strength. This polymer is widely used in membrane preparation for various applications (Nasrul et al., 2012). Water contact angle measurement was performed to investigate the hydrophilicity of a membrane by observing the angle formed when the water dropped on outer surface of the membrane. The bigger the angle formed the higher the hydrophobic property of the membrane. Water contact angle obtained in the experiment was in the range of 73-78^o.

Membrane Performance

The flux of PES membranes as function of Trans Membrane Pressure (TMP) in biodiesel purification is displayed in Figure 4. It is seen that the higher the given temperature the greater the flux attained. The separation process was occured with the help of driving force in the form of the pressure difference in the inlet and outlet of membrane module. This suggests that the increase in pressure could boost the flow rate of flux because this pressure strengthens the driving force of water to cross over the membrane thus the effect of concentration polarization can be minimized. From thisfigure, it also informs that on the occasion of polymer concentration increased, the flux will be generated lower. The higher the concentration of polymer, the thicker the dense layer of the membrane will be, hence the permeate that pass throughthen it will be on the wane. The highest flux achieved was at the pressure of 1.75 bar and polymer concentration of 10%wt which amounted to 140 ml/sec.m^2. Meanwhile on the experiment using membrane with PES concentration of 15%wt, obtained biodiesel flux was 75 ml/sec.m^2.

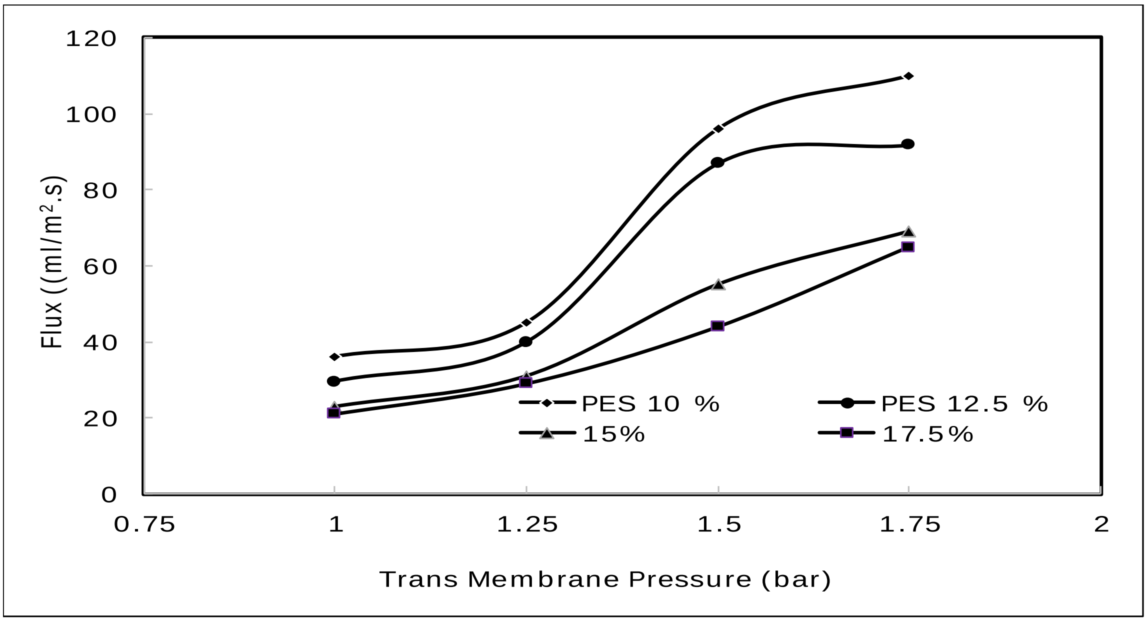

Figure 4. The flux of PES membranes as function of Trans Membrane Pressure (TMP) in biodiesel purification

Conclusions

From the results of the study, conclusions are drawn as follows:

1. Permeability coefficient (Lp) of polyethersulfone membrane of all concentrations was obtained in the range of 20-40 ml/m^2.sec.bar which represented the membrane to be in the ultrafiltration category.
2. On the membrane with PES concentration of 15 and 17.5%w tthe rejection of dextran solution with the molecular weight 18.8 Kda was above 90%.

3. The higher the Trans Membrane Pressure (TMP), the greater the flux attained. The highest amount of flux retrieved was 110 ml/sec.m^2 on the membrane with PES concentration of 10 %wt.

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