Effect of Dimethyl Sulfoxide (DMSO) as a Green Solvent and the Addition of Polyethylene Glycol (PEG) in Cellulose Acetate/Polybutylene Succinate (CA/PBS) Membrane’s Performance

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Abstract. In this study, performance of CA/PBS membranes, prepared with the non-toxic solvent dimethyl sulfoxide (DMSO) compared to Dimethylformamide (DMF), was investigated. The membranes were prepared using immersion precipitation technique at coagulation bath at temperatures of 25°C. Ternary diagram was used to show the interaction between polymer (CA/PBS), solvents (DMF or DMSO) and isopropanol as non-solvent by measuring the cloud point instead of Hansen solubility parameter differences. The effect of Polyethylene Glycol (PEG) to the membrane performance was also investigated. Morphology and tensile strength were analyzed by using Scanning Electron Microscopy (SEM) and Dynamic Mechanical Analysis (DMA), respectively. Experimental results proved that DMSO could be used to replace (DMF) as solvent, as the membranes had a higher value of salt rejection (84.9%). With the addition of PEG, salt rejection, mechanical property, and porosity of CA/PBS membrane also increases. The best result shows that PEG with concentration of 7% wt of polymer, the salt rejection is 88.89%; Permeate Flux is 29.036 L/m².hr; tensile strength is 68.366 kPa; porosity is 75.16 % and the morphology is sponge-like with the pore size diameter in average is 0.262 µm

1. Introduction
The phase inversion is the most widely used method in the fabrication of membranes resulting asymmetrical membrane, both flat sheet or hollow fiber membrane types [1]–[3]. The phase inversion method involves transformation from a homogeneous polymer membrane solution to a final membrane structure (solid) through a phase separation of polymer solution [4],[5]. In the phase inversion method, there are two basic techniques, dry technique and wet technique. In the dry technique the solvent is evaporated from the membrane solution into the atmosphere and or absorbs the vapor phase (non-solvent) from humidity. However, the membrane structure is formed by immersing the solution membrane into a coagulation bath containing non-solvents in the wet technique. Both of these methods are known as the immersion precipitation method where it has been widely used in the manufacture to fabricate membranes commercially [6]. The solidification process is very frequently started by the change from one liquid phase into two liquid phases (liquid- liquid demixing). At certain period during demixing, the polymer rich phase solidifies; thus, a solid membrane matrix is formed. Membrane morphologies, the pore size distributions can be controlled by selecting the different solvent, non-solvent, polymer, pore former, and fabrication parameters depending on the particular application [5]–[9]. Liquid-liquid demixing (L-L demixing) is strongly influenced by the thermodynamic and kinetic
properties of the membrane system. The formation of membrane structure related to the phase equilibrium which is in the thermodynamic property can be described by Flory-Huggins theory and in the kinetics of transport process can be described by the mutual diffusion and transport between components. A ternary diagram is commonly used as a tool to analyze the thermodynamic and kinetic behavior of the membrane system.

Petroleum-based non-biodegradable polymers are widely used in industry due to cheaper and affordable production costs, but the materials are recalcitrant to these microorganisms and result in the formation of solid waste which is a problem [10]. Alternative to reduce sources of environmental pollution is to select and replace membrane-forming materials with materials that have a high biodegradation rate and are more environmentally friendly [11]. Cellulose Acetate is an economically valuable polymer of particular concern in the field of green and sustainable chemistry and stimulates the development of environmentally friendly technologies. Cellulose Acetate is a polymer which has a high biodegradation rate, is low cost, is non-toxic and is a renewable source [12], but he first generation cellulose acetate (CA) membranes yield low flux and are susceptible to chemical and bacteriological agents. CA performance can be improved by mixing it with appropriate additives to meet new requirements and related membrane properties [4]. Among the different polymeric materials that are used for the membrane preparation Poly Butylene Succinate is high biodegradable, such as softness, tensile and gas barrier properties, melt viscosity for further processing [13]. However, the solvents used for membrane preparation are largely toxic to human health and the environment, typical examples are chloroform, dichloromethane, N-dimethylformamide (DMF), dichloromethane and dimethylacetamide (DMAc) with more environmentally friendly solvents for the manufacture of polymeric membranes. DMSO, which can be extracted from lignin or synthesized by oxidation of dimethyl sulfide, is an aprotic organic solvent for membrane synthesis. DMSO not only has relatively low intrinsic toxicity, but is also biodegradable, forming a non-toxic product. Therefore, DMSO is considered a green solvent for the manufacture of CA/PBS membranes [14], [15].

Therefore, many researchers wished to further modify it to obtain a high-performance membrane. In this study, Polyethylene Glycol (PEG) was added to the CA/PBS membrane. PEG, commonly used as additive in membrane manufacture, because it is known as pore former and can act as plasticizer [16]. It is expected that the pore size of membrane decreases, and its mechanical property increases. Thus, the effect of using DMSO as a green solvent and the addition of PEG to the performance of CA/PBS membrane was investigated.

2. Experimental

2.1. Materials
Cellulose acetate (CA, Mn = 30.000 Da, density = 1.3 g/mL) and poly(1,4-butylene succinate) (PBS, density = 1.3 g/mL at 25°C) were purchased from Sigma Aldrich. Polyethylene Glycol (PEG, 400 Da) was purchased from Merck and used as additive. Dimethylformamide (DMF) and Dimethyl Sulfoxide (DMSO) were purchased from Merck and used as solvent. Isopropanol with a purity of 96 wt% were purchased from Merck and used as non-solvents. NaCl were purchased from Merck and used to make saline solutions for desalination tests.

2.2. Membrane Preparation
CA/PBS membranes with a composition of 80/20 with total polymer 2.5 g were prepared by phase inversion method. CA/PBS were dissolved in DMF or DMSO with volume of 17 mL. PEG was added by dissolving it into CA-PBS solution with composition: 0; 2; 7 and 8 wt% of total polymer until the solution is homogeneous. The homogeneous solution was cast on a glass plate by a film applicator then immersed into coagulation bath containing non-solvents for 15 minutes at room temperature. Then, CA/PBS membranes were dried and then before analyzed, defects in the membrane are visually observed put the membrane under the light. To make sure that the membrane has no defects, all the membranes are checked using this visual observation before further use.
2.3. **Phase Diagram**  
The phase diagram of the CA-PBS/Solvent/Isopropanol ternary systems was determined by cloud point measurement. Ternary phase diagram was obtained from the turbid points. To reach the turbid point, the non-solvent was added slowly into the polymer solution under stirring. During titration, the solution temperature was controlled at 25°C with a water bath and the addition of isopropanol was continued until the clear polymer solution visually turned to look milky-like, the observation of the first sign of turbidity, the addition of non-solvent was stopped and the solution was stirred for an additional 20-40 min to see whether the turbid solution changes to a clear solution or not. If the solution turned to a clear solution, more non-solvent was added; otherwise, the determined point was considered as the onset of real turbid point. The ternary composition of the turbid point was then calculated from the amount of non-solvent, solvent, and polymer present in the glassware.

2.4. **Hansen Solubility Parameter**  
The affinity between the solvent, polymer and nonsolvent can be predicted by solubility parameter ($\delta$), which is described as the square root of the cohesive energy density and express the strength of attractive forces among the molecules. Solvent - polymer; solvent – non-solvent; and polymer - non-solvent interaction in a polymer solution has been estimated based on the difference between polymer, solvent and nonsolvent as already clearly mentioned by Hansen Solubility [17]. According to Hansen, the permanent dipole-dipole interaction ($\delta_p$), dispersive ($\delta_d$), hydrogen bonding forces ($\delta_h$) should be taken into consideration. Consequently, the solubility parameters can be calculated by using Equation 1 [18].

$$\delta^2 = (\delta_d^2 + \delta_h^2 + \delta_p^2)$$  

(1)

The Hansen Solubility parameter differences ($\Delta$) between solvents and polymers was calculated by using Equation 2 [18].

$$\Delta = \sqrt{[(\delta_{p,d} - \delta_{s,d})^2 + (\delta_{p,p} - \delta_{s,p})^2 + (\delta_{p,h} - \delta_{s,h})^2]}$$  

(2)

where, S and P denotes as solvent and polymer, respectively. $p$, $d$ and $h$ represent for polar, dispersive, and hydrogen bonding elements of Hansen Solubility Parameter, respectively.

2.5. **Membrane Characterization**  
2.5.1. **Morphology of Membrane.** Morphology of the membranes were observed by using scanning electron microscopy (SEM) (Hitachi, FlexSEM 1000, Japan) with 5 kV. To observe cross sectional area of the membrane, it must be broken during dipping in liquid nitrogen; then, sputtered with gold. The thickness of the membranes was measured using SEM Photo and Image J. The membrane porosity $\varepsilon$ was determined by gravimetric method [13], [19],[20]. The membranes were immersed in water for 24 hours, and the wet membranes are dried in an oven (Oven Memmered UN55, Germany) at 70°C for 48 h, the weights of wet and dry membrane using analytical balance (Ohaus PA214, USA). The membrane porosity $\varepsilon$ was determined using the Equation (3).

$$\varepsilon = \frac{(w_1 - w_2)/d_w}{w_1 - w_2}$$  

(3)

Where $w_1$ is the weight of the wet membranes (g), $w_2$ is the weight of the dry membranes (g), $d_w$ is the density of water (0.998 g/cm$^3$) and $d_p$ is the density of the polymer (1.3 g/cm$^3$). The pore membrane was measured using by combining the threshold in the Image J application assisted by Photoshop and by using Guerout–Elford–Ferry equation based on the data of porosity and permeation rate of pure water [19]–[22].
\[ r_m = \sqrt{\frac{(2.901.75e) \times 8 \eta Q}{\varepsilon A \Delta P}} \]  

(4)

Where \( \eta \) is the viscosity of water \((8.9 \times 10^{-4} \text{Pa.s})\), \( l \) is the thickness of membrane \((\text{m})\), \( A \) is the effective of membrane area \((\text{m}^2)\), \( Q \) is the permeation rate if water \((\text{m}^3/\text{s})\) and \( \Delta P \) is the pressure membrane \((\text{Pa})\).

2.5.2. Hydrophilicity of Membrane. Water content analysis is carried out to determine the ability of the membrane to absorb water. It indicates hydrophilicity of membrane. Water content analysis is done by calculating the difference between the wet mass and the dry mass. The wet mass is the mass of the membrane after immersion into water; while, the dry mass is the mass of the membrane that has been oven-dried up to a constant weight. Water content \((WC)\) analysis was calculated using Equation 5 [23] where \( W_w \) is wet sample \((\text{g})\) and \( W_d \) is dry sample \((\text{g})\).

\[
WC\% = \frac{W_w - W_d}{W_w} \times 100\%
\]  

(5)

2.5.3. Mechanical Property. Tensile strength was analysed using Dynamic Mechanical Analysis (DMA/SDTA 861°, Mettler Toledo, Singapore). All tests were done according to ASTM D5026 standard.

2.5.4. Membrane Performance. Membrane performance was analysed in terms of salt rejection with NaCl for make saline solutions for desalination tests and permeate flux in cross flow system using Sterlitech CF042A membrane cell with an effective membrane area of 42 cm². The flat sheet membrane with a size of 13 x 15.5 cm was used to analyze membrane’s performance by using synthetic salt water with concentration of NaCl is 1000 ppm. Salt rejection \((R)\) and Permeate flux was calculated by using Equation 6 and 7, respectively.

\[
\%R = \left( \frac{C_f - C_p}{C_f} \right) \times 100\%
\]  

(6)

where \( R \) is the salt rejection \((\%)\), and \( C_f \) and \( C_p \) are the concentration of the feed and permeate \((\text{ppm})\), respectively [24].

\[
Permeate Flux = \frac{Q}{A \Delta t}
\]  

(7)

where \( Q \) is the quantity of permeate \((\text{L})\), \( A \) is the effective membrane area \((\text{m}^2)\) and \( \Delta t \) is the sampling time \((\text{h})\) [25].

3. Result and Discussion

3.1 Effect of Dimethyl Sulfoxide as a Green Solvent

3.1.1. Phase Diagram CA-PBS/ Solvent/ Isopropanol. Phase diagrams are useful for predicting the phase transitions occurred when the phase separation is induced according to the immersion precipitation method. The membrane formation mechanism is based on the demixing process. This process occurs when the addition of non-solvents to the compaction process so that the polymer solution becomes thermodynamically unstable, the demixing process is one sign that the binodal process in the solution has been achieved [9], [15], [26], [27].
Figure 1. Ternary Diagram CA-PBS/Solvent/Isopropanol Membrane.

In Figure 1, the binodal curve with DMSO and DMF solvents shows that the DMSO solvent has a curve closer to polymer (CA-PBS) and isopropanol as non-solvent which means that liquid-liquid and solid-liquid demixing processes will occur compared to DMF. It means the polymer will diffuse more stably and form a membrane matrix. Thus, the membrane made using DMSO solvent resulting more porous membrane because the non-solvent - solvent exchange rate will be higher [15].

3.1.2. Solubility Parameter CA-PBS/ Solvent/ Isopropanol. The polymer-solvent-nonsolvent concentration parameter is inversely proportional with Hansen solubility parameters (Δ)[18]. The solubility parameter difference between polymer- solvent (Δp,s) and solvent – non-solvent (Δs,ns) shown in the Table 1. The results show that Δp,s for DMSO is smaller than that for DMF. It means that interaction between CA/PBS-DMSO is higher than that of CA/PBS-DMF. However, the Δs,ns value show that the interaction between DMF – isopropanol is higher than that of DMSO – isopropanol. It indicates that DMF as a solvent can accelerate the process of exchange rate from solvent to non-solvent which causes instantaneous demixing processes. Because the process is very fast, resulting in the formation of large pore size, large cavities and macrovoid on the membrane. However, with DMSO as a solvent, the exchange process from solvent to non-solvent is slower which causes a liquid-liquid demixing process followed by solid-liquid demixing. Because of this, CA/PBS with DMSO membrane resulting in smaller pores and can remove macrovoid in the pores that is in agreement with the previous study [7], [11].

Table 1. Hansen solubility parameters for CA/PBS membrane with variation of solvents.

| Component                  | Δp,s   | Δs,ns   |
|----------------------------|--------|---------|
| CA/PBS-DMF-Isopropanol     | 6.4765 | 9.2914  |
| CA/PBS-DMSO-Isopropanol    | 5.7361 | 12.3000 |
3.1.3. Morphology of Membrane CA-PBS/ Solvent/ Isopropanol

SEM analysis was carried out with magnification of 5000x and 10,000x to show the morphological of the membrane in the top surface and cross-section area, respectively in Figure 2. Morphology of membrane CA/PBS either with DMF or DMSO as solvents exhibit dense membrane with sponge like-structure. Top surface in the both membranes shows rough surface; however, the top surface of membrane with DMSO as solvent is denser than that of membrane with DMF as solvent. Porosity of CA/PEG membrane with DMF and DMSO as solvent is 58.258% and 58.843%, respectively. These porosity values are almost the same, because the pore size of both of them is also similar, about 0.3 μm.

3.1.4. Hydrophilicity of Membrane CA-PBS/ Solvent/ Isopropanol. Hydrophilicity plays an important role in liquid filtration [15]. The membrane hydrophilicity was determined by a water content measurement. Table 2 shows the measured water content of CA/PBS membrane with variation of solvent. The result shows that hydrophilicity of CA/PBS membrane with DMSO as a solvent is higher than that of CA/PBS membrane with DMF as a solvent.

| Component               | Water Content (%) |
|-------------------------|-------------------|
| CA/PBS-DMF-Isopropanol  | 51.724            |
| CA/PBS-DMSO-Isopropanol | 52.326            |
3.1.5. Mechanical Property of Membrane CA/PBS Membrane. Figure 3 shows the mechanical property of CA/PBS membranes with variation of solvent. In general, the tensile strength of membrane with DMSO as solvent is higher compared to membrane with DMF as a solvent. Membranes with DMSO solvent variations have a relatively small pore size compared to the DMF solvent (0.331 vs 0.351 \( \mu \text{m} \)), resulting in an increased of mechanical strength of membrane with DMSO as a solvent [15].

![Figure 3. Tensile Strength Results CA-PBS/ Solvent/ Isopropanol Membrane.](image)

3.1.6. Hydrophilicity of Membrane CA-PBS/ Solvent/ Isopropanol. Based on Figure 4, the membrane whose DMSO solvent has increased the value of salt rejection, this means that the DMSO solvent is proven to be in accordance with the prediction of phase separation analysis where by using the DMSO solvent the resulting line is closer to the polymer-non-solvent line which results in a liquid-liquid demixing process, resulting in a more uniform pore and more able to hold the solute that passes through the membrane and is a solvent that is more environmentally friendly can also increase the performance of the membrane [14], [15], [28].

![Figure 4. Performance Test for Variation Solvent in CA/PBS Membrane.](image)

From the membrane performance as shown in Figure 4, it can be concluded that DMSO is better than DMF. Salt rejection of CA/PBS membrane with DMSO as a solvent is 78.33 % and permeate flux is 35.19 L/m\(^2\).hr.

3.2 Effect of Additive Polyethylene Glycol in CA-PBS/DMSO/Isopropanol Membrane

3.2.1 Morphology of Membrane CA-PBS/ DMSO/ Isopropanol with additive Polyethylene Glycol. SEM analysis was carried out with magnification of 650x, 5000x and 10,000x to show the morphological structure of the membrane in the top surface and cross-section area. From the results of the SEM analysis, the morphological parameters observed were pore distribution, pore type, pore diameter and
porosity. Pore distribution in the additive PEG can be seen in Figure 5, shows asymmetrical tendencies where the membrane has sponge-like pores. When the addition of PEG is more, the smaller the pore size occurs. It is because PEG is a pore-forming and acts as a plasticizer agent that makes the membrane stronger [2], [29], [30]. However, when the addition of PEG is 8% wt of polymer resulted in a more dense, symmetrical membrane and a larger pore size.

Figure 5. SEM morphology for CA/PBS membrane with the addition of PEG by using DMSO as solvent.

Table 3 shows pore size and porosity of CA/PBS membrane with the addition of PEG. It can be seen that with the addition of PEG at 7 wt% resulting the smallest pore size diameter in the fractured surface. This result is an agreement with the value of the porosity. It exhibits the highest value in the porosity. The mass transport in Reverse Osmosis (RO) occurs due to the solution-diffusion phenomena, not by the molecular sieving. One of the characteristics of RO is dense structure [31]. The resulting pore has a dense pore structure, it's just that the pore size is still relatively large, this is what causes the use of the sample in the rejection salt test using a salt concentration only 1000 ppm, when the concentration is increased to above 1000 ppm the results obtained are not maximal. Therefore, the membrane in this research work is still limited to a salt concentration of 1000 ppm.

| PEG Content % wt | Pore size (μm) | Porosity (%) |
|------------------|----------------|--------------|
| 2                | 0.441          | 59.00        |
| 7                | 0.262          | 75.16        |
| 8                | 0.596          | 67.47        |
3.2.2 Mechanical Evaluation of CA-PBS/ DMSO Membrane with additive PEG. Tensile strength of the CA-PBS/DMSO with additive of PEG membrane is depicted in Figure 6. The result show that for membrane prepared with addition of 7%wt PEG increased the tensile strength until 68.366 kPa. At higher PEG concentration, some undesired big pores were formed at the surface. It was known that high porosity and large cavities in the membrane structure reduced the tensile strength [32], And then tensile strength of PEG 8% wt membrane was decreased.

![Figure 6. Tensille Strength Result Membrane with additive PEG.](image)

3.2.3 Performance of CA-PBS/ DMSO Membrane with the Addition of PEG. In Figure 7, it can be seen that salt rejection increases with the addition of PEG. It also increases with the increasing of PEG content up to 7 wt%. With the addition of 7 wt% PEG resulting salt rejection of 88.89 %. When CA-PBS/PEG membrane is immersed in the coagulation bath, the phase separation process begins. The PEG has an almost low molecular chain length and consequently good mobility which results in a smaller downward movement of the polymer on the membrane surface along with the outflow of the solvent to the coagulation bath. Therefore, the aggregated PEG molecules are distributed uniformly on the membrane surface causing the formation of a more porous surface [33], and the presence of PEG molecules on the surface makes the diffusion process of non-solvent molecules onto the films created during the compaction stage in the coagulation bath to occur which resulting in pore formation [34].

![Figure 7. Membrane’s Performance with the addition of PEG.](image)

**Conclusion**

DMSO is non-toxic solvent that is suitable for an alternative solvent in the casting solution for the preparation of CA/PBS membranes, instead of DMF. The ternary phase diagrams showed that using
DMSO instead of DMF in the same concentration leads to more permeable membranes. Both the salt rejection and permeate flux of the membranes with DMSO was higher than with DMF as a solvent. This difference in performance was caused by a denser morphology on the top surface and a more porous sublayer (fractured surface) of asymmetric membranes with DMSO as solvent. The effect of the addition of PEG in CA/PBS-DMSO membrane increases the membrane’s performance, mechanical strength, hydrophilicity and porosity. With the addition of 7 wt % PEG, salt rejection of CA/PBS membrane is 88.89 % and the tensile strength is 68.366 kPa.

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