Non-Solvent Selection for Cellulose Acetate/Polyethylene Glycol/Polyethylene Glycol-grafting-Graphene Oxide Membranes

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Abstract. Fabrication process of membrane is key factor for producing reverse osmosis membrane with a good performance for desalination process. One of the methods to fabricate membrane is phase inversion where casted membrane was immersed in the non-solvent. Non-solvent is important parameter to determine that phase inversion is success or not in resulting the morphology of the membrane. As known that morphology of the membrane contributes to the performance of the membrane. In this study, several non-solvents have been used to fabricate CA/PEG/PEG-g-GO membrane. Non-solvent used in this study were water, isopropanol, methanol, isopropanol-water and methanol-water. The morphology of the membrane was analyzed using a Scanning Electron Microscope (SEM). The thermodynamic and kinetic properties of non-solvent/solvent/polymer CA/PEG systems are studied and correlated with membrane morphology. Membrane performance was determined by salt rejection, permeate flux, and permeability. The experiment results show that the best non-solvent is water followed by isopropanol, isopropanol-water, methanol, and methanol-water. CA/PEG/PEG-g-GO membrane with water as non-solvent has a salt rejection (% R) of 75%, permeate flux (F) of 1,985 L/m².h, permeability of 0.0005 L/m².h.kPa and the morphology is sponge-like with the pore size diameter in average is 0.471 µm.

1. Introduction
Providing a hygiene and affordable clean water is one of an urgent global necessity. One of the urges is desalination technology which is potentially able to meet the global demand on a clean water. Reverse osmosis is one of the most promising and important methods for produce clean water from seawater sources and another wastewater [1]. In the reverse osmosis method, the reverse osmosis membrane acts as a barrier semipermeable which retains salt concentration resulting in clean water. Terms of the membrane that is good to use is a small pore size and pore distribution evenly, the smaller the pore, the more impurity will be left. Membrane research currently being developed aims to determine performance membrane such as flux, salt rejection and surface morphology, and hydrophilicity.

In this desalination process using the phase inversion method, which is a method in which the polymer solution is cast on a glass plate and immersed in a non-solvent coagulation bath after a given evaporation time, there will be an exchange of solvent and non-solvent to produce a porous membrane structure. The structure of the membrane is highly dependent on the thermodynamic and kinetic properties of the non-solvent, solvent and polymer ternary systems [2,3-5]. Of the several types of
polymers that exist, Cellulose Acetate (CA) was chosen to be used in the desalination process because it has the potential to produce high flux, moderate salt rejection properties, and chlorine resistance.

Graphene oxide (GO), is one of the nanoparticles that shows a very promising potential to improve the performance of RO membranes, due to its high surface area, mechanical strength, and high hydrophilicity. In addition, the oxygen functional groups of GO, including the epoxide, hydroxyl, and carboxyl functional groups on the GO surface provide good dispersion properties in a wide variety of polymers [6]. In the study of Ghaeminezhad et al. [6] GO with a size of 2.7 nm was dissolved in the CA polymer matrix to form a GO/CA nanocomposite membrane. The resulting CA membrane character is influenced by differences in GO concentrations. The addition of GO to the CA polymer matrix changed the pore structure of the membrane from finger-like to sponge-like. The CA/GO membrane produced in this study with the addition of 1 %wt GO showed a salt rejection rate of up to 90% (an increase of 80% compared to membranes made of pure CA); but, decreased in permeability flux by 30%. However, GO still has drawbacks because the dispersion is not optimal. So, in the study of Y-B. Li et al. [7] grafting PEG onto the GO surface (PEG-g-GO), PEG-g-GO is easily dispersed and the distribution of PEG-g-GO is better than GO, so that the uniform dispersion of PEG-g-GO in the fiber matrix composites improve mechanical properties as well as composites.

In this study, the CA/PEG/PEG-g-GO membrane, DMF as a solvent, was prepared using the phase inversion method. Water, isopropanol, methanol, isopropanol-water, and methanol-water as non-solvent are used in the CA/PEG/PEG-g-GO membrane immersion process. We added PEG-g-GO by 0.005 % wt, because of the best variable from our previous study [8]. The resulting asymmetric membrane will be analysed for its characteristics and performance to find the best non-solvent.

2. Materials and Methods

2.1. Materials
Cellulose acetate (CA, 30,000 Da, with acetyl content 39.8%) and polyethylene glycol-400 (PEG, 400 Da) were purchased from Sigma Aldrich. Graphene oxide (GO) with bulk density of 1.8 g/cm³, was purchased from Sigma Aldrich. N,N-Dimethylformamide (DMF) was used as solvent. The non-solvent were water, isopropanol (IPA), and methanol. NaOH was purchased from Sigma Aldrich.

ClCH₂COONa was purchased from Sigma Aldrich. HCl was purchased from Sigma Aldrich. N,N-dicyclohexylcarbodiimide (DCC), dicloromethane, and dimethylaminopyridine were purchased from Sigma Aldrich.

2.2. Grafting-Graphene Oxide (PEG-g-GO)
An amount of 3.75 g of NaOH and 3.75 g of ClCH₂COONa were dissolved in 300 mL GO suspension (1 mg/mL), then sonicated for 2.5 h, followed by neutralization with 0.1 M HCl. The resulting GO (GO-COOH) suspension was purified by repeated rinsing and centrifugation (15000 rpm for 7 min) then heated at 100 °C. 1.5 g PEG dissolved in 30 mL dichloromethane, add 0.06 g of N,N-Dimethylaminopyridine and GO-COOH dissolved in 20 mL of DMF then stirred for 5 min. 0.5 g of N,N-dicyclohexylcarbodiimide (DCC) was dissolved in 5 mL of dichloromethane and added to the GO-COOH solution after that stirred for 24 h at mild temperatures, repeated rinsing and centrifugation then heated to 100 °C, produced Grafting GO (PEG-g-GO) [9].

2.3. Preparation of membranes
The CA/PEG polymer ratio 80/20 was prepared by mixing 2 g of CA and 0.5 g of PEG in 17 mL DMF. PEG-g-GO was added to the CA/PEG membrane solution with PEG-g-GO composition 0.005 wt.% of the solvent. The solution was then mixed using a hotplate at 70 °C for 12 h, then cast on glass and immeresed into a non-solvent water, isopropanol (IPA), methanol, isopropanol-water and methanol-water for 15 minutes to produce a flat sheet membrane.
2.4. Solubility and interaction parameters

Solubility parameters are used to determine the solubility of a particular polymer. The representation of the solubility parameters developed by Hansen are dispersion (δd), polar (δp) and hydrogen bonding (δh) [10]. To determine the interaction between non-solvent-solvent and polymer-non-solvent, it can be seen from the results of the difference in solubility parameters between non-solvent-solvent (Δδ (ns-s)) and polymer-non-solvent (Δδ (p-ns)). The difference in solubility parameters between non-solvent-solvent (Δδ (ns-s)) were calculated using Eq. (1) [11].

$$\Delta\delta_{\text{ns-s}} = \sqrt{(\delta_{d,\text{ns}} - \delta_{d,s})^2 + (\delta_{p,\text{ns}} - \delta_{p,s})^2 + (\delta_{h,\text{ns}} - \delta_{h,s})^2}$$  \hspace{1cm} (1)

The difference in solubility parameters between polymer-non-solvent (Δδ (p-ns)) were calculated using Eq. (2) [10].

$$\Delta\delta_{\text{p-ns}} = \sqrt{(\delta_{d,p} - \delta_{d,\text{ns}})^2 + (\delta_{p,p} - \delta_{p,\text{ns}})^2 + (\delta_{h,p} - \delta_{h,\text{ns}})^2}$$  \hspace{1cm} (2)

Table 1. Molar volume, density and boiling point non-solvent, solvent and polymer [2, 10].

|             | V (cm³/mol) | Density (g/cm³) | Bp (°C) |
|-------------|-------------|-----------------|---------|
| Water       | 18          | 0.997           | 100     |
| IPA         | 76.8        | 0.781           | 82      |
| Methanol    | 40.7        | 0.7918          | 65      |
| DMF         | 77          | 0.95            | 153     |
| CA          | -           | 1.3             | 210     |
| PEG         | -           | 1.12            | 290     |

Table 2. Solubility parameter non-solvent, solvent and polymer [12, 13, 14].

|       | δd  | δp  | δh  | δt  |
|-------|-----|-----|-----|-----|
| Water | 15.5| 16  | 42.3| 47.8|
| IPA   | 15.8| 6.1 | 16.4| 23.5|
| Methanol | 15.1| 12.3| 22.3| 29.6|
| DMF   | 17.4| 13.7| 11.3| 24.8|
| CA    | 16.9| 16.3| 3.7 | 23.8|
| PEG   | 16.8| 10.2| 8.6 | 21.5|

Table 1 shows the molar volume, density and boiling point data for non-solvent, solvent and polymer. Table 2 shows the dispersion (δd), polar (δp), hydrogen bonding (δh), and the total solubility parameters of each non-solvent, solvent and polymer. Total solubility parameter (δt) was calculated using Eq. (3) [10, 15].

$$\delta_t = \sqrt{\delta_d^2 + \delta_p^2 + \delta_h^2}$$  \hspace{1cm} (3)

2.5. Membrane Characterization

Analysis of membrane characteristics among others analysis of Scanning Electron Microscopy (SEM), analysis of water content, and porosity. Scanning Electron Microscopy (SEM) is an analysis used to determine the morphology of the fracture side of the membrane. Water content analysis was calculated using Eq. (4) [16, 17].

$$\text{WC}\% = \frac{\text{W}_{\text{w}} - \text{W}_{\text{d}}}{\text{W}_{\text{w}}} \times 100\%$$  \hspace{1cm} (4)
The porosity \( \varepsilon \) of the resulting membrane was calculated using Eq. (5) [2].

\[
\varepsilon = \frac{(W_w - W_d)/\rho_w}{W_w/W_d - W_d/\rho_p}
\]

(5)

Where, \( W_w \) is the weight of the wet membrane, \( W_d \) is the weight of the dry membrane, \( \rho_w \) and \( \rho_d \) are densities of water and CA/PEG polymer, respectively.

### 2.6. Membrane Performance

Salt rejection is the efficiency of membrane and its ability to remove contaminates. Salt rejection was calculated using Eq. (6) [18, 19].

\[
R = \left( 1 - \frac{C_P}{C_f} \right) \times 100\%
\]

(6)

Where, R is the percentage of salt rejection, \( C_P \) (in ppm) is the salt concentration of the permeate, \( C_f \) (in ppm) is the salt concentration in the feed water. The permeate flux \( (J) \) represents the amount of pure water collected per unit time and per unit area at variable pressures. Permeate flux was calculated using Eq. (7) [20].

\[
J = \frac{Q}{t} \times A
\]

(7)

Where, \( J \) is the permeate flux (mL/h.m\(^2\)), \( Q \) is the amount of permeate (mL), \( t \) is the time and \( A \) is the area (m\(^2\)).

### 3. Results and discussion

The non-solvent type affects the morphological structure of the membrane. The exchange rate of solvent and non-solvent at the polymer solution/coagulation bath interfaces was theoretically estimated by the diffusion coefficient calculated from Tyn-Calus equation [21, 2]. The results of the diffusion coefficient can be seen in the Table 3.

\[
D_{AB} = 8.93 \times 10^{-8} \left( \frac{V_B^{0.367}}{V_A^{0.433}} \right) \left( \frac{T}{\eta_B} \right) \left( \frac{\sigma_B}{\sigma_A} \right)^{0.15}
\]

(8)

Where, \( D_{AB} \) (cm\(^2\)/s) is the diffusion coefficient of species A in B, \( V_A \) dan \( V_B \) (cm\(^3\)/mol) are the molar volumes of species A and B, \( T \) (K) is the absolute temperature, \( \eta_B \) (mPa/s) is the viscosity from species B, \( \sigma_A \) and \( \sigma_B \) (mN/m) is the surface tension of species A and B [2].

| Solvent or non-solvent | Surface Tension \( \sigma \) (mN/m) | Viscosity \( \eta \) (mPa/s) | \( D_{DMF-NS} \) (10\(^{-6}\)) cm\(^2\)/s | \( D_{NS-DMF} \) (10\(^{-6}\)) cm\(^2\)/s |
|------------------------|--------------------------------|-----------------------------|---------------------------------|---------------------------------|
| Water                  | 72.8                          | 1                           | 9.7442                          | 27.2972                         |
| IPA                    | 20.8                          | 2.4                         | 4.9563                          | 17.5752                         |
| Methanol               | 22                            | 0.6                         | 16.8750                         | 22.9431                         |
| IPA- water             | 46.8                          | 1.7                         | 6.9469                          | 19.1788                         |
| Methanol- water        | 47.4                          | 0.8                         | 21.2545                         | 23.5575                         |
| DMF                    | 36.4                          | 0.802                       | -                               | -                               |

In Table 3, which shows the highest \( D_{DMF-NS} \) values are methanol-water and methanol because it has a low viscosity, this indicates a faster exchange rate between solvent and non-solvent, in other words it only takes a very short time for DMF solvent to diffuse in the coagulation bath. non-solvent isopropanol. As can be seen in Table 3 the order of the \( D_{DMF-NS} \) values is methanol-water > methanol > water >
isopropanol-water > isopropanol. Having a low viscosity and high diffusion coefficient causes the non-solvent methanol membrane to become thin, brittle and the SEM results show no pores.

3.1. Solubility parameters for non-solvent-solvent and polymer-non-solvent
Solubility parameters is used to determine the solubility of a particular polymer. The representation of the solubility parameters developed by Hansen is dispersion ($\delta_d$), polar ($\delta_p$) and hydrogen bonding ($\delta_h$) \cite{10}. To determine the interaction between non-solvent-solvent and polymer-non-solvent, it can be seen from the results of the difference in solubility parameters between non-solvent-solvent ($\Delta \delta_{(ns-s)}$) from Eq. 1 and polymer-non-solvent ($\Delta \delta_{(p - ns)}$) from Eq. 2 in Table 4.

### Table 4. The difference in solubility parameters for non-solvent-solvent and polymer-non-solvent.

| Polymer | Solvent | Non-Solvent | $\Delta \delta_{(ns-s)}$ | $\Delta \delta_{(p - ns)}$ |
|---------|---------|-------------|-------------------------|--------------------------|
| CA-PEG  | DMF     | Water       | 31.1432                 | 37.6565                  |
| CA-PEG  | DMF     | IPA         | 9.2914                  | 14.8042                  |
| CA-PEG  | DMF     | Methanol    | 11.3248                 | 17.9266                  |
| CA-PEG  | DMF     | IPA- Water  | 18.3272                 | 25.0272                  |
| CA-PEG  | DMF     | Methanol- Water | 21.1095             | 27.6808                  |

The values $\Delta \delta_{(ns-s)}$ and $\Delta \delta_{(p - ns)}$ play an important role in the formation of asymmetric membranes. In this study, the non-solvent sequence of $\Delta \delta_{(ns-s)}$ and $\Delta \delta_{(p - ns)}$ values is the same, namely water $>$ methanol $>$ water $>$ isopropanol $>$ methanol $>$ isopropanol. The lowest value of the difference in solubility parameters shows high affinity and a high value of the difference in solubility parameters shows low affinity \cite{2, 10, 11}. The non-solvent with the lowest affinity for the difference in the solubility parameter of the non-solvent-solvent ($\Delta \delta_{(ns-s)}$) is water, this shows that there is a short interaction between water and DMF so that when the phase inversion process occurs instant demixing. Non-solvent with the highest affinity is indicated by the value of the difference in the solubility parameter of the polymer-non-solvent ($\Delta \delta_{(p - ns)}$) and the difference in the solubility of the non-solvent-solvent parameter ($\Delta \delta_{(ns-s)}$) which is low, namely the non-solvent-solvent isopropanol, this shows that there is an interaction between the CA-PEG polymer and non-solvent isopropanol during the phase inversion process so that there is a demixing delay. In Saeed Mazinani's research, states that non-solvent strength is in the order of water $>$ glycerol $>$ methanol. Non-solvent water has the highest $\delta_h$ value, hence higher hydrogen bonding ability and better non-solvent polymer compatibility, thus indicating lower non-solvent tolerance of polymer/solvent systems. Water is considered a strong non-solvent for membrane formation while methanol can be considered a weak non-solvent \cite{10}.

3.2. Microscopy Scanning Electron (SEM) analysis of CA/PEG/PEG-g-GO membranes with non-solvent Isopropanol, water, and methanol

### Table 5. Results Scanning Electroscopy Microscope Analysis of membrane CA/PEG/PEG-g-GO with non-solvent variations.
The mechanism for the formation of asymmetric membrane structures is determined by the thermodynamics and kinetics of the non-solvent, solvent and polymer. The SEM analysis results of CA/PEG/PEG-g-GO membranes for non-solvent isopropanol, water and methanol can be seen in Table 5. The difference in non-solvent has an effect on membrane morphology, the value of $\Delta \delta$ (ns-s) between nonsolvent water and solvent DMF is high, this indicates that nonsolvent water has the lowest affinity with DMF solvent, so when the phase inversion process occurs instant demixing occurs, so it is indicated by the presence of a finger-like morphological structure, while the $\Delta \delta$ (ns-s) value between non-solvent isopropanol and solvent DMF is low, this indicates that non-solvent isopropanol has a high affinity for DMF solvent, so it occurs demixing delays during the phase inversion process, which causes the non-solvent isopropanol morphological structure of the membrane to be sponge-like. Seen in Table 5, the asymmetric membrane made using non-solvent water has the most dense top layer and has finger-like and sponge-like sub-layer characteristics, whereas the isopropanol and methanol non-solvent membranes showed a thin top layer and a sponge-like sublayer [15]. In the SEM results, PEG-g-GO nanoparticles can be dispersed well, because it does not show any agglomeration of PEG-g-GO nanoparticles, on the top surface and fracture surface there is no showing lumps or black spots of PEG-g-GO nanoparticles. The CA/PEG/PEG-g-GO membrane which is non solvent isopropanol and methanol has an uneven top surface, this is due to the influence of non-solvent on the polymer.

| Non-Solvent | Average pore diameter sponge-like ($\mu$m) |
|-------------|------------------------------------------|
| IPA         | 1.011                                    |
| Water       | 0.471                                    |
| Methanol    | N. A                                     |

It can be seen in Table 6 CA/PEG/PEG-g-GO membrane with non-solvent isopropanol resulting a pore diameter larger than that with non-solvent water. In the research of Ghorani et al. [15] the same results...
were also obtained, the pore size of non-solvent isopropanol membranes was larger than non-solvent water. This membrane is classified as Microfiltration (MF) membrane, it is quite difficult to get 99% salt rejection.

3.3. Membrane Hydrophilicity Analysis

Table 7. Results of water content and porosity analysis CA/PEG/PEG-g-GO membrane with non-solvent variables.

| Non solvent       | Wet Weight | Dry Weight | %X  | Porosity (ε) |
|-------------------|------------|------------|-----|--------------|
| Water             | 0.0482     | 0.0096     | 80% | 0.8360       |
| Methanol-water    | 0.0212     | 0.0057     | 73% | 0.7752       |
| IPA-water         | 0.0371     | 0.0106     | 71% | 0.7602       |
| Methanol          | 0.0176     | 0.0054     | 69% | 0.7412       |
| IPA               | 0.0101     | 0.0052     | 49% | 0.5443       |

The results of water content analysis are one of the methods used to determine how hydrophilicity a membrane is. In Table 7 shows the results of water content and porosity of the CA/PEG/PEG-g-GO membrane with different non-solvents. The values of water content and porosity are obtained from equations 4 and 5. The non-solvent water membrane has the highest value in the water content analysis followed by methanol-water, isopropanol-water, methanol and isopropanol. In this study, the results of water content analysis are comparable to the results of membrane porosity. The non-solvent water membrane had the highest porosity value followed by methanol-water, isopropanol-water, methanol and isopropanol. In the CA/PEG/PEG-g-GO membrane, the non-solvent isopropanol and methanol have hydrophobic properties. In this study, the non-solvent isopropanol membrane has a thin structure like the plastic and the non-methanol solvent membrane has a structure that is easily broken. Other than that, the factors that cause water content and porosity of the non-solvent membrane isopropanol and methanol are small because they both have a sponge-like membrane structure and do not have finger-like, so that the surface area for water absorption in the non-solvent membrane structure isopropanol and methanol is small.

3.4. Analysis of Salt Rejection Performance and Membrane Flux

Figure 1. Salt rejection and permeate flux of CA/PEG/PEG-g-GO membrane with non-solvent variables.
Salt rejection analysis and permeate flux were performed to determine the performance of different non-solvent CA/PEG/PEG-g-GO membranes. This research produces membrane pores with a pore range of 0.4-1 µm which is included in the membrane microfiltration (MF) category, but this membrane can reject salt because the salt solution used has a very small concentration of 1000 ppm. When the salt solution is passed through the CA/PEG/PEG-g-GO membrane, there are NaCl ions that stick to the membrane walls and partially into the product. So that a relatively small rejection is generated. Figure 1 shows the results of the highest salt rejection analysis are isopropanol followed by water, isopropanol-water, methanol, and methanol-water. The average pore size of non-solvent isopropanol membranes is larger than that of non-solvent water membranes, but the high rejection is thought to be due to the dense or sponge-like membrane structure. So that the surface area to reject salt is wider. The non-solvent isopropanol membrane produced the smallest permeate flux followed by isopropanol-water, water, methanol, and methanol-water. This is due to the texture of the non-solvent isopropanol which resembles plastic, has low hydrophilicity, has a sponge-like sublayer and is supported by low porosity. This can also result in a blockage. The non-solvent water membrane has the smallest pores compared to isopropanol and methanol, has a sponge-like and finger-like structure, this membrane also has the highest hydrophilicity resulting in high flux and relatively small blockage. On the surface of the non-solvent water membrane is very dense, it is suspected that NaCl ions are stuck on the surface of the membrane, so that it produces high rejection. The non-solvent methanol membrane has high flux but low rejection, this is due to the leakage of the membrane by the texture of the membrane which is brittle and breaks easily. In this study, the optimum membrane was the CA/PEG/PEG-g-GO membrane with non-water solvent because it had high salt rejection and permeate flux. From the results of the flux did not show a decrease in flux due to agglomeration of PEG-g-GO nanoparticles.

3.5. Membrane Permeability Performance Analysis

![Figure 2](image)

**Figure 2.** Permeability of CA/PEG/PEG-g-GO membrane with non-solvent variables.

The results of membrane permeability CA/PEG/PEG-g-GO with different non-solvent can be seen in Figure 2 the permeability results of the CA/PEG/PEG-g-GO membrane with non-solvent variables were proportional to the flux results. The non-solvent isopropanol membrane produced the lowest permeability followed by isopropanol-water, water, methanol, and methanol-water.

3.6. Effect of non-solvent type on membrane pressure
The non-solvent type affects the membrane strength level, the membrane strength will affect the membrane pressure. In this study, the CA/PEG/PEG-g-GO membrane using non solvent isopropanol has a strong texture like plastic so that it has a large membrane pressure. The membrane that uses non-methanol solvent has a fragile texture so it has a small membrane pressure. The order of membrane pressure based on the non-solvent type is isopropanol > isopropanol-water > water > methanol > methanol-water. From Figure 3, the optimum membrane pressure is non-solvent water because the texture of the membrane is not easily brittle and the membrane pressure in water is below isopropanol so it is more economical.

4. Conclusion
In this study, the CA/PEG/PEG-g-GO membrane using a non-water solvent had high hydrophilicity and porosity. The smallest pore size is owned by membranes that use water as non-solvents. The highest salt rejection analysis results were isopropanol followed by water, isopropanol-water, methanol, and methanol-water, but non-solvent isopropanol membranes produced the smallest permeate flux followed by isopropanol-water, water, methanol and methanol-water. The non-solvent isopropanol membrane produced the lowest permeability followed by isopropanol-water, water, methanol, and methanol-water. The optimum membrane pressure is a membrane that uses water as non-solvents. From the above results it can be concluded that the most optimal non-solvent is water, able to produce high salt rejection and permeate flux with medium pressure compared to another non-solvent.

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