

## Kajian Kinetik dan Termodinamika dalam Sistem Selulosa Asetat-Polibutilena Suksinat (CA-PBS)/Pelarut Tunggal/Air untuk Membran Desalinasi

### Kinetic and Thermodynamic Studies in Cellulose Acetate-Polybutylene Succinate (CA-PBS)/Single Solvent/Water System for Desalination Membrane

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**ABSTRAK:** Bagian terpenting dari proses sintesis membran agar memiliki pori-pori yang diinginkan adalah proses solidifikasi membran. Proses solidifikasi membran berhubungan dengan proses demixing, dimana fasa yang kaya akan polimer mengeras; dengan demikian, matriks membran padat terbentuk. Parameter yang menentukan mekanisme pembentukan membran berdasarkan termodinamika meliputi pemisahan fasa Solvent-Polymer-Non-solvent yang dijelaskan melalui diagram fasa (Flory-Huggins Theory). Penelitian ini bertujuan untuk mengetahui prediksi awal pembentukan membran CA-PBS dengan berbagai pelarut yang digunakan dan variasi non-pelarut dalam sistem terbaik, yang dibuktikan dengan karakteristik dan kinerjanya ketika diterapkan pada membran desalinasi yang meliputi diagram ternary menggunakan cloud data titik, parameter kelarutan dengan kelarutan Hansesn, difusivitas pelarut-non-pelarut menggunakan pendekatan Persamaan Kalus Tyn dan pemeriksaan morfologi membran melalui foto SEM, dan kinerja membran yang dihasilkan melalui penolakan garam dan fluks permeat. Hasil dari perbedaan parameter kelarutan tersebut dapat diprediksi bahwa penggunaan pelarut DMF pada membran CA-PBS dapat memperkecil ukuran pori dan menghilangkan void dan macrovoid pada morfologi membran.

**Kata Kunci:** Membran; Titik awan; Parameter Solubilitas; Difusivitas; Termodinamika

**ABSTRACT:** The most important part of the membrane synthesis process to have the desired pores is the membrane solidification process. The membrane solidification process is related to the demixing process, where the polymer-rich phase solidifies; thus, a solid membrane matrix is formed. Parameters that determine the mechanism of membrane formation based on thermodynamics include the Solvent-Polymer-Non-solvent phase separation described through the phase diagram (Flory-Huggins Theory). This study aims to determine the initial prediction of CA-PBS membrane formation with various solvents used and non-solvent variations in the best system, as evidenced by its characteristics and performance when applied to desalination membranes which include ternary diagrams using cloud data points, solubility parameters with Hansesn solubility, solvent-non-solvent diffusivity using the Tyn Calus Equation approach and examination of membrane morphology through SEM photographs, and the resulting membrane performance through salt rejection and permeate flux. The results of the difference in solubility parameters can be predicted that the use of DMF solvent in the CA-PBS membrane can reduce the pore size and eliminate voids and macrovoids in the membrane morphology.

**Keywords:** Membrane, Cloud Point, Solubility Parameter, Diffusivity, Thermodynamic

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## 1. Introduction

One of the most popular methods in membrane manufacturing is the phase inversion method. The phase inversion method is a method in the process of establishing and solidifying the membrane by paying attention to the constituents of the membrane, including solvent, nonsolvent and polymer (Nyamiati et al., 2021; Pendi et al., 2015). The process of making membranes with the phase inversion method begins with the homogenization process of the solvent and polymer, after the homogenization process takes place, the polymer solution is printed in a membrane printer equipped with a cutting knife, after the process the membrane is immersed in a coagulation bath containing non-solvent (Nurkhamidah et al., 2020), the soaking process serves to evaporate the remaining solvent which eventually forms membrane pores, this process is called liquid-liquid mixing (L-L Demixing) which is continued with solid-liquid mixing or what is called phase separation followed by membrane compaction. The parameters that determine the mechanism of membrane formation are based on thermodynamics including Solvent-Polymer-Solvent-Non-solvent phase separation described through a phase diagram (Flory-Huggins Theory) (Aryanti et al., 2018; Heydarinasab, 2016) and Solubility Effects between Polymer-solvent and Solvent-Non-solvent (*Solubility Parameter*) (Gaikwad et al., 2017), and kinetically during membrane formation which includes solvent diffusion (out of membrane) and non-solvent diffusion (into polymer film) (Lua & Shen, 2013).

The material commonly used to manufacture membranes is Cellulose Acetate because this polymer is non-toxic and resistant to chlorine (Kim et al., 2016; Nurkhamidah et al., 2020), CA is usually an amorphous and rigid polymer, as would be expected from its molecular structure. Meanwhile, Polybutylene Succinate (PBS) is a biodegradable polymer which belongs to the aliphatic thermoplastic polyester group (Listyarini et al., 2017; Nyamiati et al., 2021.). The addition of PBS in the CA membrane can remove the macrovoid structure and reduce the fingerlike pore size to be smaller and lead to a sponge-like pore size (Ghaffarian & Mahmoud, 2012; V. G. S. M. Mousavi & Shoaie, 2017).

The objective of this study was to determine the initial prediction of the formation of CA-PBS membranes with various solvents used and variations of non-solvents in the best system, which is proven by its characteristics and performance when applied to desalination membranes which include ternary diagrams using cloud point data, solubility parameters with Hansen solubility, the solvent-non-solvent diffusivity using the Tyn Calus Equation approach and the morphological proofing of the membrane through SEM photos, and the performance of the resulting membrane through salt rejection and permeate flux.

## 2. Research methods

### 2.1 Material and Method

The material used is a cellulose acetate (CA) polymer with a molecular weight of 30,000 Da; poly(1,4-butylene succinate)

(PBS) with a density of 1.3 g/mL at 25°C which can be purchased from Sigma Aldrich. The solvents used included Dimethylformamide (DMF), Tetrahydrofuran (THF), Acetone and N-Methyl-2 Pyrrolidone (NMP) purchased from Merck, and distilled water was used as a non-solvent.

### 2.2 Fabrication of Membrane

The manufacture of the membrane begins with taking a variation of CA/PBS polymer with a ratio of 80/20 where the total polymer used is 2.5 grams. The deposition and solidification technique used is to use the phase inversion method, the first step is to dissolve the polymer into 17 mL of Acetone, NMP, THF and DMF solvents until homogeneous at temperatures below the boiling point of each solvent. The homogeneous solution is poured into glass and formed using a film applicator equipped with a cutting knife, after which it is immersed in a coagulation bath containing non-solvent water for 14 minutes, after which the membrane can be dried and tested for membrane performance and characteristics.

### 2.3 Cloud Point Diagram

The ternary diagram is a representation of the cloud point of a membrane solution before printing, cloud point measurement aims to see the initial turbidity of the polymer, solvent, and non-solvent composition of a membrane composition, this turbidity is an unstable state of the polymer and solvent which is disturbed by the presence of the non-solvent used. The method used is the CA-PBS polymer combination using a certain composition dissolved in a predetermined solvent, after which it is included in a sample bottle that has been equipped with a magnetic stirrer, where the stirring process used is for 24 hours. After 24 hours of stirring, the non-solvent used in this case is water added little by little in the polymer solution that has been homogenized, the addition takes place until the clear solution turns cloudy like milk.

During this non-solvent titration, the ambient temperature was maintained. After the polymer solution turned cloudy, the process was continued for 20-40 minutes to confirm whether the cloudy color changed back or not, after which the volume of the added non-solvent was recorded, as the ternary composition of the turbidity point after which the amount of non-solvent, solvent, and polymer present in the sample was calculated and presented in a ternary diagram.

### 2.4 Parameter of Hansen Solubility

Prediction of the determination of solvents, non-solvents, and polymers in the constituent of a membrane has a very important role, one of the predictions before the membrane synthesis is to use the Hansen solubility parameter, this prediction is used to determine the quality of solvents and non-solvents in their solubility with the polymer used, this can affect the method of deposition and compaction of the membrane and the formation of pores from the membrane. These characteristics are influenced by the interaction between the polymer and the membrane formers, in this case the solvents and non-solvents used. Solubility is an

important point in the initial process of membrane synthesis. Therefore, the Hansen solubility parameter is divided into three parts, namely the dispersion force component ( $\delta_d$ ), the hydrogen bond component ( $\delta_h$ ) and the polar component ( $\delta_p$ ), and is expressed by Eq. (1):

$$\delta^2 = \delta_d^2 + \delta_p^2 + \delta_h^2 \quad (1)$$

Where :

$\delta_d$  = Polymer dispersion energy  
 $\delta_p$  = The energy of the intermolecular polar  
 $\delta_h$  = Energy from hydrogen bond

For the value of the solubility parameter derived from the mixing solvent using the equation (2):

$$\delta_{i,s} = \frac{X_1 V_1 \delta_{i,1} + X_2 V_2 \delta_{i,2}}{X_1 V_1 + X_2 V_2} \quad i = j, k, l \quad (2)$$

Where :

X = Molar Fraction  
V = Molar Volume

(Brandrup J, Immergut E.H, 1999; Gebru & Das, 2017; Hansen, 2007b; S. M. Mousavi & Zadhoush, 2017)

After the Hansen Solubility Parameters of each compound are obtained, the difference in solubility between the 2 compounds can be predicted using the equation (3) and (4):

$$\Delta\delta_{(s-p)} = [(\delta_{ds} - \delta_{dp})^2 + (\delta_{ps} - \delta_{pp})^2 + (\delta_{hs} - \delta_{hp})^2]^{0.5} \quad (3)$$

$$\Delta\delta_{(ns-s)} = [(\delta_{dns} - \delta_{ds})^2 + (\delta_{pns} - \delta_{ps})^2 + (\delta_{hns} - \delta_{hs})^2]^{0.5} \quad (4)$$

(Gebru & Das, 2017; S. M. Mousavi & Zadhoush, 2017)

## 2.5 Solvent Diffusion (out of the membrane film) and diffusion of Non-solvent (in the membrane film)

The exchange rates of solvent-non-solvent and non-solvent-solvent can be approximated using the Tyn-Calus equation (5) :

$$D_{AB} = 8,93 \times 10^{-8} \left( \frac{V_B^{0,267}}{V_A^{0,433}} \right) \left( \frac{T}{\eta_B} \right) \left( \frac{\sigma_B}{\sigma_A} \right)^{0,15} \quad (5)$$

(Tyn & Calus, 1975)

where :

$D_{AB}$  = Diffusivity Coefficient of A to B (cm<sup>2</sup>/s)  
 $V_{A,B}$  = Molar Volume A/ B (cm<sup>3</sup>/mol)  
 $\eta_B$  = Viscosity of component B (mPa/s)  
 $\sigma_B$  = Surface tension B/A (mN/m)

To get the binary surface tension value using the equation (6) :

$$\sigma = \sigma_1 \phi_1^S + \sigma_2 \phi_2^S \quad (6)$$

Where  $\phi_i^S$  is the volume fraction of each component and is obtained from the equation (7) :

$$\phi_1^S = \frac{V_1 X_1^S}{V_1 X_1^S + V_2 X_2^S} = 1 - \phi_2^S \quad (7)$$

Where  $V_i$  is the molar volume of each component and  $X_i^S$  is the fraction of each binary mixture.

## 2.6 Morphology Membrane

The morphology of the membrane can be observed and analyzed using a Scanning Electron Microscope (SEM), SEM is an analysis to see the cross section of the membrane, can see the pore structure, protrusions, and macrovoids formed. Before analyzing using SEM analysis, the sample is first cut by breaking it in liquid nitrogen, this aims to make the fracture that occurs not damaged and neat, before being inserted in the SEM machine, the sample is first coated with gold which aims to make the sample not burn and run out when analyzed.

## 2.7 Membrane Performance

Flux and salt rejection are tests carried out to see the performance of the membrane that has been synthesized. The performance test system used uses cross flow. Membrane performance test equipment includes a feed reservoir, pump, pressure gauge, membrane holder and membrane sheets to be tested. The permeate flux equation can be seen in Eq (8) :

$$Permeate \text{ Flux} = \frac{Q}{A \Delta t} \quad (8)$$

Where Q is the amount of permeate (L), A is the effective membrane area (m<sup>2</sup>) dan  $\Delta t$  is the sampling time (h). Salt Rejection is calculated using the following equation (9) :

$$\%R = \left( \frac{C_f - C_p}{C_f} \right) \times 100\% \quad (9)$$

Where R is salt rejection, and  $C_f$  and  $C_p$  are feed and permeate concentrations.

## 3. Results and Discussion

### 3.1 Hansen Solubility Parameter

In looking at the solubility of polymer-solvent, polymer-non-solvent, it can be approached by using the hansen solubility parameter. This solubility is used to predict the quality of the resulting membrane, which affects the process of compaction of membrane characteristics related to the method used, namely the compaction method with phase inversion, besides that the Hansen solubility parameter is also used to predict the morphology of the membrane to be produced, the membrane morphology can affect the quality performance of the membrane itself. The influence in the hansen solubility parameter can be seen from the level of interaction between the polymer, solvent and non-solvent used in the formation of the membrane. The value to be used in the calculation of

these parameters can be seen in the Table 1 of each component produced.

**Table 1.** Value of Solubility Parameter for Each Component (Hansen, 2007a)

Polymer/Solvent/Non-solvent	$\delta_d$	$\delta_p$	$\delta_h$	$\delta$
Cellulose Acetate	16.9	16.30	3.70	23.80
PBS	18.57	7.71	9.63	22.30
DMF	17.40	13.70	11.30	24.90
THF	0.50	0.50	17.74	12.00
Acetone	0.40	0.60	17.90	11.10
NMP	0.30	0.70	18.07	10.30
Water	15.50	16.00	42.30	47.80

The Hansen solubility parameter can calculate the interaction between the three types of materials that will be used in membrane manufacturing, including the interaction between solvents with polymers; non-solvents with solvents and non-solvents with polymers. by using the equation (3) and (4) the solubility value can be seen in **Table 2**.

**Table 2.** Value of Solubility Difference Parameters for Polymer-Solvent-Non-solvent

No	Polymer (80:20)	Solvent	Non-solvent	$\Delta$ (p-s)	$\Delta$ (s-ns)	$\Delta$ (p-ns)
1		Acetone		9.53	33.14	37.48
2		THF		13.55	38.41	37.48
3		NMP		10.57	31.36	37.48
4	CA-PBS	DMF	Water	6.47	31.14	37.48

The value of the Hansen solubility parameter can be indicated to be inversely proportional to the solubility values of the polymer, solvent and non-solvent. If the resulting hansen solubility value is small, the interaction between polymer-solvent; solvent-non-solvent; and polymer-non-solvent will be stronger (Geburu & Das, 2017). In the solubility parameter values that have been calculated using equations (3) and (4), it is found that the solubility values of (p, s) and (s, ns) have decreased for solvent variations from DMF < Acetone < NMP < THF, while the solubility value of (p,ns) after being calculated by equations (3) and (4) has the same value because the amount and type of polymer and nonsolvent used are the same. In this case it can be concluded that by using 4 different solvents, the interaction between polymer-non-solvent and solvent-non-solvent increases with the order DMF > Acetone > NMP > THF.

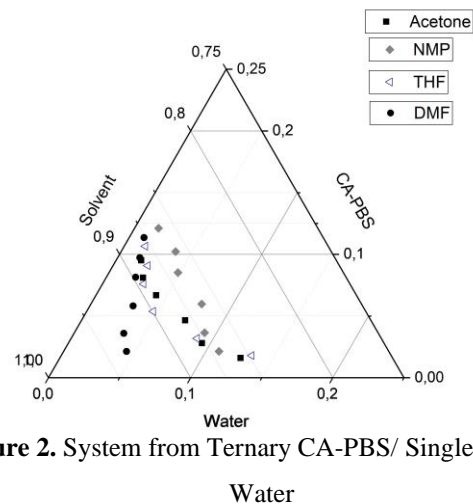
In the literature it is explained that the solubility which has a very large difference can lead to a faster membrane formation process which is commonly known as instant liquid-liquid demixing, this process has a weakness which must be avoided because it can make the formation of pores that are not optimal and result in the formation of cavities which very large which can trigger the formation of macrovoids in the morphology of the membrane, this is because the instant demixing process takes place so quickly,

that the resulting pores are not good, while The lower solubility difference means that the process that occurs is delayed-demixing which means it slows down the membrane compaction process which is predicted to produce smaller pore sizes and can remove macrovoids in the pores (Ebrahimpour et al., 2017). Therefore, in the above solvent variations, it can be predicted that using DMF solvent on the CA-PBS membrane can reduce the pore size and eliminate voids and macrovoids in the membrane morphology.

### 3.2 Ternary Diagram

CA-PBS/ Single Solvent/ Water ternary system graph from the point cloud process shown in Figure 2. The phase diagram is generated from the cloud point titration process between the solvent, polymer, and non-solvent. The titration data is then used to determine the ternary diagram commonly called the phase diagram, this diagram becomes an illustration to predict the phase transition that occurs when the phase separation process of the solvent and non-solvent in the phase inversion deposition method.

The mechanism of membrane formation, commonly called film formation, takes place in the following order; first the mixing process between the polymer and the solvent that has been homogenized, after that it continues with the membrane molding process and then finally the immersion process in a nonsolvent which is useful for separating the solvent from the polymer which finally occurs the membrane solidification process. In the third process, the thermodynamic instability of the mixture between polymer and solvent is disturbed by the presence of non-solvent in the coagulant bath.



**Figure 2.** System from Ternary CA-PBS/ Single Solvent/ Water

In the ternary diagram shown in Figure 2, it can be identified that the binodal axis generated from each solvent used shows that the DMF solvent shows binodal lines that lead closer to the non-solvent area, the process means that the membrane mixture made with DMF solvent will lead to a liquid-liquid demixing process followed by solid-liquid more than the Acetone, NMP and THF solvents. This event indicates that by using DMF solvent, the polymer will diffuse more stably and form a better membrane matrix film compared to the other 3 solvents, this finding can be compared to the solubility of Hansen that the solubility value

of DMF non-solvent with CA-PBS membrane also has good tensile strength compared to the other solvents that have been used.

### 3.3 Diffusivity

Diffusivity in membrane synthesis predisposition has a major effect on the time required for membrane solidification and formation. The diffusivity of the membrane solvent into the non-solvent used can be defined as the speed of the solvent out through the pores of the membrane matrix and out towards the coagulation bath where the non-solvent is located. In terms of the diffusivity of the non-solvent in the polymer, it can be defined as the speed of the non-solvent entering through the membrane pore and finally nesting in the membrane matrix.

A large diffusivity indicates that the exchange rate between the two components is getting faster which indicates the occurrence of an instantaneous demixing process, while a small diffusivity value indicates a delayed demixing process which means that the formation of the membrane is more controllable and results in a membrane that has smaller pores and loses shape. cavities and macrovoids on membrane morphology (Lua & Shen, 2013). In **Table 3**, the diffusivity values for non-solvent-solvent and non-solvent in various solvents are shown. The solvent-non-solvent diffusivity has a value of  $1.3397 \cdot 10^{-5}$  -  $1.0894 \cdot 10^{-5}$ , a higher value of  $D(s\text{-}water)$  causes a faster exchange rate between solvent and non-solvent which can be called instantaneous demixing (Padilha & Borges, 2019).

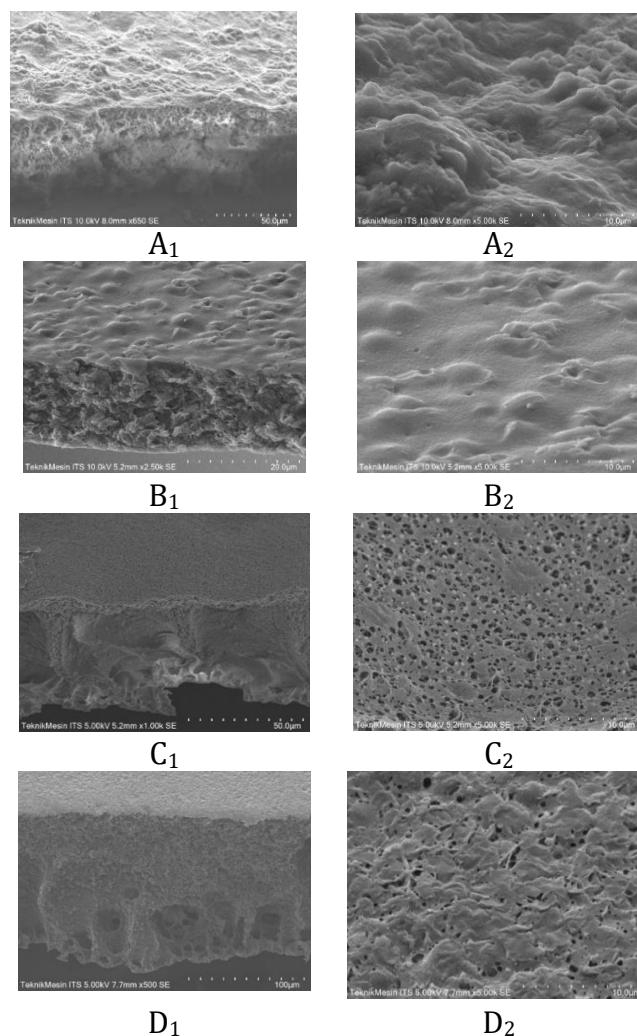
**Table 3.** Value of Diffusivity Membrane-Forming Component

Solvent /N.s	Surface Tension	Viskositas	Volume Molar	$D_{(water-s)} \times 10^{-7}$	$D_{(s-water)} \times 10^{-5}$
Water	71.15	0.79	18.07	-	-
Acetone	22.10	0.32	73.52	2.73	1.33
NMP	40.09	1.65	96.43	2.45	1.08
DMF	34.40	0.79	77.11	2.82	1.22
THF	26.03	0.48	81.09	2.60	1.25

The resulting value indicates that when the solvent diffuses into the coagulation bath, it takes a shorter time to diffuse so that the demixing process takes place faster. The order of the largest non-solvent-solvent diffusivity is  $NMP > DMF > THF > Acetone$ , this means that using Acetone solvent has a smaller diffusivity compared to other solvents, which is predicted to occur delayed demixing process in membrane compaction which will later have a pore structure that has a larger size smaller and eliminates voids and macrovoids in the membrane (Ramadhani et al., 2021).

In **Figure 3** is the result of SEM of membrane morphology with solvent variations Fracture Surface and Top Surface. For membranes made from THF solvents (B1 and B2) formed from the instantaneous demixing process which causes the formation of macrovoids and the membrane is more symmetrical, while for pores resulting from variations in acetone solvent, macrovoids and the resulting top surface are more symmetrical. For membranes with NMP solvents, which have very large cavities and the

pores formed are not maximized, while changing the solvent to DMF can reduce the size of Sponge-like and Fingerlike pores and asymmetric membrane pores, this is in accordance with predictions from the Hansen solubility and diffusivity parameters of the solvent- non-solvent that the longer a solvent diffuses into the non-solvent and the smaller the value of the difference in solubility, it will have a delayed demixing process which results in smaller pore sizes and eliminates cavities and macrovoids from the membrane morphology (Gebru & Das, 2017; Lua & Shen, 2013).

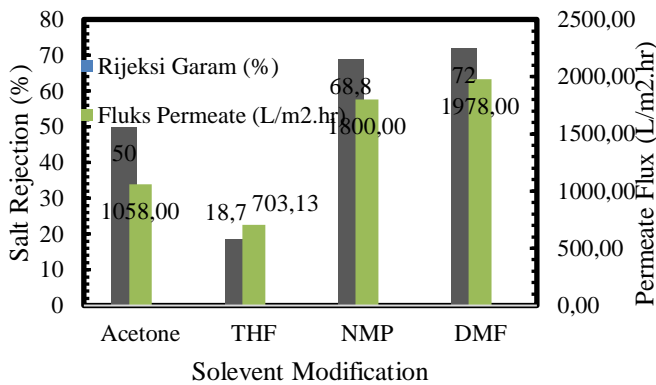


**Figure 3.** CA-PBS Membrane Morphology from variation of (A<sub>1</sub>) Fracture Surface (A<sub>2</sub>) Top Surface Solvent Acetone, (B<sub>1</sub>) Fracture Surface (B<sub>2</sub>) Top Surface Solvent THF, (C<sub>1</sub>) Fracture Surface (C<sub>2</sub>) Top Surface Solvent NMP, (D<sub>1</sub>) Fracture Surface (D<sub>2</sub>) Top Surface Solvent DMF

Salt rejection is a membrane performance test that determines whether the membrane can be used to filter components, in this case salt, salt rejection can be interpreted as the selectivity of the membrane in sorting out the components to be separated.

The membrane performance test is also supported by the permeate flux test, the permeate flux test is a parameter to determine how much product is obtained per unit area and

time. The value of salt rejection and permeate flux from the solvent variation decreased according to the solvent order DMF > NMP > Acetone > THF, this corresponds to the morphology of the resulting membrane in Figure 4. The larger the pore, the lower the salt rejection produced.



**Figure 4.** Value of Salt Rejection and Permeate Flux of CA-PBS Membrane Solvent Variation

#### 4. Conclusion

Asymmetric membrane formation for desalination can be identified and based on the interaction ability between the 3 phases used, namely solvent, polymer, and non-solvent, this interaction can be approached using thermodynamic and kinetic studies. From the results obtained, the difference in Hansen solubility for (p, s) and (s, ns) for the solvent variation decreased from DMF < Acetone < NMP < THF. In this case, it can be concluded that the polymer-solvent interaction decreases in the following order THF < NMP < Acetone < DMF.

The order of the largest non-solvent-solvent diffusivity is NMP > DMF > THF > Acetone, This means that using Acetone solvent has a smaller diffusivity compared to other solvents, which is predicted to occur a delayed demixing process in membrane compaction which will have a pore structure that has a larger size smaller and eliminate cavities and macrovoids in the membrane. In this study, it was also confirmed by conducting membrane performance tests which included salt rejection and permeate flux from solvent variations decreased in accordance with the order of solvents, namely DMF > NMP > Acetone > THF. The results of the difference in solubility parameters are using DMF solvent on the CA-PBS membrane can reduce the pore size and eliminate voids and macrovoids in the membrane morphology.

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