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**The Effect of Casting Solution and Non Solvent Composition on the
Performance of Polysulfone Membranes against Chromium (VI) .**Keshinanta Dyah Ayu Maharani¹, and Nita Kusumawati^{2*}.⁵
¹Departement of chemistry, Universitas Negeri Surabaya, Surabaya, Indonesia.²Department of Chemistry, Universitas Negeri Surabaya, Surabaya, Indonesia.**ABSTRACT**

In general, this research aims to study membrane preparation process which are capable to form asymmetry structures on the polysulfone membrane, resulting in increased permeability and selectivity. More specific, this study aims to determine the effect of casting solution and non-solvent composition on the formation of asymmetry structure, mechanical strength and membrane performance in the separation of chromium (VI). In this research, H₂O and CH₃OH are chosen as non-solvent solution which is used to induce phase inversion. To determine the effect of casting solution and non-solvent composition on membrane performance, casting solution composition was varied in the range of PSF/NMP/PEG400 (% w/w) 12/84 /4-16/84/0, and the non-solvent composition was varied in the range H₂O/CH₃OH (%v/v) 100/0, 75/25, 50/50. Permeability (flux) measurement result of PSf membrane using Dead end membrane reactor showed that the range of flux was 8060.39 L/m².h - 468.78 L/m².h. Membrane with the smallest PSf composition on the casting solution and the biggest H₂O composition on the non-solvent has the largest flux and smallest selectivity (rejection percentage). From the results of selectivity (rejection percentage) measurement against chromium (VI), which performed by UV-Vis Spectrophotometer, shows the range of 12.61% - 49.19%. Meanwhile, mechanical strength measurement results for the membrane with the smallest composition of PSf and biggest H₂O is 2458.89 N/m², and for the biggest composition of the PSf with the smallest composition of H₂O is 3427.06 N/m². Generally, in this research, polysulfone membrane having obtained the high mechanical strength and performance.

Keyword: Membranes, Polysulfone, Polyethylene glycol, Chromium (VI)**Corresponding author*

INTRODUCTION

In present, the textile industries are some manufacturing industries which have important effect in Indonesia [1]. However, the textile industry also gives serious problems to the environment [2]. The main serious problem caused by the textile industry is the problem of waste, where the levels of total chromium is one of the parameters that can be used as an indicator of polluted water, and considered dangerous for the environment [3]. In the textile industry, chromium is mainly used in dyeing process both as a dye and a mordant (binding color) [4]. Generally, the chromium ion is found as trivalent chromium [Cr (III)] and ionic hexavalent chromium [Cr (VI)], which hexavalent chromium ion is more toxic and carcinogenic [5], [6]. One method that can be used for the treatment of heavy metal chromium (VI) is the membrane technology.

During this time, preparation of PSf membranes has been successfully done by two kind methods, namely sintering and track-etching [7]. But on further development, those methods began to be abandoned because of the membrane produced by those methods still has a high mass transfer resistance, cause of symmetry structure [8]. The high mass transfer will produce membrane with distant ratio differences of flux and rejection, because the membrane is dominated by the sponge pore structure. The condition in which the membrane is dominated by sponge structure thus, can increase the likelihood of fouling. Therefore, to obtain the PSf membrane with an equal ratio of flux and rejection, in [20] research will be pursued formation of asymmetry structure. The asymmetry structure of the membrane can be produced through a phase inversion method with immersion precipitation technique [9], [10].

In order to produce PSf membrane with equal ratio of flux and rejection, PSf membrane that will be used for treatment of chromium hexavalent, made with varied composition of casting solution and non-solvent composition using phase inversion method which is induced by immersion precipitation techniques [11]. The casting solution composition in this research was PSf/NMP/PEG (%w/w) 12/84/4; 12.5/84/3.5%; 13/84/3; 13.5/84/2.5; 16/84/0, while the non-solvent composition was H₂O/CH₃OH (%v/v) 100/0, 75/25, 50/50. The results of this research analyzed using several instruments: "Dead-end" membrane reactor for measuring permeability, spectrophotometer (UV-Vis) to measure the selectivity (rejection), autograph to measure the mechanical strength, and scanning electron microscopy (SEM) instrument for imaging the surface and cross section morphology of the membrane [12].

EXPERIMENTAL

Material

Materials research include: Polysulfone (PSf) (Sigma-Aldrich, $d = 1.24 \text{ g/mL}$ at 25°C), N-methyl pyrrolidone (NMP) (Sigma -Aldrich, MW: 99.13 g/mole), technical methanol (PT. BRATACO, batch: K-02801-3), PEG (PT. BRATACO, mw : 400, batch : X14648), distilled water, sulfuric acid (H₂SO₄) (Ajax Finichem Pty Ltd, >98%), 1,5-diphenylcarbaid (Merck, GR for analysis and redox indicator), and gauze for supporting layer of the membrane (Kasa Husada Indonesia, 254 Mesh).

Equipment

Research equipment includes: Ohaus analytical balance, NESCO LAB MS-H280-Pro magnetic stirrer with temperature control for mixing the membrane material, Seiko stopwatch, coagulation bath (self-made), and OP-211 pH meter. While the research instruments include: Shimadzu AG-10TE autograph, dead-end membrane reactors (self-made) which is connected with compressor as a pressure source, Shimadzu UV-1800 UV-Visible spectrophotometer, and Zeiss EVO MA10 Scanning Electron Microscopy (SEM).

Preparation of Polysulfone Membrane

The PSf membrane was made by phase inversion methods. Polysulfone was dissolved in NMP then added PEG [13-16]. The ratio of casting solution composition: (PSF/NMP/PEG) (%w/w): 12/84/4; 12.5/84/3.5; 13/84/3; 13.5/84/2.5; 16/84/0. The solution was stirred with a magnetic stirrer at 60°C for two hours [8]. A homogeneous polymer solution then casted using casting knife on a glass plate that coated with polyester [17-18], with 0.8 mm thickness, and then soaked in a coagulation bath containing a mixture of distilled water and methanol with ratio H₂O/CH₃OH (%v/v): 100/0; 75/25; 50/50. A solid PSf membrane formed then washed

by using distilled water to remove the remnants of the solvent. After that, the solid membrane dried on the open air.

Polysulfone Membrane Performance Test

The performance of PSf membrane was determined by measuring flux and rejection of the membrane against chromium (VI). For PSf membrane permeability test using Dead end membrane reactor, dried membrane was cut ± 6 cm. On the equipment was applied pressure 1 bar as a driving force of the 5 ppm Cr (VI) feed solution. The feed solution flowed through the membrane, and the time when all of the feed solution filtered out is recorded. Furthermore, according to the recorded time, the volume of feed solution, and the surface area of the membrane, the flux was calculated.

Furthermore, to determine rejection coefficient of the PSf membrane, feed solution with maximum wavelength of 542 nm, measured concentrations before and after passing through the PSf membrane using UV-Vis spectrophotometer, where in every measurement process, feed solution was controlled at pH 2 using H₂SO₄ 1M and 1,5-diphenylcarbazide $1,44 \times 10^{-4}$ M was added too into the solution as complex agent Cr (VI) [19].

Polysulfone Membranes Characterization

The characterization of PSf membrane on this research includes physical and mechanical characterization. The physical characterization of the PSf membranes is done by imaging the surface and cross section morphology using SEM. While the mechanical characterization done by measure the mechanical strength of the PSf membrane using Shimadzu AG-10TE autograph. Mechanical strength test of PSf membrane gave the magnitude of stress and strain which experienced by PSf membrane over the force of 100 kN [20], then they were compared to calculate the young's modulus .

RESULTS AND DISCUSSION

Polysulfone Membrane Preparation

In present, phase inversion is commonly used as preparation method for polysulfone membrane because of its simple and easy to produce membrane, so it's cost less in production process. Preparation of PSf membrane was used casting solution composition range (PSf/NMP/PEG) (%w/w) 12/84/4 – 16/84/0 and non-solvent composition range (H₂O/CH₃OH) (%v/v) 100/0 – 50/50. Casting solution was stirred in specific temperature 60 °C for two hours. The temperature was chosen because that the higher stirring temperature used, the greater pore size formed in the membrane. Meanwhile the selection of NMP as PSf solvent in this study is due to smaller solubility parameter differences between PSf and NMP than PSf with the other solvent was probably used in the PSf membrane preparation. The solubility parameter differences between PSf, NMP, and DMAc shown in table 1.

Table 1. Hansen solubility parameters between PSf and the solvents (MPa^{1/2}) [13]

Material	δ_D	δ_P	δ_H	δ_t	$[\delta_{tPSf} - \delta_t Solvent]$
PSf	19,7	8,3	8,3	22,93	
NMP	18	12,3	7,2	22,96	0.03
DMAc	16,8	11,5	10,2	22,77	0.16

On the preparation process of PSf membrane, Polyethylene Glycol (PEG) added as additive substance, in order to increase porosity which automatically will be increase the permeability of PSf membrane. However, uses of additive with high molecular weight, such as PEG, can increase the formation of macrovoid which can decrease the mechanical strength of the membrane. Because of that, uses of non solvent from the combination of H₂O and CH₃OH, expected will be decrease the solubility parameter differences between solvent and non-solvent, so it can reduce macrovoid. A homogeneous polymer solution cast used a casting knife on a glass plate (thickness of 0.8 mm) that coated with polyester. The illustration of casting knife methode can be observed in figure 1. In the casting process of PSf membrane, was used polyester as supporting layer. Polyester was used to increase the mechanical strength of PSf membrane.

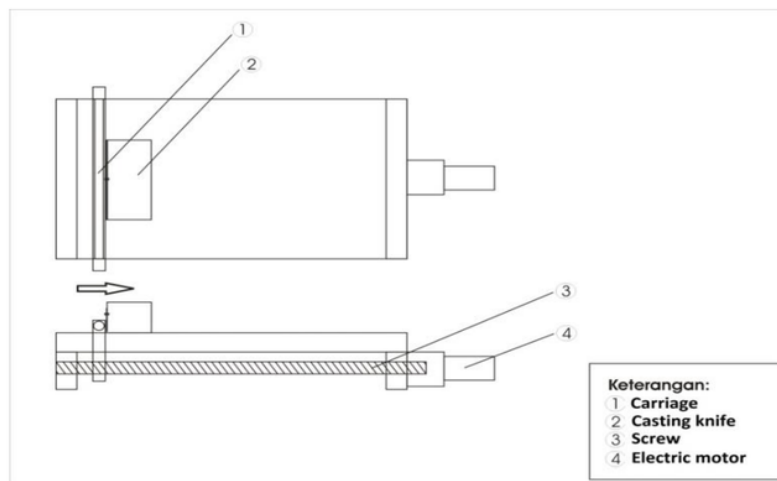


Figure 1. Illustration of polysulfone membrane casting with casting knife method

On this research, the preparation of PSf membrane has been done using phase inversion method with immersion precipitation technique, where the liquid phase changes into the solid phase induced by particle change processes between solvent (NMP) with non-solvent (H₂O and CH₃OH). The differences between the solvent and non-solvent solubility parameter that used in the membrane preparation process will be increase the mixing temperature (ΔH) of the casting solution, that will be followed by ΔH and ΔG (Gibb's free energy) increases. The increased of Gibb's free energy will trigger the occurrence of membrane casting solution coagulation, so it can generated the solid PSf membrane [21]. The solubility parameter differences between solvent and non-solvent that are used on the PSf membrane preparation shown in table 2.

Table 2. Hansen solubility parameters between solvent and non-solvent [21]

Non Pelarut	δ_d (MPa ^{1/2})	δ_p (MPa ^{1/2})	δ_H (MPa ^{1/2})	δ_t (MPa ^{1/2})	$\Delta\delta$
NMP	18	12,3	7,2	22,96	
Metanol	11,6	13,0	24,0	29,7	6,74
Air	12,2	22,8	40,4	48,0	25,04

The interactions between components of PSf membrane affect in differences of the phase inversion timing for each membrane composition, it can be observed in figure 3. Interactions that may occur between the components of PSf membrane can be observed in figure 2.

Characterization of PSf Membrane Performance

The performance of PSf membrane (permeability and selectivity) characterized with dead-end reactor and a spectrophotometer (UV-Vis).

Permeability of PSf Membrane

Flux value that was gotten, showed the amount of permeate volume which passed by the membrane at the specific wide of membrane and specific flow rate time. Permeability (flux) test aims to inform characteristic of the membrane's pore, include: porosity, pore size, and pore structure.

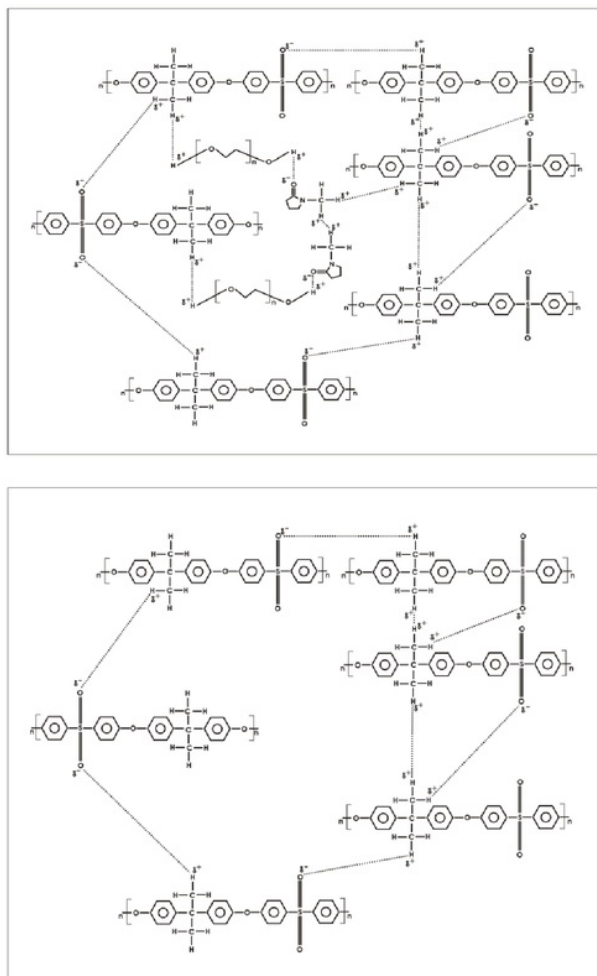


Figure 2. Interaction between the components of PSf membrane: on casting solution (a); solid membrane (b) (hypothetic)

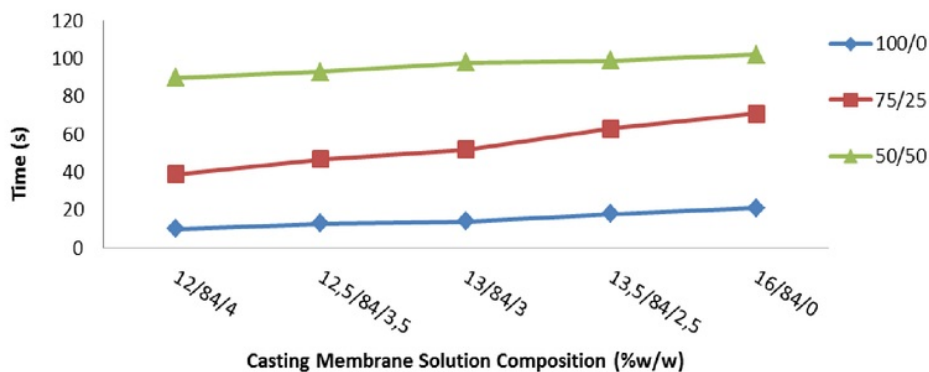


Figure 3. Phase inversion timing

In this research, permeability test done by using $K_2Cr_2O_7$ as a feed solution with 5 ppm concentration, and 250 mL volume. The pressure was set at 1 bar. The interaction between PSf membrane and Cr (VI) showed in figure 4, and the illustration of dead-end reactor membrane showed in figure 5.

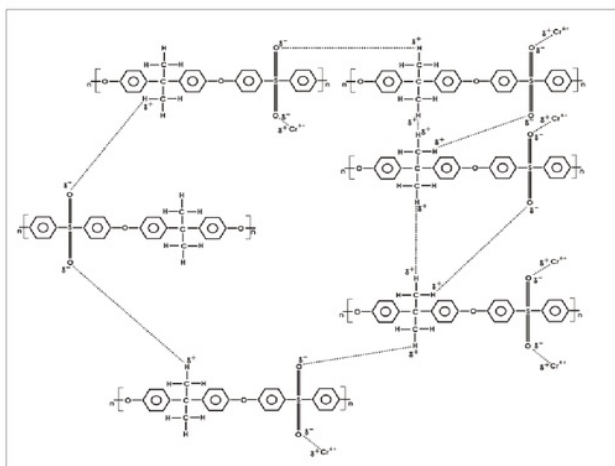


Figure 4. Interaction between solid membrane and Cr (VI) (hypothetic)

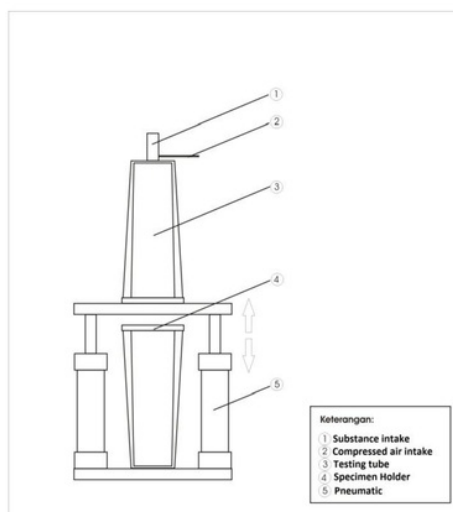


Figure 5. Illustration of dead-end reactor membrane

The PSf polymer composition in casting solution and the H_2O composition on the non solvent greatly affects the permeability of the membrane. Polysulfone membrane with the smallest amount of PSf polymer and biggest volume amount of H_2O has the highest flux value, which amounted at $8578.07 \text{ Lm}^{-2}\text{h}^{-1}$. Whereas, polysulfone membrane with the biggest amount of PSf polymer and the smallest volume amount of H_2O has the lowest flux value, which amounted at $468.74 \text{ Lm}^{-2}\text{h}^{-1}$. In addition, the effect of the casting solution and non solvent composition on the membrane permeability can be explained using the equation [11]:

$$\Delta G = \Delta H - T \Delta S \quad (1)$$

$$\Delta H = \varphi_1 \varphi_2 V (\delta_1 - \delta_2)^2 \quad (2)$$

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When the difference in solubility parameter between the solvent and non solvent becomes higher, ΔH reaction will also increase. The increasing of ΔH , will be followed by an increase in ΔG reaction, that will cause an unspontaneous in the casting solution system. These conditions made phase inversion process of the casting solution becomes faster. Faster phase inversion process has resulted in less availability of time that sufficient to form more complex interaction between the membrane molecules. Then, this led to form larger pores size on the membrane. The pore size determines the time it takes the feed solution ($K_2Cr_2O_7$) to flow through the PSf membrane will be shorter, resulting a greater flux value, and vice versa. Flux data of the PSf membrane can be observed in figure 6.

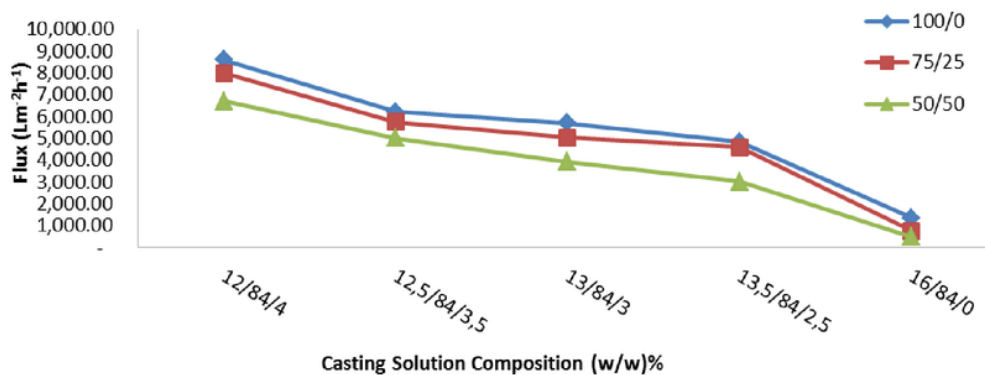


Figure 6. Flux of the PSf membrane against $K_2Cr_2O_7$

PSf membrane selectivity

Selectivity is one of the parameters that can determine the ability of the membrane to withstand a particular species and other species that passed in this case a very influential to it is the large size of the particular species and the pore size of the membrane. The parameter used to measure the selectivity of the membrane, called the rejection coefficient. In this research, $K_2Cr_2O_7$ feed solution with a maximum wavelength of 542 nm, measured concentrations before and after passing through the PSf membrane using UV-Vis spectrophotometer. The rejection coefficient of the PSf membrane shown in figure 7.

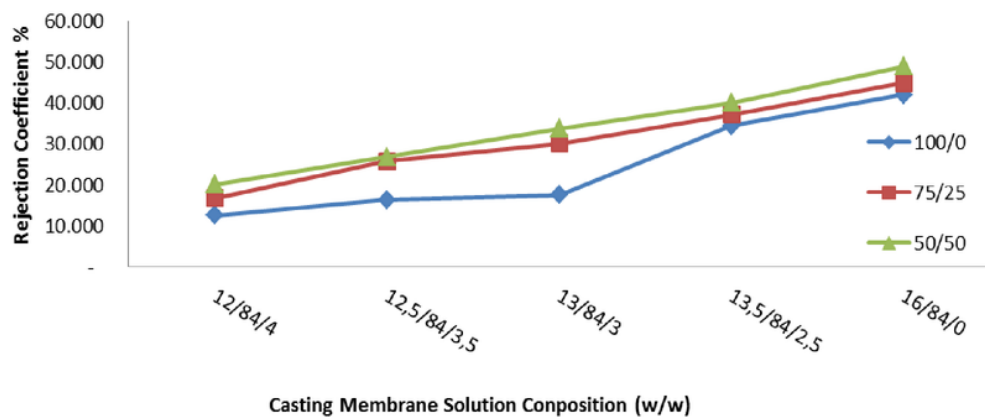


Figure 7. The rejection coefficient of the PSf membrane against $K_2Cr_2O_7$

Based on data from the rejection coefficient, the membrane with the smallest amount of PSf polymer and the biggest amount of H₂O volume has a minimum rejection coefficient, which amounted 12.67%. While the membrane with the biggest amount of PSf polymer and the smallest amount of H₂O volume has a maximum rejection value amounting to 49.19%. In this research, the highest value of selectivity PSf membrane, only reached 49.19%, those values are still far from the maximum value of selectivity, i.e. by 100%. It indicates that filtration role doesn't really exist to filter Cr (VI) ions on membrane filtration, PSf. That is because the ionic formed of Cr (VI) are relatively smaller than the molecule of water. So it is confirmed that the ions Cr (VI) which only filtered less than 50%, and 50% other passed along with the water through the membrane of the PSf. The highest value of this selectivity has been obtained 49.19% of this research, because the selectivity of PSf membrane more caused by adsorption phenomena than filtration phenomena. The adsorption phenomena on this case worked through physical interactions between the membrane and chromium (VI). The physical interactions occur by electrostatic force between positive charge of the chromium (VI) and the opposite charge on the membrane in which case are oxygen atoms. Statements regarding the existence of the membrane filtration with adsorption function is also reinforced in the research on PSf ultrafiltration membrane in addition to get filtration function also has adsorption role. Adsorption role is caused due to a physical interaction between the surface membrane with the feed solution, which in that research is protein. Besides that, larger pore size would cause K₂Cr₂O₇ component that successful rejected lower, and vice versa, smaller pore sizes would cause the membrane selectivity against the KCr₂O₇ feed solution becomes higher.

Mechanical strength of PSf membrane

The mechanical strength gave the magnitude of stress and strain on PSf membrane. The magnitude of mechanical strength that belongs to PSf membrane measured by young's modulus, that is the ratio of the stress to the strain. The mechanical strength data of the PSf membrane, can be observed in table 3.

Table 3. Young's Modulus of PSf membrane

Casting Solution Composition (%b/b)	Non Solvent Composition (H ₂ O/CH ₃ OH) (%v/v)	Young's Modulus (N/m ²)
12/84/4	100/0	2458.89
16/84/0	50/50	3427.05

The data showed that the casting solution composition affect the mechanical strength of the PSf membrane, where the membrane with the largest quantity of PSf, will tend to form a tighter space between the particles. This caused a membrane having a smaller pore size and a higher mechanical strength. Likewise, the membrane with the smallest quantity of PSf, will tend to form the larger space between the membrane particles and led to the lower mechanical strength of the membrane.

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The surface and cross section morphology of the PSf membrane

Based on the results of SEM analysis in figure 8, PSf membrane with casting solution composition of PSf/NMP/PEG (%w/w) 12/84/4 and non-solvent composition of H₂O/CH₃OH (%v/v) 100/0, as a larger pore size, compared with membrane with casting solution composition of PSf/NMP/PEG (%w/w) 16/84/0 and non-solvent composition of H₂O/CH₃OH (%v/v) 50/50, has a smaller pore size. But unfortunately, despite having obtained the surface morphology characteristic of the membrane, but not significantly observed the formation of the asymmetry structure in the polysulfone membrane that formed.

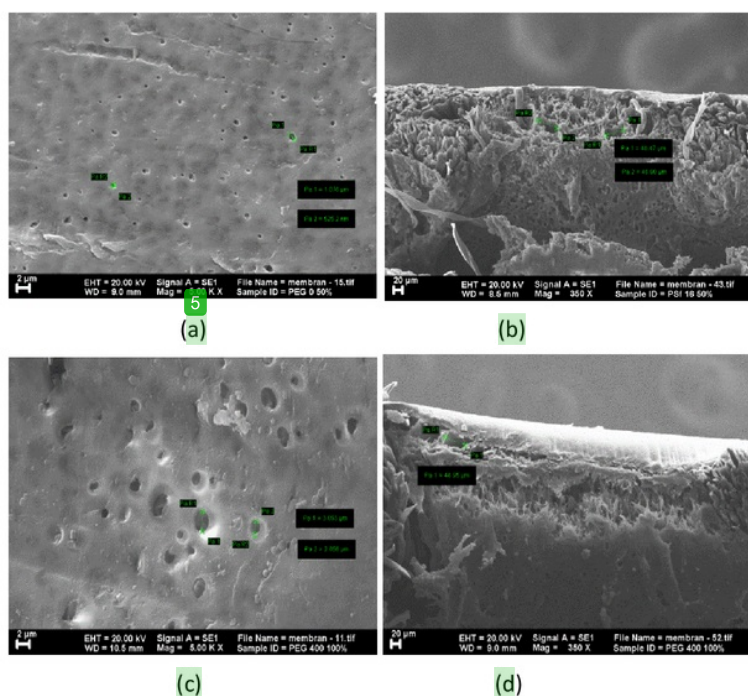


Figure 8. The surface morphology of the PSf membrane from 12%PSf/84%NMP/4%PEG (a) and 16%PSf/84%NMP (c); the cross section morphology of the PSf membrane from 100% H_2O (b) and 50% H_2O /50% CH_3OH (d)

CONCLUSION

2 Based on the results of the data that has been obtained, the conclusion that can be drawn are: (1) casting solution and non solvent composition affect the permeability of PSf membrane, where the membrane flux obtained were in the range $8603.39 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ – $468,78 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$, (2) casting solution and non solvent composition affect the selectivity of PSf membrane, where the membrane rejection obtained were in the range 12.61% - 49.19; (3) the results of the mechanical strength test (Young's modulus) show that membrane with largest permeability (flux) has the smallest Young's modulus, which is 2458.89 N/m^2 and membrane with largest selectivity has the largest Young's modulus, which is 3427.05 N/m^2 ; (4) the imaging result of the surface morphology shows that the PSf membrane resulted from preparation using casting solution composition 12%PSf/84%NMP/4%PEG and non solvent composition 100% H_2O has larger pores than PSf membrane resulted from preparation using casting solution composition 16%PSf/ 84%NMP and non solvent composition 50% H_2O /50% CH_3OH , at the same magnification. But unfortunately, despite having obtained the surface morphology characteristic of the membrane, but not significantly observed the formation of the asymmetry structure in the polysulfone membrane that formed.

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