The Effect of Polymer Concentration on Flux Stability of Polysulfone Membrane

D Ariono[1]*, P T P Aryanti[2], S Subagjo[1] and I G Wenten[1]

[1]Chemical Engineering Dept., Institut Teknologi Bandung, Jl. Ganesha 10, Bandung 40132, Indonesia
[2]Chemical Engineering Dept., Jenderal Achmad Yani University, Jl. Terusan Jendral Sudirman, Po Box 148, Cimahi, West Java, Indonesia

The influence of various polymer concentrations on flux stability of polysulfone membranes was investigated. The polysulfone membrane was prepared by blending polysulfone in DMAc with 25%wt concentration of PEG400 and 4% wt concentration of acetone. It was found that the pure water flux was sharply decreased from 1230 to 7 $Lm^{-2}h^{-1}$, when the polysulfone concentration was increased from 14% to 24%wt. Furthermore, the increase of polysulfone concentration also affects the fouling behavior of the membranes, in which almost of 90% of FRR was achieved by the addition of 18 %wt of polysulfone concentration. It was suggested that fouling formed on the membrane surface was dominated by reversible fouling, thus it could be easily cleaned by flushing method. In addition, the applied transmembrane pressure (TMP) also plays an important role in fouling behavior of polysulfone membrane. It was observed that irreversible fouling of organic matter was deteriorated by the increase of TMP, which contributed to the reduction of water flux. More stable membrane flux performance was achieved although it was operated at high TMP, when 20% wt concentration of polysulfone was added into membrane solution.

Introduction

Ultrafiltration (UF) membranes have been challenged to maintain their flux stability and selectivity. In water treatment field, the ultrafiltration membrane is severe to flux decline due to accumulation of natural organic matter (NOM) on the membrane surface or in the membrane structure, which known as fouling phenomenon [1, 2]. The development of low fouling ultrafiltration membrane has significant growth as a promising approach to maintain its stability for a long term of membrane operation. Aryanti et al.[3] modified PSf membrane by adding PEG400 and a low concentration of acetone into the casting membrane solution. The influence of the PEG400 concentration on the membrane morphology and selectivity was studied. Their experimental results showed that more than 80% humic substance was achieved when 4%wt of acetone and 25% wt of PEG400 were added into membrane solution. Furthermore, the tight structure in the membrane skin layer due to the rapid loss of acetone reduced the interaction between the membrane surface and the organic matter, which therefore could be easily removed by the cross-flow

*Author for Correspondence
E-mail: igw@che.itb.ac.id

mode of filtration and resulted in stable flux during five (5) hours of peat water filtration. The UF membrane properties can also be affected by polymer concentration in the casting membrane solution that controls the formation of membrane structure [4, 5]. Higher concentration of polymer contributes to kinetic hindrance against phase separation due to the rise of membrane solution viscosity. As a result, macrovoid formations in the membrane structure can be eluded [6]. Most of commercial UF membrane is prepared from hydrophobic polymers, such as polysulfone, which is susceptible to organic matter fouling due to its natural hydrophobic property [7, 8]. Therefore, most of membrane modification methods are focused on improving membrane hydrophilicity [9-12]. Blending with hydrophilic polymers has been widely used to accomplish this task as it is also the simplest method. Aryanti et al[3, 13] blended 35%wt of PEG400 into polysulfone membrane solution and found that the pure water flux was improved to 20 times compared with the unmodified membrane. However, the presence of high concentration of PEG400 induced the formation of open pore in membrane structure that reduced the membrane selectivity. In the present work, the effect of polysulfone concentration on fouling and rejection of

the resulted UF membranes towards humic substances were investigated. The polysulfone membrane was prepared by blending polysulfone in DMAc with 25%wt concentration of PEG400 and 4%wt concentration of acetone. The polysulfone concentration was varied to investigate the influence of its concentration on the membrane performances. Further evaluation was performed using different transmembrane pressure for a better understanding of the fouling and rejection of humic substances during peat water filtration.

The main objective of this experiment was prepared low-pressure ultrafiltration membrane by simple blending method but does not sacrifice its humic substances rejection performance.

Experimental method

Materials

The polysulfone (UDEL-P3500 MB7) used in this paper was provided by Solvay Advanced polymer, meanwhile DMAc with 99.9% purity was used as solvent and supplied by Shangshai Jingsan Jingwei Chemical Co. Ltd. PEG400 and acetone were used as additives to improve the polysulfone membrane performances. Demineralized water was used as coagulant and peat water, with a pH less than 4, was taken from Pekanbaru river-Riau, Indonesia.

Preparation of polysulfone membrane

The flat-sheet UF membrane was prepared by immersion precipitation method. Polysulfone was dissolved into DMAc at room temperature and then mixed with 25% wt of PEG400 and 4%wt of acetone concentration as additives. The membrane solution was stirred in a closed stirred tank until homogenous and then left without stirring until no bubbles appeared. Afterward, the membrane solution was casted on a flat glass plate with 200 μm thickness and immediately immersed into coagulation bath filled with demineralized water. The compositions of casting membrane solution used in this experiment are shown in Table 1. The peat water filtration was operated in different transmembrane pressures, i.e. 10, 15 and 30 psig.

Flux and Selectivity Measurement

The configuration of our experimental set up is the same as our previous work[13], which performed in crossflow mode. Prior to the experiment circular flatsheet membrane was placed inside membrane module and rinsed by demineralized water for 30 minutes at 1 atm. The active surface area of each membrane is 45 cm^2. The pure water flux (PWF) was determined using demineralized water at a fixed pressure, followed by the peat water flux measurement. The peat filtration was operated for two (2) hours, where the flux measurement and sampling were conducted periodically every 20 minutes. Humic acid content in raw water and permeate solution were analyzed using UV/vis spectrophotometer with 254 wave length (λ=254 m) [14]. The PWF and peat water flux were calculated with the following equation:

$$J_{w1} = V / (A \times \Delta t) \qquad \qquad \dots (1)$$

Where J_{w1} is pure water flux (PWF) (Lm^{-2}h^{-1}), V is volume of permeate (m^3), Δt is permeation time (s), and A is membrane surface area (m^2). Peat water flux is mentioned as J_t that is measured at the first time of filtration (h) and calculated by the same equation as eq. (1). Meanwhile, the humic substances rejection (R,%) is calculated by following equation[7]:

$$R (\%) = [1 - (C_p/C_f)] \times 100\% \qquad \dots (2)$$

Where C_p and C_f are concentration of humic substances in permeate and feed solution.

After two (2) hours of peat water filtration, the flat sheet membrane was flushed by demineralized water for around 45 minutes. The pure water flux (PWF) of the cleaned membrane was measured and denoted as J_{W2}. Flux recovery ratio (FRR) was calculated by equation (3) to investigate the effect of organic matter on membrane fouling during peat water filtration[15].

$$FRR (\%) = (J_{w2}/J_{w1}) \times 100 \qquad \dots (3)$$

Flux loss due to reversible (r_r) and irreversible (r_{ir}) fouling in polysulfone membrane were also calculated. Reversible fouling (r_r) is formed by gel formation on membrane surface, which can be easily removed by flushing or backwash method. Meanwhile, irreversible fouling (r_{ir}) is pore blocking fouling that shall be removed by chemical cleaning. In this

Table 1 — Casting membrane composition

Membrane code	Casting membrane composition (%-wt)		
	PSF	PEG400	Acetone
PSf-1	14	25	4
PSf-2	16	25	4
PSf-3	18	25	4
PSf-4	20	25	4
PSf-5	22	25	4
PSf-6	24	25	4

research, both reversible and irreversible flux loss were evaluated by flushing method. The flux loss was calculated by the following equation[16]:

$$r_r = (J_{w2} - J_{w1}) / J_{w1} \qquad \qquad \dots (4)$$

$$r_{ir} = (J_{w1} - J_{w2}) / J_{w1} \qquad \qquad \dots (5)$$

Total flux loss (r_t) is the sum of r_r and r_{ir}, which indicates the total flux loss during peat water filtration.

Resistance Series in Model

The total resistance of fouled polysulfone membrane was calculated by the resistance-in-series model, as shown in eq (6).

$$J = \Delta P / (\mu \times R_t) = \Delta P / [\mu \times (R_m + R_f)] \qquad \dots (6)$$

ΔP is transmembrane pressure (atm), μ is viscosity of solution (atm.h), R_t is total resistance (m^{-1}), R_m is membrane resistance (m^{-1}), and R_f is fouling resistance (m^{-1}). The membrane resistance (R_m) was determined by filtering demineralized water through the membrane, then calculated by the following equation:

$$Rm = \Delta P / (\mu \times J_{w1}) \qquad \qquad \dots (7)$$

After two (2) hours of peat water filtration, the peat water flux was measured to calculate the total fouled membrane (R_{ft}). Then, R_f was determined by subtracting the membrane resistance (R_m) from the total fouling resistance (R_{ft}), as shown in Eq. (8).

$$R_f = R_{ft} - R_m \qquad \qquad \dots (8)$$

Result and Discussion

The effect of polysulfone concentration on membrane permeability and selectivity

The pure water flux (PWF) and humic substance rejection in various polysulfone concentration in membrane solution is shown in Fig. 1. This figure shows a trade-off between membrane selectivity and permeability that has been widely known as one of the drawbacks in polymeric membranes. The PWF is decreased with the increase of polysulfone concentration. It may be attributed to the formation of smaller membrane pore size in high polysulfone concentration. As the increase of polysulfone concentration, the membrane solution viscosity is enhanced, which inhibits the growth of membrane pore [17, 18]. Consequently, it contributes to the reduction of PWF. In contrast, humic substances rejection of the membrane is raised

with the addition of higher concentration of polysulfone. In low polysulfone concentration, a larger pore size has been formed, thus it easily passes through the membrane and leads to low rejection of humic substances. During the peat water filtration, rejection of humic substances is not only affected by the membrane pore size. The accumulation of humic substances on the polysulfone membrane is also attributed to hydrophobic interaction between the membrane surface and humic substances molecules[19,20]. The increase of polysulfone concentration improves the membrane hydrophobicity, which then escalates the humic substances accumulation on the membrane surface. In addition, low pH of peat solution also exacerbates membrane fouling since it enhances the interaction between membrane and molecules due to increase in solutes hydrophobicity. The negative charge of phenolic and carboxylic functional groups is neutralized with the decrease of pH, which leads to lower water solubility and greater hydrophobicity [21, 22]. The accumulation of humic substances on the membrane surface can serve as a second layer, which influences the membrane selectivity.

Fouling of humic substances analysis in different polysulfone concentration

The effect of polysulfone concentration on relative water flux at constant transmembrane pressure is shown in Fig. 2a. This relative flux measurement is performed to investigate the fouling behavior on the membrane surface that responsible to flux decline during the peat water filtration. It shows that the most severe flux decline phenomenon occurs when 14%wt of polysulfone concentration is blended into membrane solution. In low concentration of polysulfone, the rapid adsorption of humic substances on the membrane surface may be attributed to the larger pore
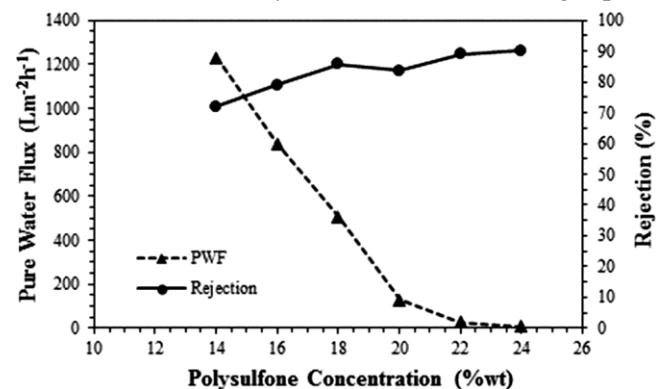

Fig.1 — The effect of polysulfone on Rejection of humic substances and pure water flux at a constant transmembrane pressure (15 Psig)
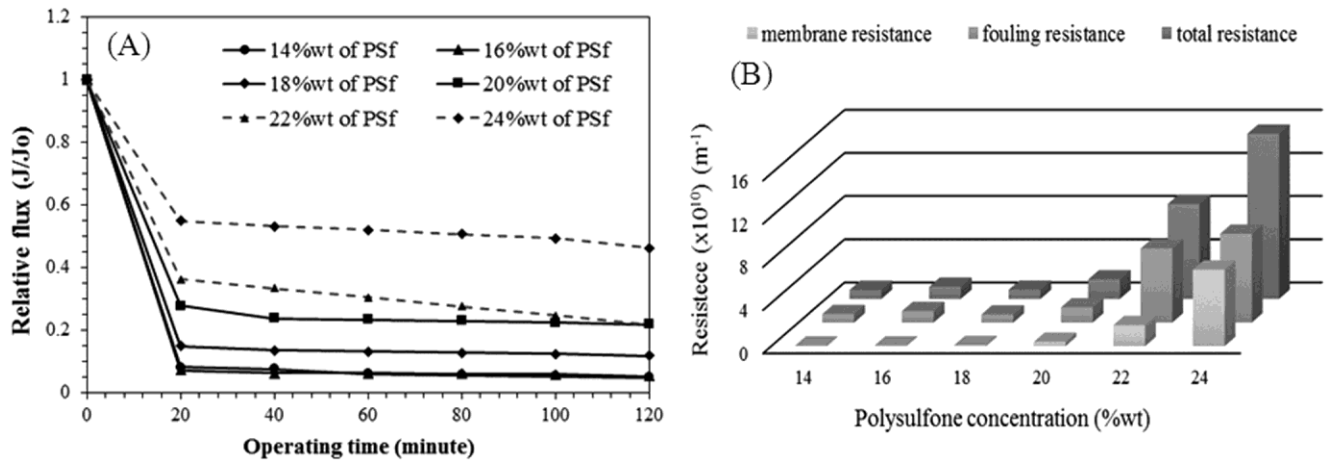
Fig. 2 — The effect of polysulfone concentration on humic substances fouling: (A) profile of peat water flux and (B) total resistance of fouled polysulfone membranes

formed in membrane skin layer. Furthermore, drag permeation during cross-flow filtration deteriorates the fouling formation in membrane structure, which contributes to the extreme flux decline at 20 minutes of filtration time. This rapid flux decline could be minimized by adding the polysulfone concentration above 16%wt. Further increase of polysulfone concentration, a slight decline of flux is observed. Although the number of humic substance that accumulates on the membrane surface enhances at higher polysulfone concentration, weak interaction between the humic substances and the membrane is resulted, thus it can easily be cleaned by the cross-flow filtration. The optimum stable flux is achieved by blended 20%wt of polysulfone concentration. Fig. 2b exhibits the effect of polysulfone concentration on total resistance of the fouled membrane during two (2) hours of peat water filtration. The increase of polymer concentration enhances the viscosity of the casting membrane solution and a higher crystallization propensity was expected, which contributes to the formation of a thick skin layer and reduces the membrane water flux[23]. Furthermore, the increase of polysulfone concentration raises the hydrophobicity of the membrane, thus it enhances the adsorption of humic substances on the membrane surface due to hydrophobic interaction [7]. Therefore, fouling resistance of the membrane is increased and causes greater loss towards membrane flux. The influence of polysulfone concentration on both flux recovery ratio (FRR) and flux loss after two (2) hours of peat water filtration is presented in Table 2. FRR of the membrane is increased up to 89% when 20 % wt of polysulfone concentration is added into membrane solution.

Table 2 — The effect of polysulfone concentration on flux recovery ratio and flux loss

Membrane Code	FRR (%)	Total Flux loss	Reversible flux loss (rir)	Irreversible flux loss (rr)
PSf-1	13,325	0,951	0,084	0,867
PSf-2	16,129	0,947	0,108	0,839
PSf-3	25,000	0,881	0,131	0,750
PSf-4	89,189	0,781	0,673	0,108
PSf-5	88,338	0,543	0,426	0,117
PSf-6	88,824	0,465	0,368	0,097

Note: all experiments are conducted in a constant pressure (15 psig)

However, this tendency is limited since further addition of polysulfone in membrane solution leads to slight decrease of FRR before it reaches its stationary point. Overall performance evaluation shows that higher concentration of polysulfone gives antifouling property to the membrane as clearly shown by lower flux loss. Regarding membrane flux loss, both reversible and irreversible fouling of humic substances plays an important role. As shown in Table 2 that higher concentration of polysulfone, which resulted in smaller membrane pore size, minimizes the irreversible fouling in the membrane structure. It is found that the irreversible fouling can be reduced from 0,867 to 0,097, when the polysulfone concentration is increased from 14 to 24%wt. However, the decrease of irreversible fouling is not quite apparent towards reversible fouling. It shows that the addition of polysulfone concentration at above 20%wt results in the decrease of reversible fouling. Even though the accumulation of foulant on membrane surface is increased, the interaction between the membrane and the foulant is weak and reversible. It then means that initial

membrane performance can be well recovered by simple cleaning method, such as through flushing or backwashing method.

Fouling and rejection of humic substances in different transmembrane pressure

It has been known that fouling on a membrane surface alters the separation characteristic of the membrane[24,25]. Low cross flow velocity and transmembrane pressure lead to higher concentration of the rejected solute on the membrane surface, due to the longer-time of membrane exposure to the raw water. Therefore, higher flux decline of membrane water flux during the peat filtration process can occur. In this sub-chapter, the effect of transmembrane pressure on polysulfone membrane performance is discussed. Fig. 3a shows the relative flux of polysulfone membranes and rejection of humic substances at various concentration of polysulfone and TMP. It is found that very low relative flux is found at low concentration of polysulfone. Beside of the larger pore in the membrane skin layer, fouling of organic matter in membrane structure deteriorates by the increase of TMP. As the increase of TMP, cross-flow velocity on the membrane surface has enhanced that impact to the increase of drag permeation rate of

solute through the membrane. Therefore, lower rejection of humic substances is obtained at higher TMP. Beside of the drag permeation effect, low rejection of humic substances could also be attributed to the presence of PEG400 in the membrane solution, which leads to the formation of open pore in the membrane skin layer. The optimum level of humic substances rejection is obtained at 18%wt concentration of polysulfone. It may be attributed to the uniform pore size distribution of the resulted membrane compared with other compositions. In the other hand, low concentration of polysulfone results in a weak mechanical resistance of the membrane, which indicated by the damage of resulted membrane at 30 psig. More stable membrane performance is found at 20% wt concentration of polysulfone, although it is operated at high TMP. Very tight pore structure is formed by further increasing of the polysulfone concentration, which could not be operated in lower operating pressure than 15 psig. The influence of transmembrane pressure on total resistance of the fouled polysulfone membrane is displayed in Fig. 3b. It has been explained that the increase of polysulfone concentration improves the resulted membrane hydrophobicity, which leads to the enhancement of humic substance interaction with the membrane surface and results in higher fouling resistance[26]. By increasing the transmembrane pressure, the total resistance of the fouled membrane is increased. In this case, higher drag permeation effect plays an important role in increasing the fouling resistance of the membrane. at 24%wt of polysulfone concentration, the total resistance seems slight decrease as the increase in transmembrane pressure. Improvement in fouling resistance may be attributed to the tight skin layer formation due to high concentration of polysulfone, which is mainly dominated by reversible fouling. Flux loss of the polysulfone membranes at various transmembrane pressures is shown in Table 3. As the transmembrane pressure is raised, flux loss of the membranes during two (2) hours of peat water filtration becomes higher. It suggests that higher transmembrane pressure
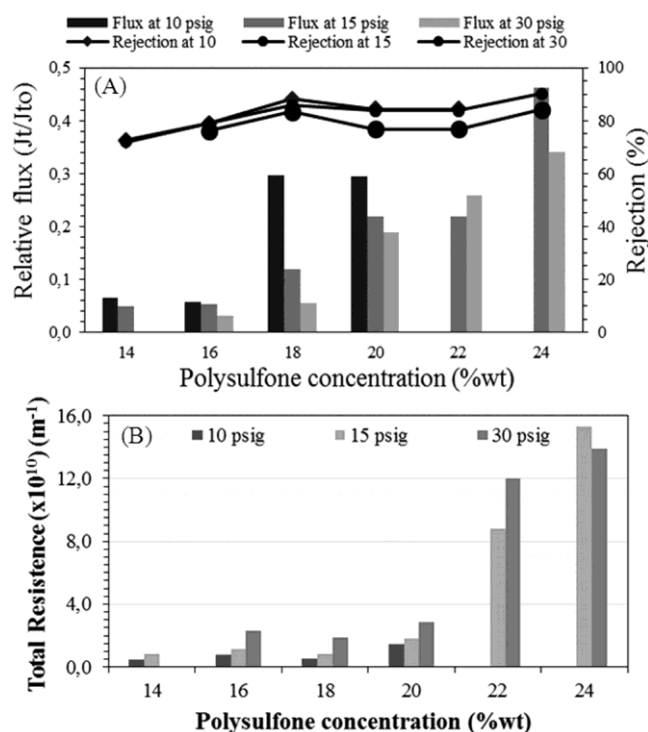

Fig. 3 — The effect of polysulfone concentration on rejection and fouling of humic substances in different transmembrane pressure: (A) Humic substance rejection and (B) Total resistance of polysulfone membranes during two (2) hours of peat water filtration

Table 3 — Flux loss during two (2) hours of peat water filtration in different transmembrane pressure

TMP (psig)	Flux loss (%)					
	PSf-1	PSf-2	PSf-3	PSf-4	PSf-5	PSf-6
10	94	94	70	71	-	-
15	95	95	88	78	54	45
30	-	97	94	81	66	55

enhances the accumulated solutes on the membrane surface, which increase the fouling resistance on the polysulfone membrane [27]. However, the insignificant increase of flux loss during peat water filtration is found with the increase of transmembrane pressure.

Conclusions

Fouling and rejection of humic substances in various composition of polysulfone membrane and transmembrane pressure have been investigated. It is found that the increase of polysulfone concentration in membrane solution reduces the pure water flux of the membrane due to smaller pore size formation that attributed by the enhancement of kinetic hindrance of the membrane solution. The pure water flux (PWF) significantly decreases from 1230 to 7 $Lm^{-2}h^{-1}$, when the polysulfone concentration is increased from 14% to 24%wt. However, the tight membrane pore size contributes to higher humic substances rejection. More than 80% rejection of humic substances is achieved when 18%wt of polysulfone concentration is added into membrane solution. The increase of polysulfone concentration also affects fouling behavior of the membranes. High concentration of polysulfone leads to the improvement of membrane hydrophobicity. The fouling formed on the membrane surface is dominated by reversible fouling that could be easily cleaned by flushing method and results in higher flux recovery ratio (FRR) of the membrane after two (2) hours of peat water filtration. Almost of 90% of FRR is achieved by the addition of 18%wt of polysulfone concentration. Further addition of polysulfone in membrane solution leads to lower FRR before it reaches its stationary point. The applied transmembrane pressure (TMP) during peat water filtration also plays an important role on fouling behavior of polysulfone membrane. It is observed that irreversible fouling of organic matter is deteriorated by the increase of TMP from 15 to 30 psig due to drag permeation effect. More stable membrane flux performance is found at 20% wt concentration of polysulfone, although it is operated at high TMP.

Abbreviation

μ	Viscosity of solution (atm.h)
ΔP	Transmembrane pressure (atm)
Δt	Permeation time (s)
A	Membrane surface area (m^2)
C_f	Humic substance concentration in feed
C_p	Humic substance concentration in permeate
FRR	Flux Recovery Ratio
J_t	Filtration time (h)
J_{w1}	Pure water flux (PWF) ($Lm^{-2}h^{-1}$)
J_{w2}	Pure water flux of the cleaned membrane ($Lm^{-2}h^{-1}$)
NOM	Natural organic matter
PEG	Polyethylene glycol
PWF	Pure water flux ($Lm^{-2}h^{-1}$)
R	Rejection (%)
R_f	Fouling resistance (m^{-1})
r_{ir}	Flux loss due to irreversible fouling
R_m	Membrane resistance (m^{-1})
r_r	Flux loss due to reversible fouling
r_t	Total flux loss
R_t	Total resistance (m^{-1})
TMP	Transmembrane pressure
V	Volume of permeate (m^3)

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