



Influence of Additives on Polysulfone-Based Ultrafiltration Membrane Performance during Peat Water Filtration

Putu Teta Prihartini Aryanti, Khoiruddin, I Gede Wenten*

Department of Chemical Engineering, ITB, Jl. Ganesha 10, Bandung 40132, Indonesia

ABSTRACT

The effect of additives on polysulfone membrane performance in peat water filtration was investigated. Polysulfone membrane was prepared by immersion precipitation from quaternary membrane systems containing N,N-dimethylacetamide (DMAc) as solvent, polyethylene glycol (PEG) and acetone as additives. The performance of membrane was evaluated by pure water flux and humic acid rejection. Results show that rejection of humic acid in peat water increased with the increase of acetone concentration. The maximum rejection was achieved at 8% wt acetone concentration. The presence of acetone contributed to the tight skin layer formation of polysulfone membrane. Other results suggest that membrane hydrophilicity can be improved by increasing of PEG concentration in membrane matrix, which was indicated by the increase of water flux around 127 LMH at the presence 25% wt of PEG. In high PEG concentration, the humic acid rejection was decreased which may be attributed to more open pore formed in the skin layer of the membrane and leads to a more severe irreversible fouling.

Keywords: Polysulphone membrane; hydrophilicity; fouling; humic substances

1. INTRODUCTION

Polysulfone ultrafiltration membranes have been widely applied in various industrial fields, especially for water and wastewater treatment. Polysulfone as membrane material is preferred due to its several advantages, such as good mechanical properties, high temperatures and chemicals resistance, processing easiness, and its wide availability in the market (Tweddle et al., 1983). However, the nature of the hydrophobic polysulfone membrane leads to low water flux and prone to fouling of organic compounds. In order to overcome this problem, further research of polysulfone membrane has been conducted to increase hydrophilicity of polysulfone membrane, thus further reduce the tendency of membrane fouling (Jucker and Clark, 1994; Ma et al., 2001).

Humic substance, an organic compound, is the major foulant during water treatment process, which gives the waters a yellowish to brownish color (Wenten and Widiasa, 1998). Peat water, where is usually found at dark-brown colour, consists of highly concentrated dissolved organic matter, particularly humic substances and other acidic compound. During the peat water treatment using ultrafiltration membrane, fouling becomes major problems that should be minimized. The main factor that influenced the membrane fouling mechanism is interaction between organic compounds and hydrophobic membrane surface, which could lead to significant reduction of membrane performance (Nghiem and Hawkes, 2009). Aside from membrane mate-

*Corresponding to: igw@che.itb.ac.id

rial, fouling tendency is also influenced by membrane pore structure and surface roughness, where larger pore size could lead to pore constriction up to complete blocking, while a tight membrane tends to form a cake layer (Costa et al., 2006; Mousa, 2007).

Blending method with additive is a simple widely used method in polysulfone membrane modification. The effects of additives concentration and molecular weight on the membrane structure and morphology have been reported by several researchers (Han and Nam, 2002; Hou et al., 1991; Khayet et al., 2002; Kim et al., 2003; Zheng et al., 2006). Commonly used additives for this method are hydrophilic polymer, such as polyvinyl - pyrrolidone (PVP) (Chakrabarty et al., 2008b; Matsuyama et al., 2003) or PEG (Chakrabarty et al., 2008a; Kim and Lee, 1998; Ma et al., 2011), which has high hydrophilic properties and is easily dissolved in water. The presence of additive in membrane solution influences thermodynamic and kinetic properties of membrane solution. It reduces the strength of polymer-solvent interaction and increases solvent-non solvent exchange rate, thus the precipitation rate of membrane could be enhanced. The effect of additives as pore former on membrane structure has been investigated. Enlarged or suppressed macrovoid can be obtained by varying additive concentration and molecular weight. Ma et al. (2011) found that an increase of PEG 400 additive in membrane solution between 2% - 8% wt increased the number and size of finger-like pores. The existence of PEG leads to higher polymer concentration and increases the viscosity of polymer, thus membrane solution becomes less stable. Due to hydrophilic properties of PEG, the increase of PEG concentration increases the inflow rate of water diffusion in the polymer solution, which results in the more developed of lean phase growth and allows the possibility of pore coalescence. They also found that the increase of PEG concentration

also increases porosity and water permeability of the membrane. High polymer concentration in membrane solution leads to delayed demixing, which gives an opportunity for nucleation and growth mechanism of new pore in membrane structure.

Han et al. (2002) investigated the effect of PVP additive concentration on correlation between thermodynamic and rheological properties of polysulfone membrane. At low PVP concentration, instant demixing occurred and contributed to enlargement of membrane pore. However, membrane prepared with high concentration of PVP, i.e 20% wt, showed a delay demixing due to the increase of polymer solution viscosity and suppressed the macrovoid formation in membrane structure. Lee et al. (2003) suggested that this reversibility indicates a trade-off between thermodynamic enhancement and kinetic hindrance. It means that PVP fails to induce thermodynamic enhancement at high concentration due to kinetic hindrance induced by high viscosity of membrane solution. Matsuyama et al. (2003) reported that the increase of molecular weight of PVP led to formation of a sponge like structure. The same tendency was also found when using PEG as an additive (Chakrabarty et al., 2008a).

Membrane performance can also be modified by the addition of volatile solvent into a polymeric membrane solution. By adding co-solvent, macrovoid formation in membrane structure can be eliminated, where the morphology is altered from finger-like to sponge-like structure (Aroon et al., 2010). When non solvent is used as additive, the phase separation of membrane casting solution and coagulation process can be accelerated during immersion in coagulation bath. Polymer solidification can occur rapidly at the membrane surface, where membrane with thinner skin layer can be formed. Wang et al. (1996) used alcohol as non solvent additives (NSA), such as methanol, ethanol, 1-propanol, 1-butanol

and 1-pentanol, in polyethersulfone (PES)/NMP system. They observed that addition of NSA reduced the coagulation value and lowered the intrinsic viscosity of polymer solution. Acetone is weak non-solvent that can be used to modify polysulfone membrane. Barth et al. (2000) prepared polysulfone and polyethersulfone membrane from quaternary mixture containing solvent (DMF) and weak non solvent acetone as additive. They found that membrane prepared with 3% acetone consisted of dense coating layer with small pores on the top of membrane structure. As volatile additive, acetone will be lost rapidly by evaporation from the casting solution before immersed in coagulation bath which results in higher polymer concentration in the top layer of membrane and forms denser coating layer. Kim et al. (2003) found that increasing acetone additive will decrease pore size and increase rejection of the solute (PEG 35000). Idris and Ahmed (2006) calculated that besides improving membrane performance, the presence of acetone in polymeric membrane also reduced the production cost of membrane, where the addition of acetone can reduce the use of expensive solvent that common used in membrane preparation.

It has been notified that phase separation of membrane solution could be accelerated by the presence of additives in membrane solution. The addition of additives changes the condition of membrane solution close to the liquid-liquid demixing boundary and reduces the degree of solubility between polymer and solvent (Reuvers et al., 1987). Consequently, less non-solvent concentration is needed to reach phase separation condition during immersion process in coagulation bath. To obtain a fine membrane structure, membrane casting solution must be prepared in equilibrium and stable condition prior to casting process. Principally, liquid-liquid phase separation is indicated by two fully separated layers or turbidity change in the membrane solution. In the present work,

the influence of acetone concentration as non-solvent additive on membrane performance was investigated. The polysulfone membrane was prepared with the addition of PEG400 and various acetone concentrations as additives. The purpose of this research is to obtain high selectivity and low fouling of polysulfone ultrafiltration membrane. The performance of membranes was investigated by pure water flux and rejection of humic acid in peat water.

2. MATERIALS AND METHOD

2.1 Materials

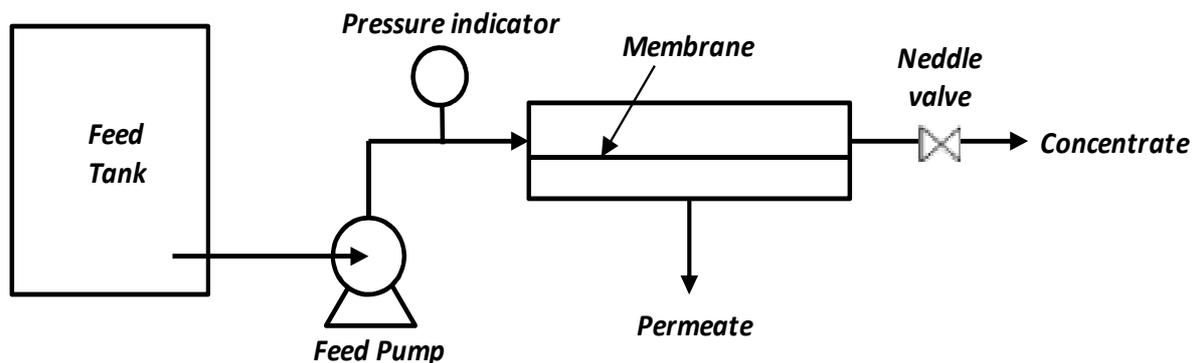
Polysulfone (UDEL-3500) was obtained from Solvay Advanced Polymer. N,N-dimethylacetamide (DMAc, 99.9% purity) was used as solvent, which was obtained from Shanghai Jingsan Jingwei Chemical CO., Ltd. Polyethylene glycol (PEG) 400 and Acetone (96% purity) were used as additives. Deionized water was used as non-solvent in the coagulation bath. Peat water was obtained from Pekanbaru river – Riau.

2.2 Membrane preparation

Flat sheet membrane was prepared by immersion precipitation method using deionized water as coagulation bath. Measured polysulfone polymer was dissolved in DMAc at room temperature mixed with PEG400 and acetone as additives. The membrane solution was stirred for 14 hours at 30°C. Concentrations of casting membrane solution varied as shown in Table 1. The membrane solution was casted on glass plate with 200 μ m thickness, then immediately immersed in deionized water for 5 hours until all of the solvent was completely evaporated. Finally, the membrane sheet was cut in circular form with 8 cm diameter, and placed inside flat sheet membrane module for flux and rejection measurement.

Table 1 Casting membrane composition

Membrane code	Casting membrane composition (%wt)			
	Polysulfone	PEG 400	Acetone	DMAc
PSf 1a	20	0	4	76
PSf 2a	20	5	4	71
PSf 3a	20	10	4	66
PSf 4a	20	15	4	61
PSf 5a	20	20	4	56
PSf 6a	20	25	4	51
PSf 1b	20	25	0	55
PSf 2b	20	25	2	53
PSf 3b	20	25	6	49
PSf 4b	20	25	8	47
PSf 5b	20	25	10	45

**Figure 1** Schematic diagram of flux measurement

2.3 Flux and rejection measurement

Flux and rejection measurement were performed in cross-flow filtration mode, where the set-up is shown in Figure 1. Flat sheet membrane was initially washed by deionized water prior to the experiment. Circular polysulfone flat sheet membrane cutting, which had active surface area of 45 cm², was prepared inside the membrane module. The module consists of one inlet for feed solution and two outlets for concentrate and permeates. The module was connected to a vessel, which

contained feed solution and pressure gauge was placed at the module inlet. Pure water flux was determined using deionized water as feed solution at different pressure (69, 103, and 207 kPa), followed by measurement of peat water flux where the membrane was operated for 2 hours. During the membrane operation, permeate was collected periodically (20, 40, 60, 90 and 120 minutes). Humic acid content in the permeate solution was analyzed using UV/Vis Spectrophotometer with 254 nm wavelength ($\lambda=254$ nm) (Imyim and Prapalimrungsi, 2010).

Pure water flux (PWF) is calculated by the following equation:

$$J_{w1} = \frac{V}{A \cdot \Delta t} \quad (1)$$

where J is PWF ($\text{m}^3/\text{m}^2 \cdot \text{s}$), V is volume of permeate (m^3), t is permeation time (s), and A is membrane surface area (m^2).

Humic acid rejection (R , %) is calculated by equation (2) (Baker, 2004) :

$$R = 1 - \left(\frac{C_p}{C_f} \right) \quad (2)$$

where C_p and C_f are concentration of humic acid in permeate and feed respectively.

After filtration of peat water, the flat sheet membrane was rinsed with deionized water for 45 minutes, then PWF of cleaned membrane was measured using equation (1) and mentioned as J_{w2} . Flux recovery ratio (FRR) was calculated by equation (3), as follows:

$$FRR(\%) = \left(\frac{J_{w2}}{J_{w1}} \right) \times 100 \quad (3)$$

Resistance of polysulfone membrane was also calculated. Reversible fouling (r_r) is formed by a polarization layer and gel formation, which can be easily eliminated by backwash or cross flow method. While irreversible fouling layer (r_{ir}) can be initially formed by pore blocking, which can be reduced by chemical cleaning. Reversible and irreversible can be calculated by the following equations:

$$r_r = \frac{J_{w2} - J_t}{J_{w1}} \quad (4)$$

$$r_{ir} = \frac{J_{w1} - J_{w2}}{J_{w1}} \quad (5)$$

where J_t is peat water flux. Total resistance (r_t)

is the sum of r_r and r_{ir} .

3. RESULT AND DISCUSSION

3.1 Effect of PEG concentration on membrane performance

In membrane preparation, PEG 400 was added into membrane solution in various concentrations as hydrophilic additive in order to increase membrane hydrophilicity and suppress macrovoid occurrence, despite act as pore performer agent (Ma et al., 2011). The effect of PEG 400 additive to membrane performance was investigated and the experimental result is shown in Table 2. Both pure water flux (PWF) and peat water flux were increased by increasing of PEG concentration in membrane solution. It is believed that the water flux increase is caused by the increase of hydrophilicity of membrane due to the addition of hydrophilic material. The PWF reached the maximum value at $3.53 \times 10^{-5} \text{ m}^3/\text{m}^2 \cdot \text{s}$. Meanwhile in the absence of PEG additive in membrane solution, the obtained membrane structure was very dense, indicated by PWF around $8 \times 10^{-7} \text{ m}^3/\text{m}^2 \cdot \text{s}$ at 344 kPa.

Flux loss is predominantly caused by solute adsorption and gel layer formation on membrane surface (Figure 2). It has been notified that the nature properties of polysulfone membrane is hydrophobic, which is prone to fouling by different fractions of natural organic matter in raw water. This fouling phenomenon could be reduced by reducing skin membrane pore size and hydrophilicity of the membrane. Increasing membrane hydrophilicity by addition of PEG reduces the interaction between organic solute and membrane surface. Thus, membrane fouling can be minimized and easier to remove by periodic cleaning. Antifouling performance of polysulfone membrane was characterized by measuring water flux recovery ratio (FRR). As indicated in Table 2 FRR was increased by increasing PEG concentra-

tion. It means that the filtration performance of the prepared polysulfone membrane was enhanced by the presence of PEG additive. At 25% wt concentration of PEG, hydrophilicity seemed to decrease which was indicated by the

decrease of reflux ratio. It may caused by the increase of irreversible fouling in membrane structure due to more open pore formed in the skin layer of the membrane.

Table 2 Effect of PEG 400 concentration in casting membrane solution to pure water flux

Membrane Code *	PWF**, J _{w1} ($\times 10^{-5}$ m ³ /m ² ·s)	Peat Water Flux after 1 hour filtration **, J _t ($\times 10^{-5}$ m ³ /m ² ·s)	FRR (%)	R _t	rr	rir
PSf 1a	0.00	0.00	-	-	-	-
PSf 2a	0.58	0.18	95.261	0.688	0.641	0.047
PSf 3a	1.12	0.36	97.584	0.681	0.657	0.024
PSf 4a	1.68	0.54	98.026	0.679	0.660	0.020
PSf 5a	2.24	0.72	98.114	0.679	0.660	0.019
PSf 6a	3.52	0.82	89.189	0.766	0.658	0.108

*) contain 20% wt of polysulfone and 4% wt of acetone

***) measured at 103 kPa

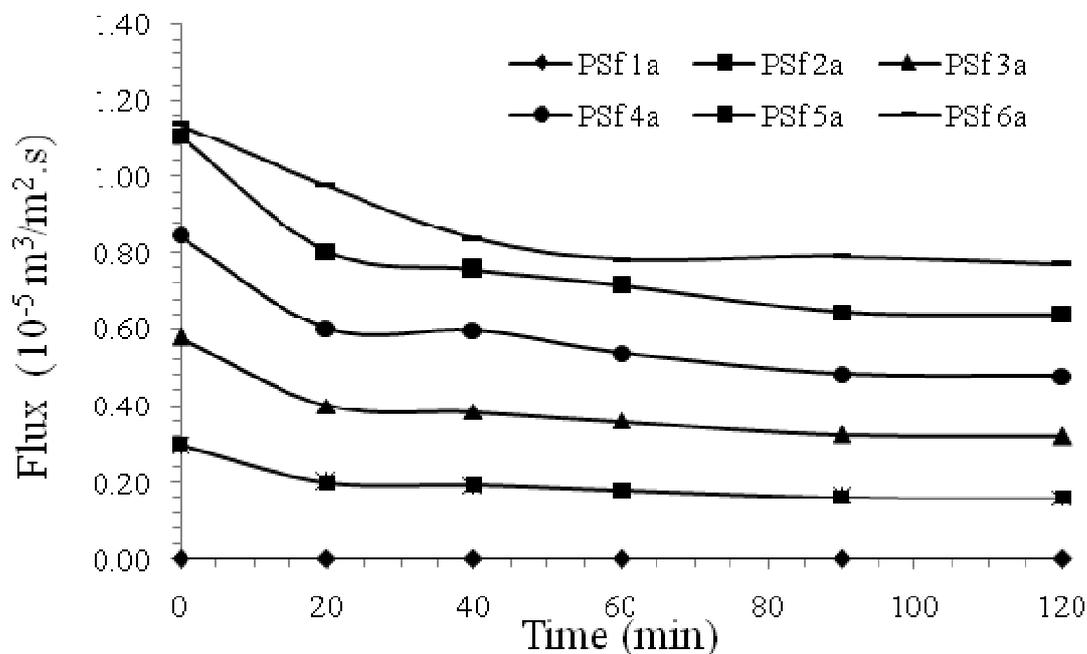


Figure 2 Flux profile during peat water filtration period at 103 kPa

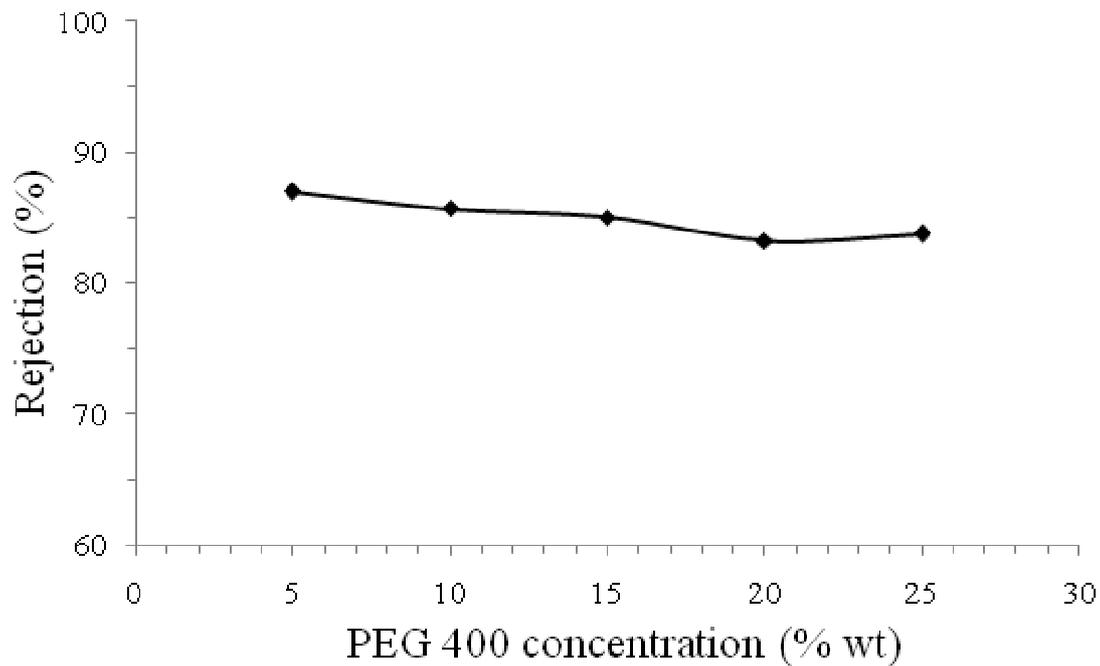


Figure 3 Effect of PEG 400 concentration in casting membrane solution (contain 20% wt polysulfone and 4% acetone) to humic acid rejection

Figure 3 shows that increasing PEG concentration decreases rejection of the humic acid. It may be caused by the increase of pore size in the skin membrane (Kim et al., 1998). The increase of pore formation in the skin membrane, due to high PEG concentration, leads the more severe irreversible fouling (Kim et al., 1998). However, reversible fouling was still dominant in total fouling on the membrane due to tight skin layer of the membrane, which can be eliminated by backwash cleaning.

3.2 Effect of acetone concentration on membrane performance

Effect of acetone to the membrane permeability and humic acid rejection is shown in Table 3 and Figure 4. It can be seen that the increase of acetone concentration in membrane solution decreased the pure water flux. As explained previously acetone concentration in membrane solution induces tight skin layer formation in the top layer of the membrane and suppresses macrovoid in membrane structure. Acetone is

categorized as weak co-solvent (Aroon et al., 2010), thus the presence of acetone could reduce the interaction between solvent (DMAc) and polysulfone. Due to high volatility combined with good interaction with water, acetone could be easily separated and lost from membrane solution. The rapidly lost of acetone results in higher polymer concentration in skin layer membrane and produce a thicker tight skin membrane.

The significant flux decline could also be induced by organic matter fouling on membrane surface. The increase of acetone concentration reduces total resistance, which means that the antifouling performance of polysulfone membrane was improved. Increasing acetone concentration also contributed to the increase of FRR value. It is caused by tight skin layer formation which prevents the membrane from irreversible fouling. On the contrary in the absence of acetone in membrane solution, the irreversible fouling was found which is attributed to the more open

pore formation in membrane skin layer and membrane structure.

Increasing tight skin layer thickness due to the presence of acetone has a significant effect to humic acid rejection (Figure 5). Membrane skin layer plays a very important role in

membrane selectivity. Highest rejection was obtained at 8% wt of acetone. Further increase of acetone concentration to 10% wt, the composition crossed the liquid-liquid demixing boundary prior to the casting process.

Table 3 Effect of acetone concentration in casting membrane solution to pure water flux

Membrane Code *	PWF **, J_{w1} ($\times 10^{-5} \text{ m}^3/\text{m}^2 \cdot \text{s}$)	Peat Water Flux after 1 hour filtration **, J_t ($\times 10^{-5} \text{ m}^3/\text{m}^2 \cdot \text{s}$)	FRR (%)	rt	rr	rir
PSf 1b	7.57	2.05	34.043	0.729	0.069	0.660
PSf 2b	5.95	1.56	47.098	0.738	0.209	0.529
PSf 6a	3.52	0.78	85.986	0.778	0.638	0.140
PSf 4b	2.88	0.78	87.788	0.819	0.638	0.122
PSf 5b	1.60	0.43	81.618	0.732	0.548	0.184
PSf 6b	demixing	-	-	-	-	-

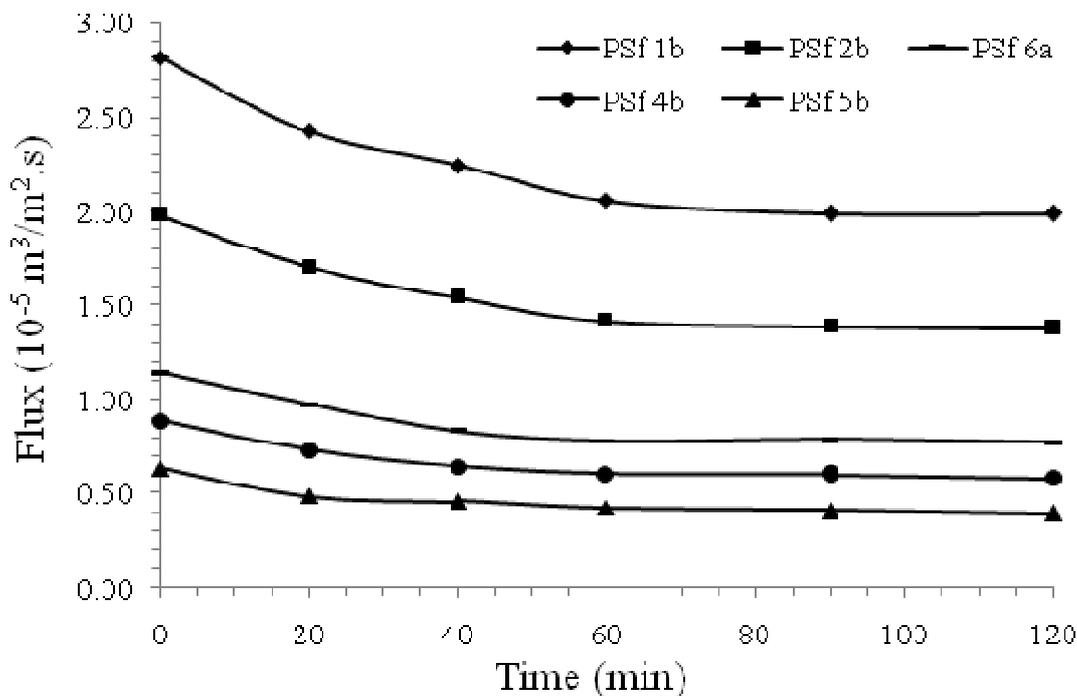


Figure 4 Flux profile during peat water filtration period at 103 kPa

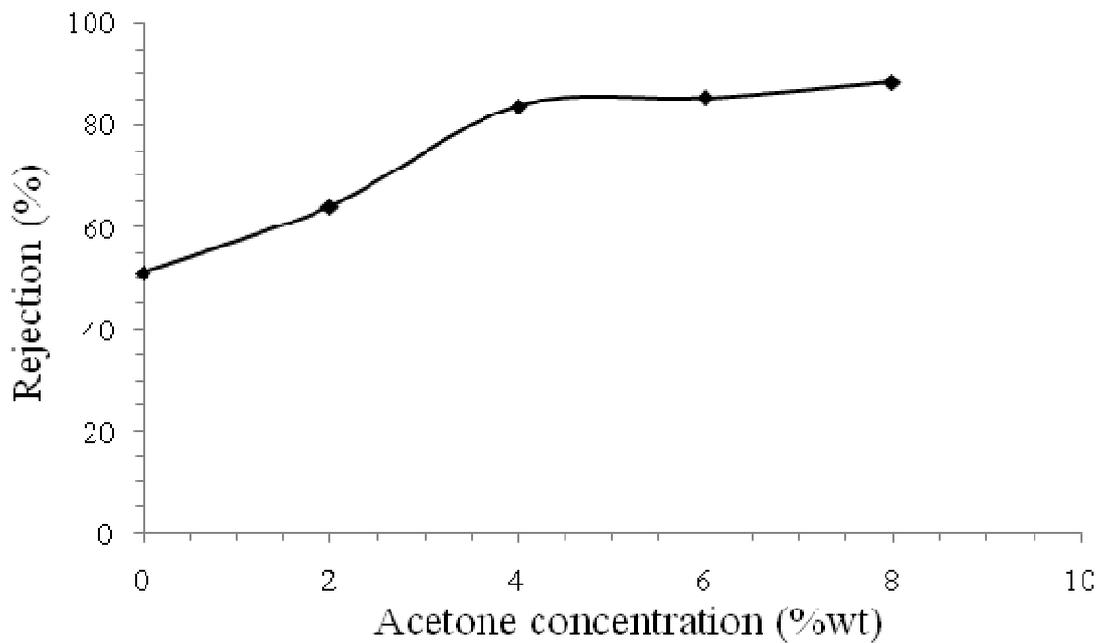


Figure 5 Effect of acetone concentration in casting membrane solution (contain 20% wt polysulfone and 25% wt PEG 400) to humic acid rejection

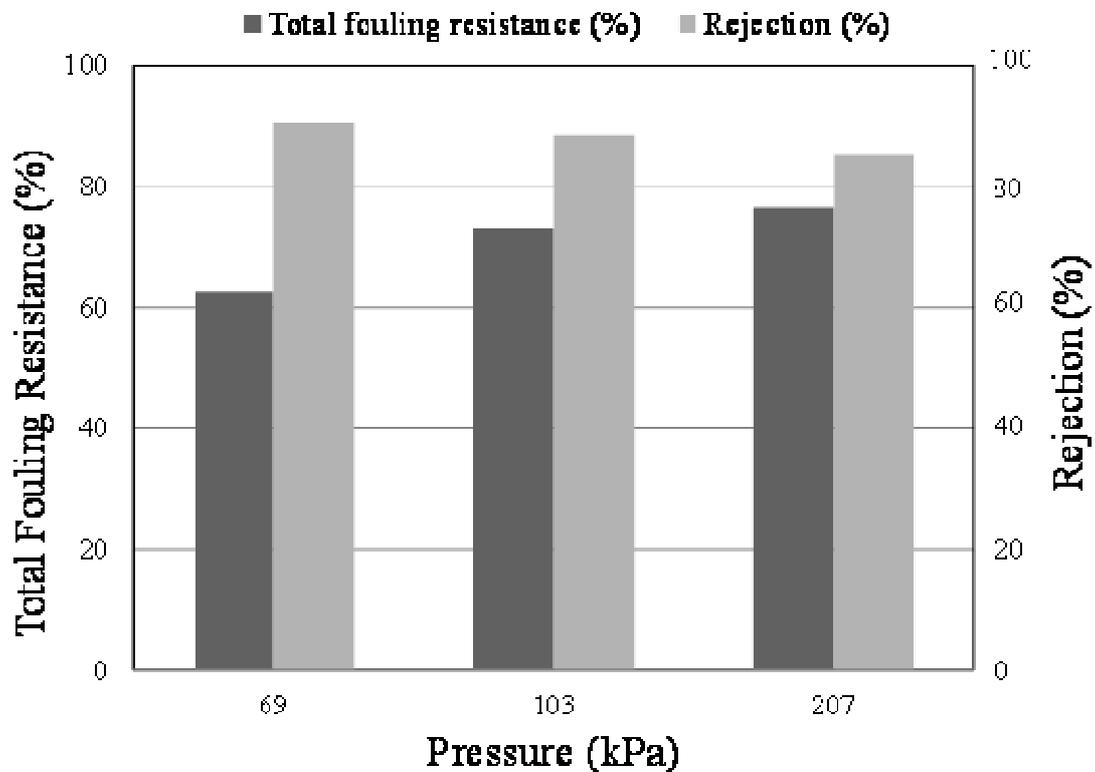


Figure 6 Effect of pressure operation to humic acid rejection using membrane PSf 6a

3.3 Effect of pressure condition on peat water flux and humic acid rejection

The rejection of solutes present in peat water during ultrafiltration process is influenced by several phenomena (Chen et al., 1997; Van Oers et al., 1995). Low velocity and higher pressure lead to the increase of concentration polarization on membrane surface, due to the increase of membrane exposure to organic matter and accumulation of the rejected component taking place at membrane surface. The deposition of organic matter on membrane surface can change the separation characteristic of the membrane. In this paper, membrane performance was evaluated under different trans membrane pressure (TMP) during peat water filtration using PSf 6a. The increase of flux leads to the increase in total resistance due to the rapid organic matter deposition on the membrane surface or into the membrane pore. The more open pore membrane leads to a lower humic acid rejection in which the increase pressure decreases membrane rejection as shown in Figure 6.

CONCLUSIONS

Flat sheet polysulfone ultrafiltration membrane was prepared by blending 20% wt of polysulfone in DMAc solvent, with two different additives namely PEG and acetone. The effect of additives concentration and operating pressure on membrane performance were investigated.

The addition of both PEG and acetone into membrane solution improved membrane performance. The increase of PEG concentration contributed to the increase of membrane hydrophilicity. At 25% wt of PEG, the water flux increased around 127% compared to that with the absence of PEG. Meanwhile, the increase of acetone concentration increased humic acid rejection, due to tight skin layer formation. The maximum rejection was achieved at 8% wt acetone concentration.

Effect of operating pressure on membrane performance was also investigated. The increase of operating pressure increased total fouling resistance and decreased humic acid rejection. It suggested that membrane prepared by blending 20% wt of polysulfone, 25% wt PEG and 4% wt of acetone resulted in a more open pore structure in the skin layer.

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