



## The Influence of PEG400 and Acetone on Polysulfone Membrane Morphology and Fouling Behaviour

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**Abstract.** Modification of polysulfone ultrafiltration membrane was conducted by blending polysulfone with PEG400 and acetone as additives. The influence of each additive on the resulted membrane morphology and fouling characteristics were investigated. The experimental results showed that the hydrophilicity of the polysulfone membrane was improved by the increase of PEG400 in the polysulfone membrane. The water contact angle of the membrane was decreased from 76.1° to 38.31° when 35 %wt of PEG400 was added into the polysulfone solution, while the water content of the membrane was increased by around 38%. The high concentration of PEG400 in the polysulfone solution led to the formation of longer finger-like cavities in the membrane structure and resulted in a thicker membrane skin layer. The high concentration of PEG400 also contributed to the increase in hydraulic resistance of the membrane due to organic matter fouling. This problem could be minimized by the addition of acetone into the polysulfone solution, which resulted in a lower fouling resistance of organic matter during up to five hours of peat water filtration.

**Keywords:** *additives; blending method; fouling; hydrophilicity; polysulfone membrane.*

### 1 Introduction

Ultrafiltration membrane based processes are increasingly used in water treatment since they have many advantages, such as better product quality, a smaller footprint area and less energy consumption compared to using conventional processes. In addition, membrane processes significantly reduce chemical consumption, such as chlorine, coagulants, flocculants or chemicals for pH adjustment. One of the polymers commonly used as UF membrane material is polysulfone due to its good chemical and temperature resistance. However, polysulfone membrane is naturally hydrophobic and susceptible to natural organic matter (NOM) fouling during surface water treatment [1,2]. As the major fraction of NOM, humic substances are considered the most foulant, inducing the water to turn brown and acidic [3]. The continuous deposition of substances on the membrane surface contributes to pore constriction or plugging, which declines membrane permeability and deteriorates selectivity

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performance. As a consequence, hydraulic resistance of the membrane is increased and filtration efficiency is reduced.

Fouling of humic substances on the membrane surface depends on several parameters, such as molecule characteristics (molecular weight, aromaticity, hydrophobic and charge), solution condition (pH, ionic strength), membrane properties (hydrophobicity, MWCO, charge, pore size and distribution), and hydrodynamic operating conditions (flow geometry, transmembrane pressure). It has been observed that the permeate flux declines rapidly at high ionic strength and low pH of solution [4]. In this condition, humic substances tend to become more hydrophobic due to neutralization of the negative charge in their functional groups. As a result, the interaction between the membrane and the substances is enhanced. With a further increase of the solution pH from neutral to high, the organic matter becomes more negatively charged. It is suggested that higher hydrophilicity, negative charge and smaller pore size in the membrane skin layer are needed to reduce organic matter fouling tendencies in the UF membrane.

Blending a polymer with hydrophilic materials or charged additives is considered to be an attractive and simple method to modify the membrane properties in order to minimize organic matter fouling [5]. Additives that are frequently used to improve polysulfone membrane hydrophilicity are hydrophilic polymers – such as polyvinylpyrrolidone (PVP) and polyethylene glycol (PEG) – or charged polymers – such as sulfonated polyether ether ketone (SPEEK). Bowen, *et al.* blended polysulfone with sulfonated poly(ether ether ketone) (SPEEK) to induce a high negative charge into the membrane structure [6,7]. The resulted membrane showed high rejection of humic acid and had low fouling properties. Hwang, *et al.* blended hydrophobic polyphenylsulfone (PSU) with polyetherimide (PEI) and PEG [8,9]. The blended polymeric materials attained a weak negative charge and exhibited good resistance to humic substance fouling. Furthermore, the increase in PEG concentration enhanced the pore size distribution and permeability of the membrane. Meanwhile, the increase of PEI concentration changed the membrane surface charge from negative to neutral. The effects of type, molecular weight and concentration of additives on the polysulfone membrane structure have been studied [10-13]. It is known that the presence of a polymeric additive in a polysulfone solution acts as pore former and suppresses the formation of macrovoids in the membrane structure [14,15]. As a result, higher porosity and modification of the skin layer structure can be obtained. Chakrabarty, *et al.* [16] found that increase of the PEG molecular weight increased the pore number and hydraulic permeability of the membrane. Kim, *et al.* [10] also investigated the effect of molecular weight and concentration of PEG on the formation of the polysulfone membrane. They

found that an increase of molecular weight and concentration of PEG enhanced water flux but reduced solute rejection.

A tight skin polysulfone membrane with high selectivity to humic substances was obtained in our previous work [17] by blending polysulfone with PEG400 and acetone as additives. It was found that an increase of the acetone concentration in the polysulfone solution improved membrane selectivity: 80% rejection of humic substances was achieved. In the present work, the influence of each additive, i.e. PEG400 and acetone, on the polysulfone membrane characteristics was further investigated. Special attention was given to the influence of the additives (PEG400 and acetone) on the membrane morphology and its organic matter fouling characteristics. The membrane morphology was evaluated by SEM, while the organic matter fouling behavior on the resulted membrane was investigated by calculating the hydraulic resistance of the membrane during peat water filtration. Also, the hydrophilicity of the polysulfone membrane was evaluated by measuring the water contact angle and equilibrium water content (EWC).

## **2 Materials and Method**

### **2.1 Materials**

The type of polysulfone used in this experiment was UDEL-P3500 (MB7), supplied by Solvay Advanced Polymer. DMAc with 99.9% purity, supplied by Shanghai Jingsan Jingwei Chemical Co. Ltd. PEG400, was used as solvent without further purification and acetone was used as additive. Demineralized water was used as non-solvent in the coagulant bath to induce the formation of the membrane structure. The peat water was obtained from Pekanbaru River, Riau, Indonesia. The measured pH of the peat water was around 3.

### **2.2 Membrane Preparation**

The experiment was conducted based on our previous research [17,18]. The polysulfone was dissolved into the DMAc and then mixed with the additives (PEG400 and acetone) at room temperature. The polysulfone solution was stirred for around 12 hours in a closed stirred tank up to homogenous and left without stirring until no bubbles were observed. Then, the polysulfone solution was casted on a flat glass plate with 200  $\mu\text{m}$  thickness and immediately immersed in a demineralized water bath as coagulant. The casted membrane was immersed for five (5) hours or until all solvent had completely evaporated. The variations of casting polysulfone solution used in this experiment are shown in Table 1.

## 2.3 Characterization of Membrane

### 2.3.1 Contact Angle Measurement

The hydrophilicity of the polysulfone ultrafiltration membrane was characterized by measuring the water contact angle on the membrane surface using the sessile drop method. Around five (5) microliters of water was dropped on the membrane surface by stainless steel needle at room temperature. The contact angle of each membrane was measured two-three times by optical camera.

**Table 1** Composition of polysulfone solution.

Membrane Code	Composition of polysulfone solution (%wt)			
	PSF	PEG400	Acetone	DMAC
PSf-1a	20	0	0	80
PSf-2a	20	5	0	75
PSf-3a	20	10	0	70
PSf-4a	20	15	0	65
PSf-5a	20	20	0	60
PSf-6a	20	25	0	55
PSf-7a	20	30	0	50
PSf-8a	20	35	0	45
PSf-1b*	20	0	4	76
PSf-2b*	20	5	4	71
PSf-3b*	20	10	4	66
PSf-4b*	20	15	4	61
PSf-5b*	20	20	4	56
PSf-6b*	20	25	4	51
PSf-7b*	20	30	4	46
PSf-8b*	20	35	4	41

(\*) Refer to our previous research [17].

### 2.3.2 Equilibrium Water Content (EWC) and Porosity of Membrane

Equilibrium water content is one of the membrane characterization methods to determine the degree of membrane hydrophilicity, which is related to water flux and membrane porosity. The wet flatsheet membrane was wiped with tissue paper and weighed in an electronic balance. The weight of the wet flatsheet membrane is referred to as  $W_w$ . Then, said membrane was dried in vacuum condition at room temperature for 16 hours. The weight of the dried membrane is referred to as  $W_d$ . The EWC of the membrane was determined by the following equation [16]:

$$EWC(\%) = \frac{W_w - W_d}{W_w} \times 100 \quad (6)$$

The membrane porosity was calculated by the following equation [16]:

$$Porosity = \frac{W_w - W_d}{\rho_w \cdot xV} \quad (7)$$

where  $\rho_w$  is the density of pure water at room temperature ( $\text{kg/m}^3$ ) and  $V$  is the volume of the wet membrane ( $\text{m}^3$ ).

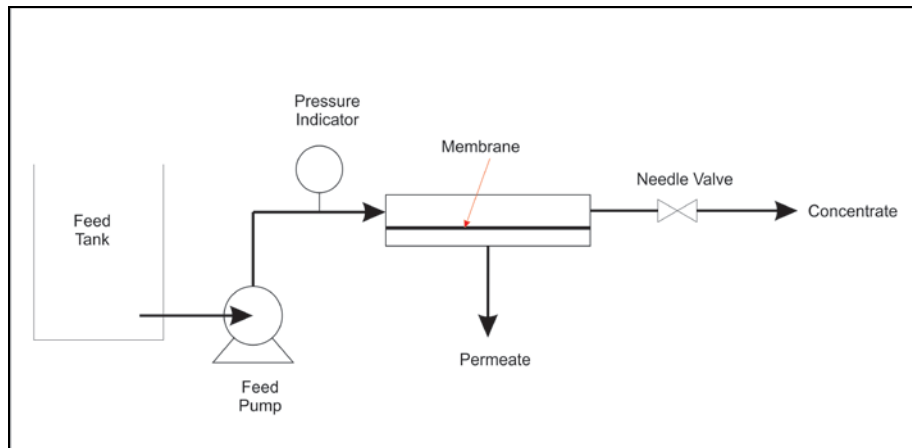
### 2.3.3 SEM

The prepared membrane was characterized by scanning electron microscopy (SEM) to obtain visual information of the cross-sectional morphology of the membrane. To obtain the cross-sections, membrane sample was fractured cryogenically in liquid nitrogen and deposited with gold using a sputter coater. Then, the sample was imaged using a scanning electron microscope (JSM-6510LV low vacuum SEM equipped with a super conical lens) with a magnification of 500 times.

## 2.4 Hydraulic Resistance of Polysulfone Membrane During Peat Water Filtration

In this research, fouling of organic matter on the polysulfone membrane was analyzed by investigating the change of hydraulic resistance during five (5) hours of peat water filtration. The hydraulic resistance ( $R_T$ ) was calculated by the following equation:

$$R_T = \frac{\Delta P}{\mu J} \quad (1)$$



**Figure 1** Experimental setup.

$R_T$  is the hydraulic resistance of the membrane ( $m^{-1}$ ),  $J$  is the peat water flux ( $Lm^{-2}h^{-1}bar^{-1}$ ),  $\Delta P$  is the transmembrane pressure (bar) and  $\mu$  is the viscosity of solution (bar.h) [19]. The flux of the resulted polysulfone membranes was measured in our previous work [18] and was used to calculate the hydraulic resistance ( $R_T$ ). The experiment setup is shown in **Error! Reference source not found.** The experiment was performed in cross-flow mode at 1 atm. Prior to the experiment, the circular flatsheet membrane was placed inside the membrane module and rinsed by demineralized water for 30 minutes at 1 atm. The active surface area of the flatsheet membrane was  $45\text{ cm}^2$ . The measured pH of the peat water solution was around 3.

### 3 Result and Discussion

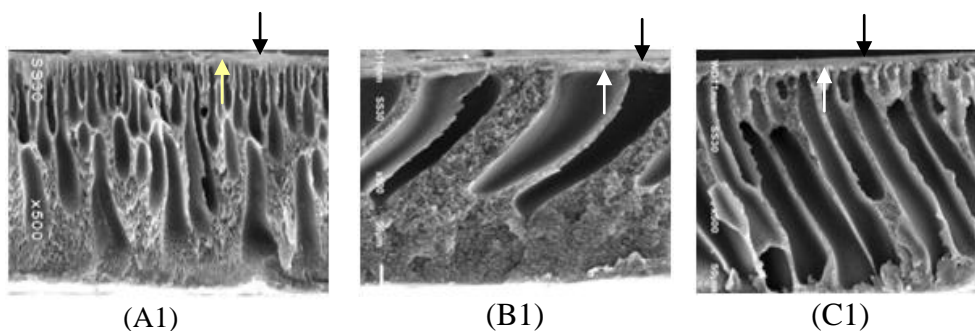
In the following section, an analysis of the relationship between additive concentration and membrane structure, hydrophilicity and fouling behaviour is presented. The experimental results are used to describe the obtained membrane performance for organic matter removal in water treatment. The evaluation of the effect of the additives on membrane performance provides a basis for the rational selection of the polysulfone solution composition for low molecular weight compounds in water.

#### 3.1 Effect PEG400 Concentration to the Polysulfone Membrane Morphology

The addition of PEG400 at different concentrations influenced the formation of pores in the membrane structure. **Error! Reference source not found.** shows a cross-section SEM image of the casted membrane consisting of 20 %wt polysulfone in DMAC and blended with different PEG400 concentrations. The obtained polysulfone membranes had an asymmetric structure consisting of a dense top layer and a porous sublayer. The finger-like cavities in the membrane sublayer were changed by the presence of PEG400 in the polysulfone solution. As presented in **Error! Reference source not found.**, the length of the finger-like cavities in the membrane structure became more extended with the further addition of PEG400.

It has been known that an asymmetric membrane structure depends on the solvent and additives [12]. The high mutual affinity between DMAC and water contributes to instantaneous demixing when immersed in a coagulation bath and the formation of finger-like cavities in the sublayer of the membrane [20,21]. Meanwhile, the presence of hydrophilic PEG influences the obtained membrane structure due to an increase of water diffusion into the polymer solution and thus enhances precipitation. Since water is a strong non-solvent, coagulation occurs rapidly when a polymer solution is brought into contact with water and

also leads to instantaneous demixing. The concentration of PEG400 also contributes to the final structure of the membrane skin layer that is immediately formed after immersion of a casted polysulfone solution into a coagulation bath [22,23]. As shown in **Error! Reference source not found.**, the increase of the PEG400 concentration in the membrane induced a higher solid concentration in the top layer of the membrane during solvent evaporation, resulting in a thicker membrane skin layer.



**Figure 2** SEM of polysulfone membrane prepared with different concentrations of PEG400: (A) 0% wt, (B) 25% wt, (C) 30 % wt.

### 3.2 Effect of PEG400 on Hydrophilicity and Porosity of The Polysulfone Membrane

It has been noted that the addition of PEG400 contributes to the improvement of membrane hydrophilicity, which is determined by the contact angle method and equilibrium water content (EWC) [16,24]. **Error! Reference source not found.** shows the characteristics of the membrane when the concentration of PEG400 was varied from 0 up to 35 %wt. The addition of PEG400 to polysulfone solution leads to an improvement of membrane hydrophilicity, which is implied by the decrease of contact angle and the increase of EWC. In this experiment, the unmodified polysulfone membrane was categorized as a hydrophilic membrane. Membranes are considered hydrophilic when the surface contact angle is smaller than  $90^\circ$  [25]. The hydrophilicity of the unmodified membrane was further improved by the addition of PEG400 in the polysulfone solution. Compared to the unmodified membrane, the contact angle of the membrane decreased up to 32% when the concentration of PEG400 increased from 0 to 35 %wt. The decrease of contact angle was followed by an improvement of EWC: the EWC value of the membrane was increased by around 38% at the addition of PEG400 up to 35 %wt.

In this research, calculation of membrane porosity was also performed. As presented in **Error! Reference source not found.**, a membrane with higher porosity was obtained by increasing the PEG400 concentration. The opposite

result was obtained for membranes 7a and 8a: after addition of PEG400 over 20 %wt, the calculated membrane porosity began to decline. This result can be explained by referring to **Error! Reference source not found.**, which shows that a higher PEG400 concentration induces the formation of longer finger-like pores in the membrane structure. The results suggest that a higher PEG400 concentration induces a more developed polymer-lean phase, which allows growth and coalescence of the formed membrane pore. However, despite the decrease of membrane porosity, the membrane hydrophilicity still increased, which is indicated by a further decrease of the membrane's contact angle.

**Table 2** Effect of PEG400 concentration on hydrophilicity and porosity of polysulfone membrane.

Membrane code	Composition of polysulfone solution (%wt)				Contact angle (°)	EWC (%)	Porosity (%)
	PSF	PEG400	Acetone	DMAc			
PSf-1a	20	0	0	80	76.1	55.6	0.4
PSf-2a	20	5	0	75	69.6	57.2	0.5
PSf-3a	20	10	0	70	64.6	61.7	0.6
PSf-4a	20	15	0	65	55.2	65.2	0.9
PSf-5a	20	20	0	60	53.1	68.8	1.0
PSf-6a	20	25	0	55	50.5	71.3	1.3
PSf-7a	20	30	0	50	48.2	75.1	0.7
PSf-8a	20	35	0	45	38.3	76.5	0.8

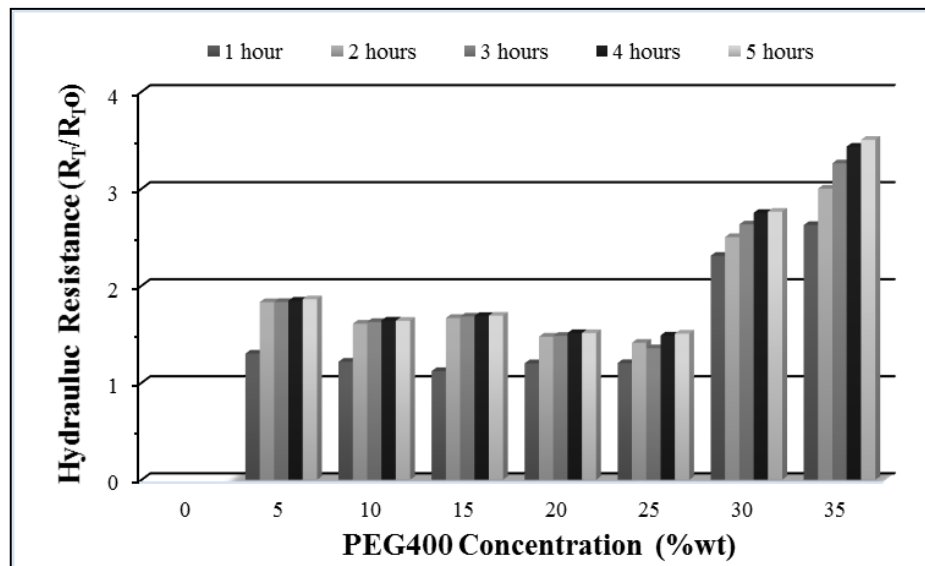
### 3.3 Effect of PEG400 Concentration on Hydraulic Fouling Resistance of Polysulfone Membrane

Fouling by organic matter of the membrane surface during peat water filtration contributed to an increase in hydraulic fouling resistance. The measured hydraulic fouling resistances at various PEG400 concentrations are shown in **Error! Reference source not found.** The hydraulic resistance was calculated as the ratio of hydraulic fouling resistance at a certain time ( $R_T$ ) to the initial hydraulic resistance at the beginning of the filtration ( $R_{T_0}$ ). It seems that the hydraulic fouling resistance slightly decreased with the enrichment of the PEG400 concentration up to 25 %wt, after which it started to increase with the further addition of PEG400. This means that the presence of PEG400 in polysulfone solution may not necessarily reduce organic matter fouling of the polysulfone membrane. The total fouling resistance of the membrane system was stable after two (2) hours of peat water filtration.

At a high concentration of PEG400, fouling of the membrane surface is governed by irreversible fouling due to open-pore formation in the membrane structure [10,26]. This irreversible fouling contributed to the increase of the total fouling resistance of the membrane surface during five (5) hours of peat



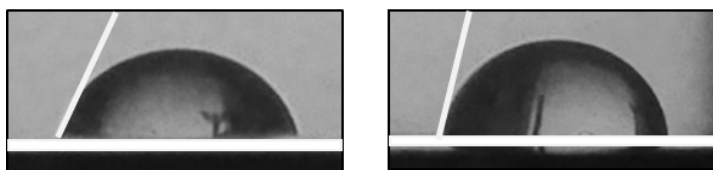
water filtration. Furthermore, the low pH of the peat water solution also contributed to the enhancement of organic matter hydrophobicity [27,28]. This initiated further interaction between the organic matter in the bulk solution and the organic matter deposited on the membrane surface. Therefore, cleaning of the membrane system should be conducted after two (2) hours of membrane filtration.



**Figure 3** Effect of PEG400 concentration on polysulfone membrane fouling resistance during five (5) hours of peat water filtration.

### 3.4 Effect of Presence of Acetone on Hydrophilicity and Hydraulic Fouling Resistance of Polysulfone Membrane

In this research, the influence of acetone on modified polysulfone membrane characteristics was observed by adding 4 %wt of acetone to the polysulfone solution. As presented in **Error! Reference source not found.**, the presence of acetone contributed to the increase of polysulfone membrane hydrophobicity, indicated by the higher water contact angle on the membrane surface. This is attributed to the formation of a tight structure in the membrane skin layer, which enhances membrane fouling resistance [17]. Due to the higher volatility of acetone combined with good interaction with water, acetone is easily separated and evaporated from polysulfone solution. The rapid loss of acetone leads to a higher polymer concentration in the membrane skin layer and produces tight pore structures [29].



**Figure 4** Influence of acetone in polysulfone solution on the contact angle of polysulfone membrane: (A) without acetone and (B) with 4 %wt of acetone.

**Error! Reference source not found.** shows the effect of 4 %wt acetone on the hydrophilicity and porosity of the unmodified polysulfone membrane. It appears that the contact angle of the membrane was reduced from  $81.4^{\circ}$  to  $56.2^{\circ}$  when PEG400 was added to the polysulfone solution up to 25 %wt. Meanwhile, the EWC of the membrane increased from 45% to 60%. At a further increase of the acetone concentration, the polysulfone solution was demixed by the earlier liquid-liquid phase separation before being casted on a glass plate. This implies that the additional acetone reduced the equilibrium boundary of the polysulfone solution. Furthermore, the presence of acetone also played an important role in the obtained membrane porosity. As shown in Table 3, membrane porosity was increased up to 0.7 by the addition of 25% wt of PEG400. The presence of acetone reduces the viscosity and increases the coagulation value (CV) of the polysulfone solution [12].

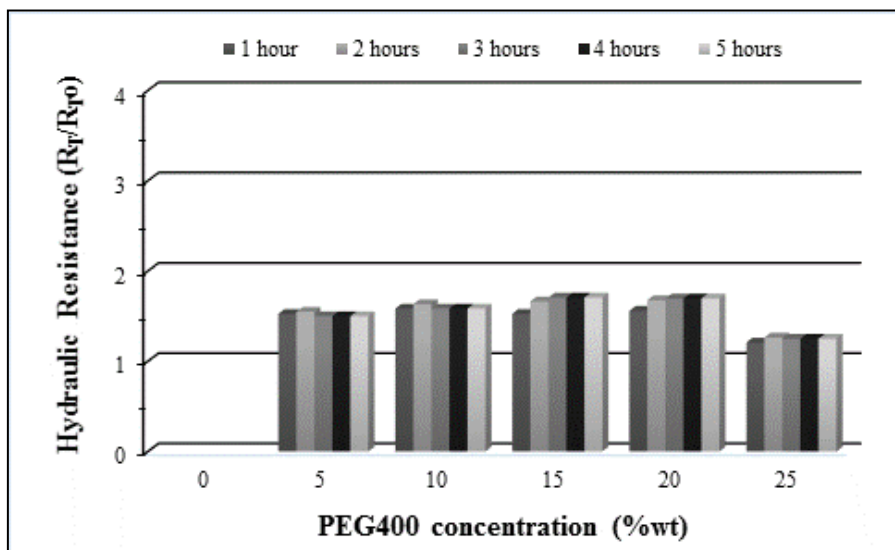
**Table 3** Effect of presence of acetone on membrane hydrophilicity and porosity.

Membrane code	Composition of Polysulfone Solution (%wt)				Contact angle (o)	EWC (%)	Porosity (%)
	PSF	PEG400	Acetone	DMAc			
PSf-1b	20	0	4	76	81.4	45.5	0.3
PSf-2b	20	5	4	71	75.5	48.4	0.4
PSf-3b	20	10	4	66	68.1	52.8	0.5
PSf-4b	20	15	4	61	67.2	61.3	0.6
PSf-5b	20	20	4	56	62.7	65.7	0.6
PSf-6b	20	25	4	51	56.2	65.9	0.7
PSf-7b	20	30	4	46		demix	
PSf-8b	20	35	4	41		demix	

This suggests that the formation of tight pore structures in the membrane skin layer due to rapid loss of acetone results in delayed demixing of the membrane substructure. The tight skin layer reduces the inflow rate of non-solvents into the polysulfone solution, which contributes to the formation of a high number of nuclei in the membrane substructure. Due to the lower viscosity of the polysulfone solution, coalescence of the amount of pore growth in the

membrane structure occurs. On the other hand, the addition of PEG400 leads to the increase of polysulfone solution viscosity, which increases the rheological hindrance for the pore growth [30]. This leads to the formation of higher porosity in the membrane substructure.

Due to the increase of membrane hydrophobicity, the addition of 4 %wt acetone results in a higher hydraulic fouling resistance, as indicated in Error! Reference source not found.. As explained previously, organic matter is more hydrophobic in low pH conditions and thus the interaction between the hydrophobic membrane surface and hydrophobic organic matter is induced. However, the addition of acetone contributed to a slight increase in hydraulic resistance during up to five (5) hours of peat water filtration. This means that the interaction between the modified membrane surface and the organic matter was weak, thus further accumulation of organic matter on the hydrophobic membrane could be easily removed by the cross-flow mode of filtration. Furthermore, a reduction of the hydraulic fouling resistance was achieved by the addition of 25 %wt of PEG400 to the polysulfone solution. This result shows that a low concentration of acetone and a high concentration of PEG400 can be chosen as additives in order to obtain a good performance of the polysulfone membrane for water treatment with high organic matter content.



**Figure 5** Effect of PEG400 concentration on polysulfone membrane fouling resistance (with 4 %wt of acetone).

#### 4 Conclusion

Modification of polysulfone membrane was conducted by dissolving polysulfone in DMAc with PEG400 and acetone as additives. The experimental results indicate that these additives have an important influence on the membrane's morphology and its fouling behaviour.

The addition of PEG400 enhanced the polysulfone membrane's hydrophilicity as indicated by a reduction in the water contact angle from 76.1° to 38.31° and an increase in equilibrium water content (EWC). A higher PEG400 concentration in the polysulfone solution enhanced the formation of pores in the membrane structure, which contributed to higher membrane porosity. The fingerlike cavities in the membrane sub-layer were also longer with the increase of PEG400 concentration. This can be attributed to the higher water inflow into the polysulfone solution during membrane preparation and the formation of the finger-like structure. Hydraulic resistance of the polysulfone membrane decreased slightly as the PEG400 concentration was elevated up to 20 %wt and then started to increase by further addition of PEG400. This suggests that the formation of open pores due to the high PEG400 concentration facilitates the membrane-solute interaction and thus the organic matter fouling on the membrane surface becomes more severe.

The formation of open pores in the membrane structure could be minimized by the addition of a low concentration of acetone in the polysulfone solution. The tight structure in the membrane skin layer due to the rapid loss of acetone during membrane structure formation induces higher membrane hydrophobicity. However, the increase of the membrane hydrophobicity only slightly increased the hydraulic fouling resistance of the membrane. This indicates that the interaction between the membrane surface and the organic matter was weak, which therefore could be easily removed by the cross-flow mode of filtration.

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