

# Surface Modification of Asymmetric Polysulfone Membrane by UV Irradiation

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## Article history

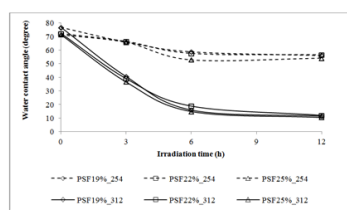
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## Graphical abstract



## Abstract

The effects of ultraviolet (UV) irradiation for surface modification of hydrophobic asymmetric polysulfone membranes have been investigated. The asymmetric polysulfone (PSF) membranes were prepared by phase inversion method using 19%-25% of PSF in two solvents, viz. dimethylacetamide (DMF) and Acetone (Ac) collectively. The surface of asymmetric polysulfone membranes were modified by UV ray with 254 and 312 nm wavelength. Chemical and physical properties of the untreated and the treated membranes were characterized. Scanning electron microscope (SEM) was used to determine asymmetric structure of polysulfone membranes. Contact angle device was used to analyzed the effected of UV ray treatment on hydrophilicity of membranes surface. Polar functional groups introduced by UV irradiation were examined using FTIR. The water flux was measured under a pressure of 500 kPa to 2,500 kPa with a feed temperature of 25°C. It was shown that asymmetric polysulfone membranes were produced and the UV ray treatment significantly alters the hydrophilicity of membranes surface indicated by the reduction of water contact angle with increasing treatment time. The FTIR analysis showed the formations of polar functional groups such as hydroxyl and carbonyl groups. Consequently, the surface of asymmetric polysulfone membranes was changed from hydrophobic to hydrophilic by UV irradiation leading to the enhancement of the water flux.

**Keywords:** Surface modification; asymmetric polysulfone membrane; UV irradiation; water flux; hydrophilicity

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## 1.0 INTRODUCTION

Polysulfone were widely used in the preparation of asymmetric membranes with different pore sizes in the skin layer [1]. Among many methods, phase separation was the common method for fabrication of flat sheet asymmetric membranes [2]. Aroon *et al.* [2] prepared asymmetric polysulfone membranes using a mixture of a more volatile solvent (THF) and less volatile solvent (NMP) instead of one single nonvolatile solvent. They found that the skin layer is thicker and less defects. This was due to a volatile solvent can rapidly evaporate from the outermost surface of the membrane during polymer casting, which results in higher polymer concentration in the upper layer of the membrane lead to delayed liquid-liquid demixing and as a result, a dense skin layer with less defects and pin-holes was prepared. Acetone is a more volatile solvent with has boiling point 56°C, while boiling point of THF is 66°C.<sup>3</sup> It mean acetone more rapidly evaporate than THF so that it is possible to prepare asymmetric membranes with dense skin layer using acetone [2, 4-6]. Madaeni *et al.* [5] found that addition of acetone in to polymeric solution consist of PSF

and DMAc cause a decline in flux and an improvement in the retention of protein. In the same condition, Aryanti *et al.* [6] reported humic acid rejection increase with acetone concentration increase. This is confirmed that acetone could improve dense membrane.

Although polysulfone (PSF) has been widely used as a membrane material due to its high performance engineering thermoplastic, which resists degradation, low cost, excellent thermal, chemical and mechanical properties [7]. However, hydrophobicity of PSF membranes limits its wider application. Therefore, numerous investigations have been focused on the modification of PSF membranes to impart hydrophilicity to their structure [8-9]. There are many methods to modify the membrane surface such as plasma treatment, ozone treatment, ion beam treatment, gamma-rays or UV irradiations.

The plasma processes can alter the physico-chemical properties of the polymer surface. It improves polymer wettability, permeability, conductivity, adhesion or biocompatibility [10]. However, it resulted in only transient hydrophilicity with contact angle changes within 24 h of plasma

treatment, only the outer membrane surface was modified [11] and it difficult to control the chemical structure of the plasma treated polymer. Ozone is known to change the chemical structure of polymer membranes. It is anticipated that ozone-treated polymer membranes can show more stable properties, compared to plasma-treated membranes, due to ozone treatment which can change the bulk properties of the polymer while the plasma process only changes the surface properties of the polymer [12]. Ion beam irradiation has long been recognized as an effective method for the modification of diverse materials [13]. It can improve polymer properties such as surface hardness, resistance to mechanical wearing and chemical erosion, and conductivity [14]. With appropriate choice of irradiation conditions (i.e. ion type and energy), energetic ions modify the surface layer of materials within a well-defined depth [15]. It is known that many important physical and chemical properties of polymers can be modified with gamma-rays irradiation [16]. It has been reported that it can change electrochemical characterization (salt permeability, ionic permselectivity and electrical resistance) of cellophane membranes [17]. However, these methods are expensive and difficult to perform.

Many studies about the effects of these methods on several types of polymers have been reported. But the information about the effects UV irradiation on microporous PSF membrane especially, is scarce. However, it has been reported that it increased flux and the hydrophilicity of the membranes.<sup>18</sup> Compared with other surface modification techniques such as plasma treatment, ozone treatment, ion beam treatment, and gamma-rays irradiation, UV irradiation has distinct advantages over other techniques due to its simplicity, inexpensive and widespread industrial applications [19].

The objectives of this research were preparation asymmetric polysulfone membranes and surface modification by UV irradiation. The asymmetric polysulfone membranes were prepared by a phase inversion process using acetone as volatile solvent. The effect of acetone on the morphology was investigated by SEM. The UV ray with 254 and 312 nm wavelength and power 2000 and 360  $\mu\text{w}/\text{cm}^2$ , respectively, was used for the treated membranes. Also, the effect of UV irradiation on hydrophilicity of membranes and was studied using contact angle device, FTIR and dead-end filtration techniques by comparing the results with the untreated membranes.

## 2.0 EXPERIMENTAL

### 2.1 Materials

Polysulfone (Udel P-3500) resin was supplied by Sovay (China). N,N-dimethylacetamide (DMAc) and Acetone (Ac) were used as solvents for PSF membrane and supplied by Sigma–Aldrich Co. (USA) and Guangdong Guanghua Chemical Factory Co., Ltd. (China), respectively. Tap water at room temperature (25°C) was used as a coagulation medium during phase-inversion process.

### 2.2 Preparation of Asymmetric Flat Sheet Membranes

PSF resin was dried in an oven at about 80°C for 24 h to remove humidity before use. PSF was dissolved in solvents of DMAc and Ac different concentrations (Table 1). The solutions were mixed at 60°C for 24 h and placed in an ultrasonic water bath to remove air bubbles. Each solution was cast on a clean glass plate. The thin polymer sheet was immersed in the coagulation bath for 24 h. During the phase inversion process, the exchange between the solvent and nonsolvent lead to the formation of the asymmetric

PSF membrane. The wet membranes were dried at room temperature for 24 h before being used.

**Table 1** Membranes with different dope solution compositions and UV irradiation conditions

Sample	PS F (%)	DMAc (%)	Ac (%)	UV ray wavelength (nm)	Irradiation time (h)
PSF19%_0h	19	54	27	-	0
PSF22%_0h	22	52	26	-	0
PSF25%_0h	25	50	25	-	0
PSF19%_3h_254	19	54	27	254	3
PSF22%_3h_254	22	52	26	254	3
PSF25%_3h_254	25	50	25	254	3
PSF19%_6h_254	19	54	27	254	6
PSF22%_6h_254	22	52	26	254	6
PSF25%_6h_254	25	50	25	254	6
PSF19%_12h_254	19	54	27	254	12
PSF22%_12h_254	22	52	26	254	12
PSF25%_12h_254	25	50	25	254	12
PSF19%_3h_312	19	54	27	312	3
PSF22%_3h_312	22	52	26	312	3
PSF25%_3h_312	25	50	25	312	3
PSF19%_6h_312	19	54	27	312	6
PSF22%_6h_312	22	52	26	312	6
PSF25%_6h_312	25	50	25	312	6
PSF19%_12h_312	19	54	27	312	12
PSF22%_12h_312	22	52	26	312	12
PSF25%_12h_312	25	50	25	312	12

\*The ratio of DMAc and Ac was fixed at 2:1 (wt/wt)

### 2.3 UV Irradiation

The UV ray source (VILBER LOURMAT, VI-215.MC) with 254 and 312 nm wavelength of 2000 and 360  $\mu\text{w}/\text{cm}^2$ , respectively, was used for the membranes treatment. Dried PSF membranes of 2  $\text{cm}^2$  were irradiated by UV ray in air at room temperature for 3–12 h (Table 1).

### 2.4 Membrane Characterizations

#### 2.4.1 Scanning Electron Microscopy (SEM)

Membrane thickness and porous substructure of the untreated and the treated membrane morphology were determined by a scanning electron microscopic (SEM) technique. For cross-sections analysis, the samples were frozen in liquid nitrogen and fractured followed by gold sputtering before scanning on a scanning electron microscope (FEI, Quanta 400) with potentials of 20 kV under magnifications ranging from 1,000 $\times$  to 5,000 $\times$ .

#### 2.4.2 Fourier Transform Infrared Spectrometer (FTIR)

Fourier Transform Infrared Spectrometer (FTIR: Bruker, EQUINOX 55) was used to investigate changes in functional groups of the membranes. The membrane was scanned at 400–4000  $\text{cm}^{-1}$  wave numbers.

### 2.4.3 Water Contact Angles

Water contact angle of PSF membranes was measured using a contact angle device (Dataphysics, COCA 15 ED) immediately after the UV irradiation.

### 2.4.4 Water Flux and Hydraulic Permeability Coefficient ( $L_p$ )

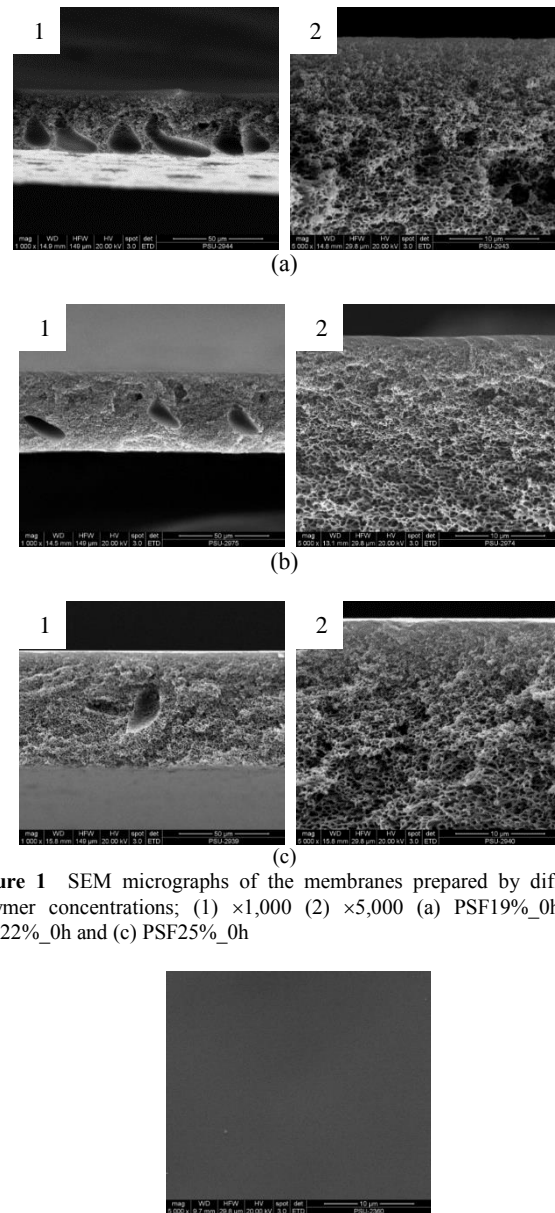
Dead-end filtration method was used to characterize the water flux of the untreated and the treated membranes. A piece of circular membrane of 6 cm diameter was immersed in ethanol (70%) for around 3 min before being used as a filter. Water flux measurement was made by placing a membrane in a dead end filtration unit, using applied pressures between 0.5 and 2.5 MPa. The water flux  $J$  ( $L/m^2h$ ) was calculated and the slope of the graph between corresponding fluxes and applied pressures was used to deduce the membrane hydraulic permeability coefficient ( $L_p$ ), using the Hagen–Poiseuille equation,  $J = L_p \Delta P$  [20].

## 3.0 RESULTS AND DISCUSSION

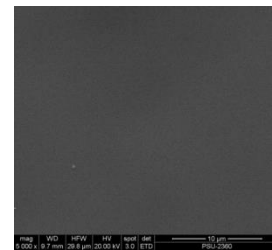
### 3.1 Scanning Electron Microscopy (SEM)

The morphology of the prepared membranes was investigated by SEM. The formation of thick skin layer on sponge-like support was shown in Figure 1. This structure was asymmetric membranes structures [21]. This was in accordance with result determined by Aroon *et al.* [2] The skin layer was thicker and less defect due to a volatile solvent (acetone) can rapidly evaporate from the outermost surface of the membrane during polymer casting, which results in higher polymer concentration in the upper layer of the membrane lead to delayed liquid–liquid demixing and as a result, a dense skin layer with less defects and pin-holes was prepared.

The asymmetric polysulfone membranes prepared by different polymer concentrations (19%-25%) shown similar surface morphology. The surface morphology of PSF 25% was shown in Figure 2. No surface pores could be observed on membrane surface, even at magnifications of 5000. This indicated that the diameter of surface pores were less than 200 Å [7]. Furthermore, it also showed the macro-voids of sponge-like support reduce at higher polymer. The formation of this structure is due to higher resistance for exchange of solvent and nonsolvent for more concentrated polymer. This results in liquid–liquid demixing. This produces a membrane with thicker skin layer and denser support [2, 22]. This is confirmed that acetone could improve dense membrane. The surface morphology of prepared membranes was shown Figure 2.



**Figure 1** SEM micrographs of the membranes prepared by different polymer concentrations; (1)  $\times 1,000$  (2)  $\times 5,000$  (a) PSF19%\_0h, (b) PSF22%\_0h and (c) PSF25%\_0h

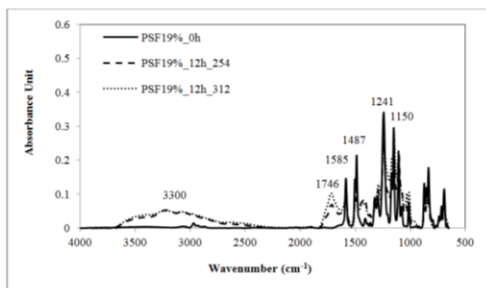


**Figure 2** SEM micrographs of asymmetric polysulfone membrane surface prepared by 25% of polymer concentrations (PSF25%\_0h.)

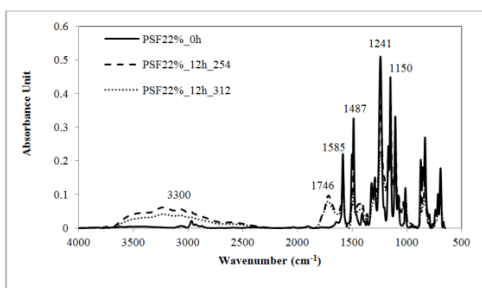
### 3.2 Fourier Transform Infrared Spectrometer (FTIR)

Figure 3 shows the FTIR spectra of the treated membranes by UV ray with 254 and 312 nm wavelength for 12 h, compared to the untreated membranes. Based on the spectral change, there is not much difference in each PSF concentrations. The PSF consists of a backbone made up of diaryl sulfone (Ar-SO<sub>2</sub>-Ar), diaryl ether (Ar-O-Ar) groups showed strong bands at 1150 and 1241  $cm^{-1}$ , respectively. The bands at 1487 and 1585  $cm^{-1}$  belong to the vibration of the aromatic (C=C) in PSF molecule [7]. However, the intensities of the peak at 1150, 1241, 1487 and 1585  $cm^{-1}$  decreased after UV irradiation, while new broad peaks arose around 3300  $cm^{-1}$  and near 1746  $cm^{-1}$ . The peaks appeared around 3300 and 1746  $cm^{-1}$  are ascribed to the stretching vibration of hydroxyl (-OH) group and carbonyl (C=O) group, respectively. The appearance of the peaks around 3300 and 1746  $cm^{-1}$  by UV irradiation indicates that the carbons in methyl group

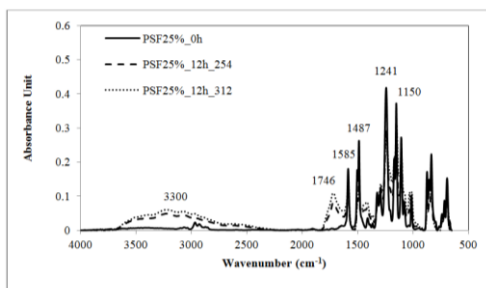
and in benzene ring of PSF were attacked and oxidized by UV ray to form carbonyl (C=O) group and hydroxyl (-OH) group [12]. Due to carbonyl and hydroxyl group were polar functional group so resulting in an increase of the hydrophilic property of the membrane [23]. In addition, it was found that the UV ray with 312 nm wavelength affected carbonyl and hydroxyl functional group of the treated membranes more than 254 nm wavelength.



(a)



(b)



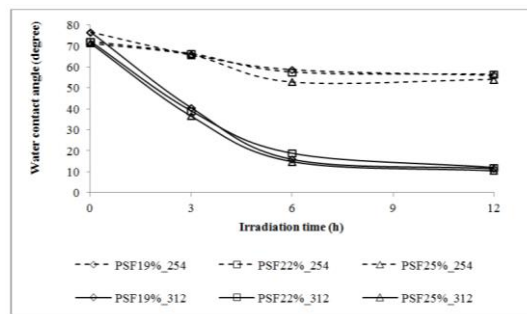
(c)

**Figure 3** Comparison of FTIR spectra of the untreated and the treated membrane by two UV ray wavelengths (a) PSF19%, (b) PSF 22% and (c) PSF 25%

### 3.3 Water Contact Angle

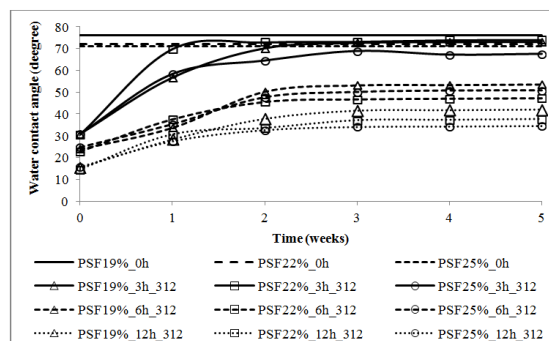
The water contact angles on the membrane surface of the treated membranes are shown in Figure 4 in comparison to the untreated ones. For the untreated, they were reduced to 76°, 72° and 71° while the polymer concentration was increased to 19%, 22%, and 25%, respectively. However, those of the treated membranes reduced greatly after UV irradiation. In addition, the 312 nm UV wavelength affected the water contact angles of the treated membranes greater than the 254 nm wavelength because the increment of polar functional groups on membranes surface by 312 nm UV greater than the 254 nm wavelength. This result was confirmed with FTIR. The angles reduced from 71°–76° to about 18°–15°, after 6 h of 312 nm ray treatment and being stable about 10°–12° after 12 h of the treatment. Using 254 nm wavelength,

they were about 52°–59° after 6 h treatment and became stable about 52°–56° after 12 h treatment. The results indicate that the UV irradiation increased the hydrophilicity of PSF membranes [24].



**Figure 4** Water contact angles studies vs irradiation time treated membranes by two UV ray wavelengths

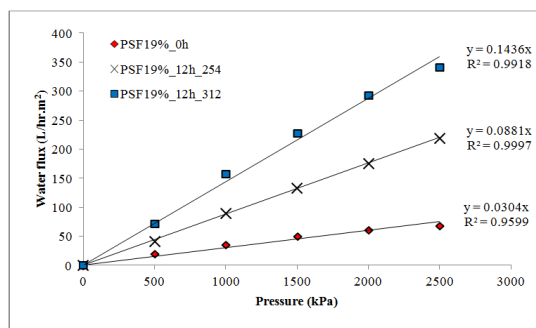
Further experiment was carried out by varying the time after membrane irradiation was made and shown in Figure 5. The treated membranes by 312 nm UV wavelength for 12 h were selected for this study because it more affected on membranes surface. A change in water contact angle with time was observed. The contact angle for the treated membranes increased rapidly up to 1 week. They increased from 30° to 70°, from 25° to 35° and from 15° to 25° while the irradiation time was increased to 3, 6 and 12 h, respectively. They remained rather steady after this treatment. For a membrane which was irradiated for 3 h, the water contact angle increased nearly equal to the untreated membranes within 2–4 weeks after the irradiation. It indicated that hydrophilicity gained by short irradiation time is not stable; the effect can be smaller and even disappear completely, namely “hydrophobic recovery” [25]. However, hydrophobic recovery decreased when irradiation time increased. Moreover, the polymer concentration affect water contact angle slightly at the same irradiation time.



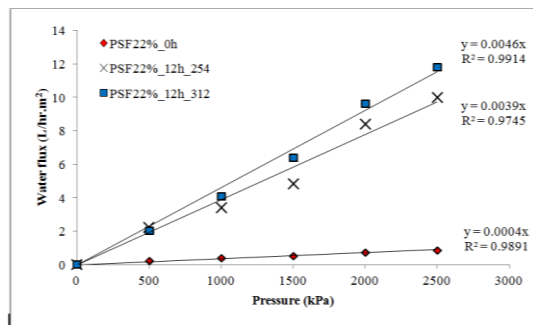
**Figure 5** Variation of water contact angles of the treated membranes by the UV ray with 312 nm wavelength (0-12h) vs storage time

### 3.4 Water Flux

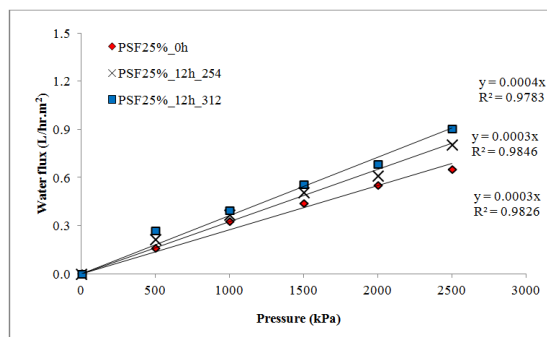
The water fluxes of the untreated and the treated membranes of different mixtures are shown in Figure 6. The treated film by UV ray 12 h was selected for this study because the contact angle shows minimum value at this condition. The results show the increased water flux with increased applied pressure, but the water flux was reduced with increased polymer concentration.



(a)



(b)



(c)

**Figure 6** Permeate of water flux vs driver pressure of untreated and treated membranes by two UV ray wavelengths (a) PSF19%, (b) PSF22% and (c) PSF25%

This is due to the increase in thickness with increased polymer concentration. Although the membrane thickness was controlled in casting process, the different polymer concentration casts the different membrane thickness after the loss of a volatile solvent from a casting solution in evaporation process [2, 22]. This results in smaller diffusion of water molecules through the membrane. In addition, the water fluxes of the treated membranes higher than the untreated membranes in overall polymer concentration. However, it was found that the UV ray with 312 nm wavelength affected water fluxes of the treated membranes more than 254 nm wavelength. This is due to the UV ray with 312 nm wavelength affected polar functional group of the treated membranes more than 254 nm wavelength. The water fluxes of the treated membranes by UV ray with 312 nm wavelength were 3.4, 12.9 and 0.3 times of the untreated membrane for polymer concentrations of 19%, 22%, and 25%, respectively. While water fluxes of the treated membranes by UV ray with 254 nm wavelength were 1.6, 11.0 and 0.2 times of the untreated membrane for polymer concentrations of 19%, 22%, and 25%,

respectively. This result confirms that the hydrophilicity of membranes increased after the UV irradiation. Due to UV ray produced polar functional group on membranes surface which according to Nyström *et al.* [18]. Table 2 shows the hydraulic permeability coefficient ( $L_p$ ) of the untreated and the treated membranes. It should be noted that the UV ray could increase  $L_p$  in the membranes. Hence UV irradiation increased hydrophilicity of the asymmetric polysulfone membranes.

**Table 2** Hydraulic permeability coefficient ( $L_p$ ) of the untreated and the treated membrane by UV ray for 12 h

Sample	$L_p \times 10^{-13} \text{ (m}^3\text{Pa}^{-1}\text{s}^{-1}\text{)}$
PSF19%_0h	84.21
PSF22%_0h	1.11
PSF25%_0h	0.83
PSF19%_12h_254	244.04
PSF22%_12h_254	10.80
PSF25%_12h_254	0.83
PSF19%_12h_312	397.77
PSF22%_12h_312	12.74
PSF25%_12h_312	1.11

#### 4.0 CONCLUSION

Asymmetric polysulfone membrane was prepared by phase inversion technique using acetone as volatile solvent. This structure was illustrated by SEM. The membrane surface was changed from hydrophobic to hydrophilic by UV irradiation. The optimum UV irradiation time was 12 h, UV ray 312 nm wavelength. Polar functional groups such as hydroxyl and carbonyl group were introduced to the skin of membrane surface and confirmed by FTIR and contact angle measurement. This study indicates that UV irradiation could increase the hydrophilicity of the polysulfone membrane.

#### Acknowledgement

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