Mercury Removal Through Thin Film Composite Membrane

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Abstract— **In this current study, a novel of chitosan, polysulfone and polyvinyl alcohol (PVA) composite membrane were prepared and applied for the removal of mercury from pure mercury solution. During membrane fabrication, two concentrations of polyvinyl alcohol were used. The resultant membranes were characterized in term of functional groups, thermal stability, membrane swelling and pure water permeability. The performance of the membrane was investigated through mercury removal and antifouling analysis. Results showed membrane incorporated with 4 wt.% PVA yield high flux with 96.73% of mercury removal. The obtained results proven that higher concentration of PVA can increase performance of the membrane.**

Keywords*— *Mercury Removal; Composite Membrane; Chitosan; Polyvinyl Alcohol; Polysulfone

1 INTRODUCTION

Mercury is one of the heavy metals that are known as one of the dangerous pollutants that contain in wastewater. The water that contain the pollutants usually came from the effluent of industrial process which need to be treated before being released to the surrounding. The release of great amount of heavy metals into water can give a huge impact to the human health and environment. Mercury that is released into river and lake water is fatal to human beings and aquatic life and upon consumed, these discharges may not be digestible in stomach and can lead to cancerous diseases. The damage of water pollution is so chronic that it cannot be recovered through regular membrane that exist nowadays (Sunil et al., 2018).

Membrane separation technologies play significant part in today's growth of sustainable industrial processes for global chemical production. This growth mostly caused by the less energy used by membrane separation compared to common separation process that being used by industry nowadays such as drying and distillation since membrane separation does not involve phase changes. Most importantly, the low cost of gas separation based on non-porous membrane attract high usage of membrane in recent years in additional to its high efficiency. According to (Su, Ye, & Hmidi, 2017), the membrane separation is proven to be a practical and environmental method to remove the heavy contaminant in wastewater.

In this study, a blend polymer of chitosan, polysulfone and polyvinyl alcohol will be produced. Since chitosan is a hydrophilic polymer, it is used to increase the hydrophilicity of the membrane. Chitosan was used as an organic additive which will mix with acetic acid. Since chitosan is hydrophobic, the function that it used was to increase the hydrophilicity of the membrane. Chitosan, a poly-2-amino-2deoxy-b-(1,4)-D-glucopyranose is a derived from chitin, which is one of the most abundant natural polysaccharides. It is easily obtained because chitin mostly can be obtained as a sub-product of seafood (Mello, Bedendo, Nome, Fiedler, & Laranjeira, 2006). Chitosan is suitable to separate desired and undesired materials including heavy metals such as mercury because it contains amino and hydroxyl group that is suitable sorbent for heavy metal ions. In addition, because of its characteristic which are non-toxic, biodegradable and biocompatibility, high selectivity and antimicrobial activity, chitosan is considered as environmentally friendly polymer. The chemical structure of chitosan is illustrated in the Figure 1-1 (Ghaee, Shariaty-Niassar, Barzin, & Ismail, 2013).

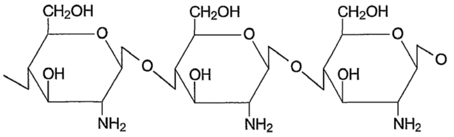

Figure 1-1: Chemical structure of chitosan

Polysulfone is one of the polymer that is important in polymeric material since it is majorly used in ultrafiltration, reverse osmosis and also pervaporation membranes (Kumar, Isloor, Ismail, & Matsuura, 2013). Polysulfone is one of the best candidate polymers because it has severe excellent characteristics of a good polymer in membrane production because of good flexibility, oxidative and have reasonable price. Moreover, polysulfone also have other good characteristic in membrane production which are good resistance to electrical, mechanical and chemical resistance properties. In this experiment, polysulfone were dissolved in N-methyl pyrrolidone (NMP) solvent then will be mixed with chitosan solution and polyvinyl alcohol solution to produce composite membrane. The chemical structure is illustrated in figure 1-2 (Kumar, Isloor, Ismail, & Matsuura, 2013).

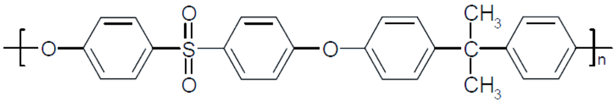

Figure 1-2: Chemical structure pf psf

Polyvinyl alcohol (PVA) is one of the hydrophilic polymers which have high water selectivity and dehydration properties. This PVA also supported by economic advantages since it is extensively available in global industry (Zhao et al., 2011). Due to its hydrophilic properties and compatible structure, PVA is broadly used by combining it with other type of polymer compounds and used in several industrial applications to improve the mechanical properties of films (Limpan, Prodpran, Benjakul, & Prasarpran, 2012). These advantages are the main reason why PVA is one of the best candidates to be use in the preparation of numerous commercial membranes.

In this study, two concentrations of polyvinyl alcohol were incorporated in the polymer blend solution of polysulfone/chitosan. The purpose of the study was to identify the effect of varying polyvinyl alcohol on the properties of the composite membrane as well as the performance of membranes in removing mercury ion.

2 METHODOLOGY

2.1 *Materials*

The material used was chitosan from Central Drug House (P) Ltd, New Delhi. N-methylpyrrolidone with a molecular weight of 99.13gmol^{-1} was obtained from Applied Biosystem Pty Ltd, Australia and Dimethyl Sulfoxide (DMSO) was obtained from Valhoma Corporation, United States.

2.2 *Membrane preparation*

There are few stages involved in the preparation of integral composite membrane.

2.2.1 *Preparation of chitosan solution*

Firstly, a chitosan with 2 wt% in acetic acid was prepared. 0.02g of chitosan was measured and then dissolved in 9.98 ml of acetic acid. The mixed solution was heated and stirred at 70℃ and 400 rpm for 1 hr. Then the solution was stored in a beaker for further process.

2.2.2 *Preparation of polysulfone solution*

Another solution that need to be prepared in the membrane production is 13 wt% Psf in NMP solution. Firstly, 13g of Psf was measured and then dissolved in 87 ml of NMP solution. The solution was heated at 80℃ and stirred by using magnetic stirrer at 400 rpm for 4 hr. The solution was cooled down to room temperature.

2.2.3 *Preparation of Polyvinyl Alcohol Solution*

3 g of PVA was measured and mixed with 97 g of distilled water. The mixed solution was stirred on a hot plate using a magnetic stirrer for 1 hr period under condition of 60℃ and 300 rpm. Since the concentration of PVA will be varied, this step was repeated by using 4 g of PVA and 96 ml of distilled water to make 4 wt% PVA solution.

2.2.4 *Preparation of composite solution*

0.1 ml chitosan solution inserted into 20 ml psf solution by using syringe and stirred on a hot plate for 1 hour under condition of 60 C and 300 rpm. After the solution have been homogeneous, 0.1 ml PVA solution was added into the composite solution and stirred for another hour until it become homogeneous solution. The composite solution then stored in a closed beaker before the casting process. Table 2-1 show the composition of the solution to produce the composite solution of the membrane.

Table 2-1: Composition of the composite solution

Solution	CS	Psf	PVA
Volume (mL)	0.1	20	0.1

2.2.5 *Casting of composite membrane*

After the solution was prepared, the composite membrane was produced by using casting method. 2 mL of the solution was measured using syringe and discharged on a glass plate. The glass plate has to be completely free from any contamination such as moisture to ensure that the casting method can be done successfully. The casting was done using an applicator, where it was set to 90 µm thickness. The solution was poured on the glass plate and it was spread uniformly into film with 90 µm thickness by using the laminator. Then, the film was soaked in distilled water for 24 h before dried it in a room temperature for another 24 h.

2.3 *Membrane characterization and performance tests*

For this section, in membrane characterization, fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA) had been used. Meanwhile in the performance testing, pure water permeability (PWP) was used for water flux.

2.3.1 *ATR-FTIR analysis*

FTIR was used to identify the presence or the interaction of functional groups as well as the types of bonding in the composite membrane sample. First of all, the membrane sample was cut off into small pieces before placed the small pieces on top of a diamond crystal plate for analysis process. The diamond crystal plate must be cleaned before used by using acetone liquid. The pressure that used in this analysis was set to 60-70 N simply by adjusting the swivel pressure tower. Finally, the reading sample membrane was collected in the range spectrum wavelength of 400 cm^{-1} to 4000 cm^{-1}.

2.3.2 *Thermogravimetric analysis (TGA)*

This analysis was conducted to determine thermal stability of the produced membranes. The membrane sample was cut into small pieces for about 5 to 10 mg for the analysis process. After that, the sample was heated under nitrogen gas at heating rate of 10℃/min from temperature 30℃ to 900℃.

2.3.3 *Water uptake*

Water uptake was used to measure the swelling properties of the composite membranes. Firstly, dried composite membrane was immersed in deionized water at room temperature for 48 hours. The wet membrane then wiped with tissue paper and directly weight to get the mass, W_S. The membrane then dried in a vacuum oven overnight at temperature of 50 ℃. After dried, the membrane was weight again to measure the mass of dried membrane (W_d). Equation 1 below show the calculation for swelling degree (S) of the membrane.

$$S(\%) = \frac{(Ws-Wd)}{Wd} \times 100\% \qquad (1)$$

2.3.4 *Water Flux study*

The apparatus used was testing rig with dead end mode of filtration mode. Firstly, the membrane was cut into a circle shape with surface area of 19.63 cm^2 and then placed in the sample holder. Then, the deionised water was filled up into the stainless steel filtration cell as the feed solution with amount of 200 ml. The filtration process was performed at room temperature and 4 bar pressure by using nitrogen gas. After that, the permeate sample was collected in a beaker at 15 minutes interval time for 1 hour filtration. The reading was started after constant flowrate of permeate stream was observed. Equation 2 was used to calculate the permeate flux.

$$Flux = \frac{V}{\Delta t.A} \qquad (2)$$

where ΔV (L) is the volume of the permeate sample, Δt is the time interval (min) and A is an effective membrane area in m^2 Δt is time permeate.

2.3.5 *Antifouling*

Humic acid is used in the study of antifouling. Initially, humic acid is prepared by dissolving 10 mg HA powder into 1000 ppm NaOH solution in 1L volumetric flask. The characterization start with membrane is stabilized in membrane filtration rig at 6 bar using deionized water for 30 minutes followed by 2 hours of humic acid with 20 minutes interval. The flux is denoted as J_o and J_P. The membrane then washed using deonized water for 30 minutes using magnetic stirrer at 200 rpm. The membrane then once again filtered using deonized water for 30 minutes and the flux denoted as J_1.

The relative flux decay (RFD) and relative flux recovery (RFR) are calculated using equations 3 and 4 below.

$$RFD = \frac{(Jo - Jp)}{Jo} \times 100\% \qquad (3)$$

$$RFR = \frac{J1}{Jo} \times 100\% \qquad (4)$$

2.3.6 *Mercury removal analysis*

3 ml mercury solution was dissolved in 1000 ml of distilled water to make 3 ppm of mercury solution. The membrane was cut into circle shape of 0.002 m^2 to be used in membrane filtration rig. The membrane is stabilized with deionized water for first 30 minutes and followed by 1 hour of mercury solution with 15 minutes interval time for another 1 hour. The membrane then backwashed using deionized water for 30 minutes on a magnetic stirrer. Finally, the membrane was run through deionized water for the second time for 30 minutes. The permeate and feed sample were sent inductively coupled plasma atomic emission spectroscopy (ICP-AES) to determine the concentration of mercury to calculate the value of rejection of the mercury. The rejection, %R can be calculated by using the following equation.

$$Rejection, \%R = 1 - \frac{Cp}{Cf} \times 100\% \qquad (5)$$

3 RESULTS AND DISCUSSION

3.1 *ATR-FTIR*

The common wavelength functional groups for raw materials in this study such as C=O, O-H and C-H was shown in Table 3.1.

Table 3-1: FTIR wavelength functional group

No.	*Chemical group*		*Wavenumber (cm^{-1})*	*Reference*
1	PVA	C=O	1750-1735	(Mansur, Sadahira, Souza, & Mansur, 2008)
2	PVA	O-H	3550-3200	(Mansur et al., 2008)
3	PVA	C-H	2840-3000	(Mansur et al., 2008)
4	CS	ν(-C=O)	1568	(Negrea et al., 2015)
5	CS	δ(-CH3)	1372	(Negrea et al., 2015)
6	CS	να s(C=O)	1152	(Negrea et al., 2015)
7	CS	ω(C-H)	892	(Negrea et al., 2015)
8	Psf	O-S-O	1151	(Singh et al., 2014)
9	Psf	C-C aromatic	1585	(Singh et al., 2014)
10	Psf	C-H	1020 & 830	(Singh et al., 2014)

In Figure 3-1, the FTIR spectra for composite membrane f PVA of 3 wt% PVA was shown the functional group C=O, O-H and C-H. The wavelength of C=O is at 1737 cm^{-1}. Meanwhile, the wavelength of O-H is at 3735.12 cm^{-1} and the functional group C-H is at wavelength of 2970.18 cm^{-1} (Mansur et al., 2008). The chitosan group shows functional group of ν(-C=O) at wavelength 1568cm^{-1}, δ(-CH$_3$) at 1372 cm^{-1}, ναs(C=O) at 1152 cm^{-1} and ω (C-H) at 892 cm^{-1} (Negrea et al., 2015). The polysulfone shown functional group of (O-S-O) at wavelength of 1151 cm^{-1}, while at wavelength of 1585 cm^{-1} (C-C aromatic) are characteristics of the sulfone group (Singh et al., 2014). These changes of spectral show the presence of molecular interaction among the polymeric blends; highlight the compatible nature between each other (Sciences, Scientific, & Corp, 2014).

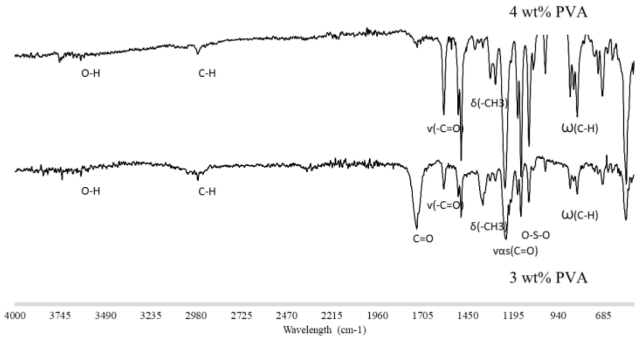

Figure 3-1: FTIR spectra of 3 wt% PVA membrane and 4 wt% PVA membrane

3.2 *Thermogravitimetric analysis (TGA)*

The main objective of thermogravitimetric analysis was to identify the thermal decomposition behaviour as well as the thermal stability of the integral membrane. Based on Fig. 3.2, both membranes showed similar pattern with only different on the percentage weight residue. Membrane with 3 wt% PVA starts to degrade at temperature 470.92 °C while membranes with 4 wt% PVA starts to degrade at temperature 488.62 °C. The maximum degradation temperature of both membranes also almost the same with 3 wt% shows at 532.95 °C while maximum degradation temperature of 4 wt% at temperature of 541.78 °C.

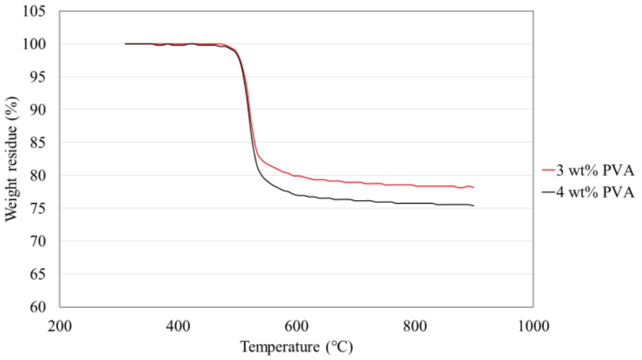

Figure 3-2: Weight residue against temperature

Table 3-2: degradation temperature of the membranes

Membrane sample	Degradation onset temperature (°C)	Maximum degradation temperature (°C)
3 wt% PVA	470.92	532.95
4 wt% PVA	488.62	541.78

3.3 *Water uptake*

Water uptake study was performed to study the hydrophilicity of the membrane whether the difference concentration of PVA affect the water uptake of the membrane. The rate of water uptake by membrane depends on its porosity (K, Isloor, Ismail, & Obaid, 2015). Table 3-3 show the value of the water uptake for the membranes. It shows that membrane with 4 wt% PVA has higher water uptake value, 204.12% compared to 198.48% which means the porosity of the membrane is higher than 3 wt% PVA membrane. The high value of water uptake result on the addition huge amount of chitosan and due to its nature properties which is a hydrophilic polymer (Kumar, Isloor, Ismail, Rashid, & Matsuura, 2013). The increasing of PVA content also increase the water uptake since PVA is one of the hydrophilic polymer (Zhao et al., 2011).

Table 3-3: Result for water uptake for each membrane

Membrane	Mass (g)		Water uptake (%)
	W_S	W_d	
3 wt% PVA	1.107	0.364	198.48
4 wt% PVA	0.822	0.275	204.12

3.4 Water flux study

The permeation studies were carried out in membrane filtration rig at a pressure at 3.45 bar for both 3 and 4 wt% PVA membranes. The result shown from the analysis that higher concentration of PVA gives higher value of flux. The flux pattern for both membranes are similar where the first 15 minutes is the highest flux values which 3 wt% PVA shows value of flux at 82 $Lm^{-2}h^{-1}$ while 4 wt% PVA is much higher which is 100 $Lm^{-2}h^{-1}$.

The trend is similar when time passes, the flux decreased as the volume of permeate is also decreased. The flux calculated at 1-hour mark is 58 $Lm^{-2}h^{-1}$ for 4 wt% membrane and 52 $Lm^{-2}h^{-1}$ for 3 wt% membrane. According to (Padaki, Isloor, Wanichapichart, & Ismail, 2012), water flux is basely influenced by the composition and type of the materials of the membrane. In this study, it is shown that higher amount of PVA in the membrane give the higher flux value. This statement can be explained with increasing of PVA content can promote the hydrophilicity of membrane as well as enhancing the diffusion of water molecules through the membranes (Hong, Xianshi, Mingcheng, & Ben, 2011).

Table 3-4: Water flux of 3 wt% PVA membrane

Time (min)	15	30	45	60
Flux ($Lm^{-2}h^{-1}$)	82	76	62	52

Table 3-5: Water flux of 4 wt% PVA membrane

Time (min)	15	30	45	60
Flux ($Lm^{-2}h^{-1}$)	100	74	64	58

The graph of flux against time is plotted below:

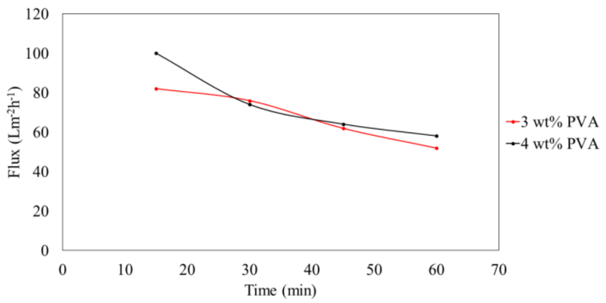

Figure 3-3: Flux against time (min)

3.5 Antifouling studies

The antifouling performances of the membranes were evaluated through the filtration of humic acid under the filtration condition described in Section 2.3.5. The related data on antifouling studies are summarized in Table 3-6.

As stated in Table 3-6, the initial pure water flux for membrane with 4 wt% PVA content is higher than 3 wt% with J_o was recorded at 279 $Lm^{-2}h^{-1}$ compared to 223 $Lm^{-2}h^{-1}$. The differences can be explained by the differences of hydrophilicity of the membrane with different content of PVA. The relative flux decay (RFD) of 3 wt% PVA membrane is at 98.25% while relative flux recovery (RFR) is only at 58.29% but still higher than 4 wt% PVA with RFR only recorded at 54.48%. These result shows that antifouling of the membrane can be improved by vary the content of the materials which contain functional copolymer that had hydrophilic segment within their molecular structure (Shen et al., 2018).

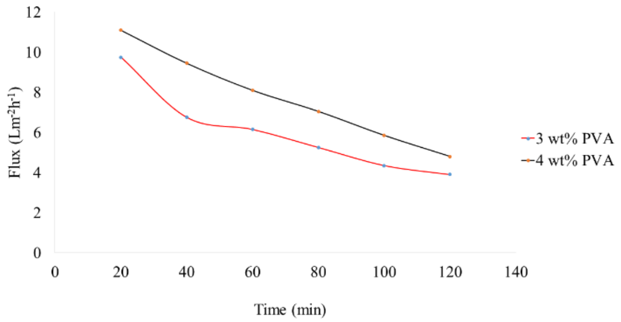

Figure 3-4: Flux for antifouling against time (min)

Table 3-6: Antifouling analysis result

Membrane	Permeate flux ($Lm^{-2}h^{-1}$)			RFR (%)	RFD (%)
	J_0	J_p	J_1		
3 wt% PVA	223	3.9	130	58.29	98.25
4 wt% PVA	279	4.8	152	54.48	98.28

3.6 Heavy metal rejection study

The heavy metal rejection study was performed by using 3 ppm mercury solution which been run through membrane filtration rig to determine the percentage of removal on the mercury concentration. The feed concentration, C_F and permeate concentration, C_P were determined and the value of rejection, %R recorded in Table 3-6. According to (K et al., 2015), rejection of the heavy metals depend on the pore size, where larger the pore size, smaller the rejection. However, in this study the characterization of the pore size was not study. In this study, the difference concentration of the PVA will result difference rejection value of Hg. Table 3-6 shows the maximum rejection of Hg where 4 wt% PVA shows 96.73% rejection while 3 wt% is 45.60.

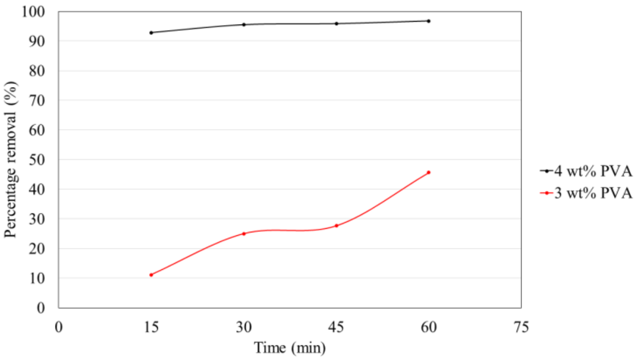

Figure 3-5: Percentage removal of mercury over time

Table 3-7: mercury removal data for 3 wt% PVA membrane

Membrane	Mercury rejection (%)
3 wt% PVA	45.60
4 wt% PVA	96.73

The trend of flux for mercury solution were quite similar similar for both membranes where the higher PVA content membrane shows higher flux value. The flux is shown in Figure 3-6.

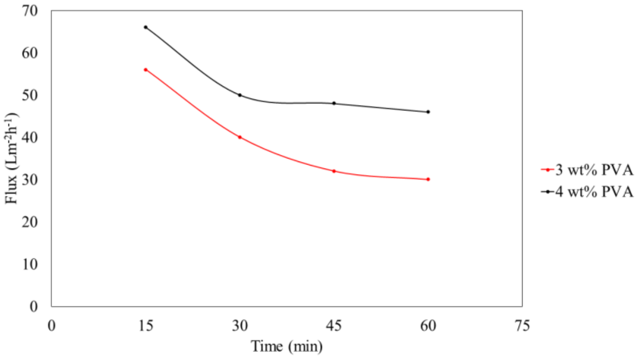

Figure 3-6: Flux for mercury solution

4 CONCLUSION

The water flux characterization result shows that higher the content of PVA in the membrane can increase the hydrophilicity of the membrane since the flux of 4 wt% PVA membrane is considerably higher than 3 wt% PVA membrane. The relative flux recovery of 54.48% was observed for the 4 wt% membrane while 3 wt% was observed at 58.29%. both of the membranes show high percentage of rejection of mercury which are 96.73% for 4 wt% and 45.60% for 3 wt%.

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