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Development of Chitosan/Pluronic F108/Polyethersulfone (PES) Nanofiltration (NF) Membrane for Oily Wastewater Treatment

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Abstract. This study discusses a new finding for nanofiltration membrane development using phase inversion technique whereby polyethersulfone (PES) polymer was added with surfactant and additive. This research focuses on the development of a membrane that is efficient in treating oily wastewater and reducing membrane's low permeation flux issues. Five PES nanofiltration membranes were synthesized with pluronic F108 surfactant and different amounts of chitosan additive for each formulation. Subsequently, the effect of adding surfactant and additive on membrane performance was studied. Results showed that the membrane with the optimal amount of chitosan gave the highest flux and the rejection of oily wastewater with up to 90%. In addition, Fourier transform-infrared (FTIR) spectroscopy technique was used to characterize and analyse the membrane's properties. Hence, the developed membranes were successfully characterized and proved to be a good treatment for oily wastewater.

INTRODUCTION

Recently, oily wastewater is most efficiently treated using membrane technology aside from the conventional water treatment technique. Nanofiltration membranes are characterized by pore size ranging within 0.5 – 2.0 nm while permeates rejected by the nanofiltration membranes are normally in a size range of 0.0005 – 0.0070 μm . In addition, the pressure needed for nanofiltration ranges from 500 to 2000 kPa so that permeates are produced and the osmotic pressure barrier can be overcome [1].

The usage of pluronic F108 surfactant coincides with the experiments conducted; pluronic could be a good pore former that increases pore size and membrane porosity [2, 3]. Moreover, pluronic is a non-ionic surfactant that has a high hydrophile-lipophile balance (HLB) number that helps to increase hydrophilicity of the membrane and improve its performance. In 2015, an article reviewed that PES membrane has higher permeation flux and better anti-fouling property when it is modified with pluronic F127, which is similar to pluronic F108, compared to unmodified membranes [4]. The additive used in this study is chitosan which is shrimp or crustacean shells treated with alkali and sodium hydroxide. Chitosan could modify the hydrophobicity of the membrane and react with the most negatively charged polymer because it is a cationic polysaccharide [5]. Furthermore, chitosan is expected to increase membrane permeability [6].

MATERIALS AND METHOD

Material Selection

The fabricated membranes in this study made up of polymer, solvent, non-solvent, additives, and surfactant. The polymer and solvent used were polyethersulfone (PES) and n-methyl pyrrolidinone (NMP), respectively, which were bought from Merck. Meanwhile, chemicals purchased from Sigma-Aldrich were pluronic F108 surfactant and chitosan medium molecular weight additive. Other chemicals used were n-hexane and ethanol.

Membrane Preparation

The nanofiltration membranes were fabricated via phase inversion technique, which includes dope preparation and membrane casting. Dope preparation is the process of dissolving the polymer, additive, and surfactant in heated NMP for six to eight hours with the temperature set between 55 °C to 60 °C. The dope was stirred until a homogenous solution obtained. The dope compositions used in this research are tabulated in Table 1.

TABLE 1. Dope component and formulation (weight percentage, wt %)

Dope (M)	PES	NMP	Water	Chitosan (additive)	Pluronic F108 (surfactant)
1	21	72.87	4.13	-	2
2	21	72.37	4.13	0.5	2
3	21	71.87	4.13	1	2
4	21	71.37	4.13	1.5	2
5	21	70.87	4.13	2.0	2

Membrane casting was started after all dissolved bubbles were released from the dope solution. Each dope was poured onto a glass plate and casted using a casting knife to achieve a thickness between 100 µm to 150 µm. The casting process took about 6 seconds to complete at room temperature. Immediately after the casting process, the membrane was placed into tap water for 24 hours to prevent the effects of capillary force. After that, the membrane was immersed in ethanol and n-hexane for 24 hours and 3 hours, respectively.

Membrane Performance and Characterization Evaluation

The fabricated membranes' performance was tested using pure water permeation and rejection tests. Deionized water was used as feed to measure the water flux and calculated over time according to equation (1); Q is the volume of permeates (L), A is the active surface area of membrane (m²) and T is the permeation time (s). In this research, two types of rejection tests were conducted. Salt and oil rejection tests were done to determine the amount of salt and oil rejected using the membrane. The salt rejection feed and permeates were measured using a conductivity meter while the concentration of both feed and permeates of oily wastewater were determined using a UV-visible spectrophotometer. Therefore, equation (2) was used to calculate the retentate whereby C_p and C_f are the solute concentration of permeate and feed, respectively.

$$\text{Pure water permeation (Jw)} = \frac{Q}{A \Delta T} \quad (1)$$

$$\text{Retention of rejection (f\%)} = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \quad (2)$$

Membrane characterization using FTIR spectroscopy was conducted to determine the functional group and chemical composition. Membranes were placed on a sample holder at 255 nm wavelength and results were recorded from 11 to 15 peaks.

RESULTS AND DISCUSSION

Pure Water Permeability

Pure water permeability test, also known as pure water flux, is defined as the water volume that passes through a membrane in time of permeation and area of membrane. The results of different weight percentages of chitosan in dope formulations on pure water flux are explained in Fig. 1 while the membrane formulations are presented in Table 1.

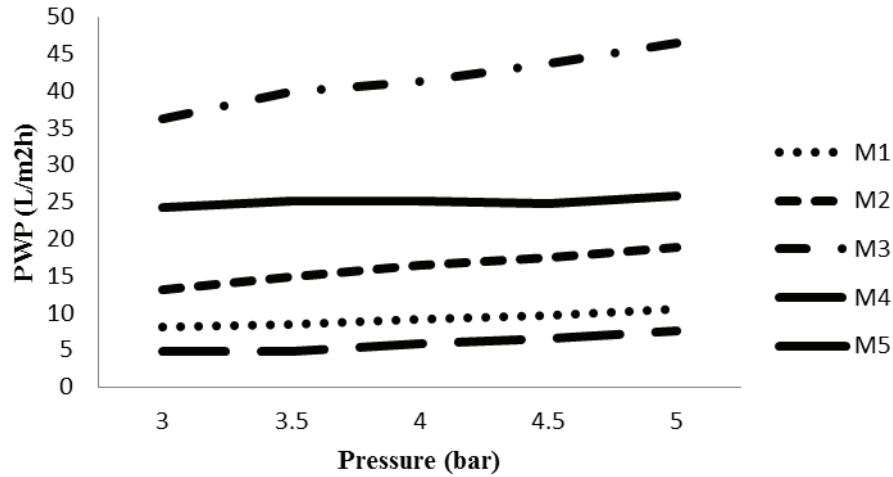
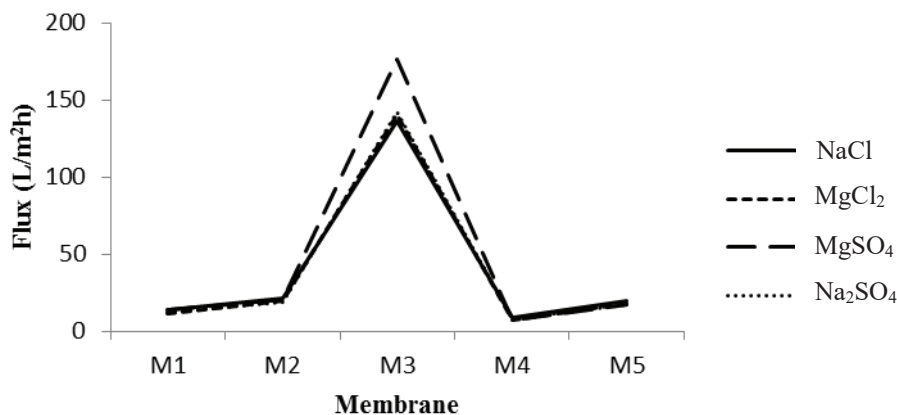


FIGURE 1. Pure water permeation performance on different pressure

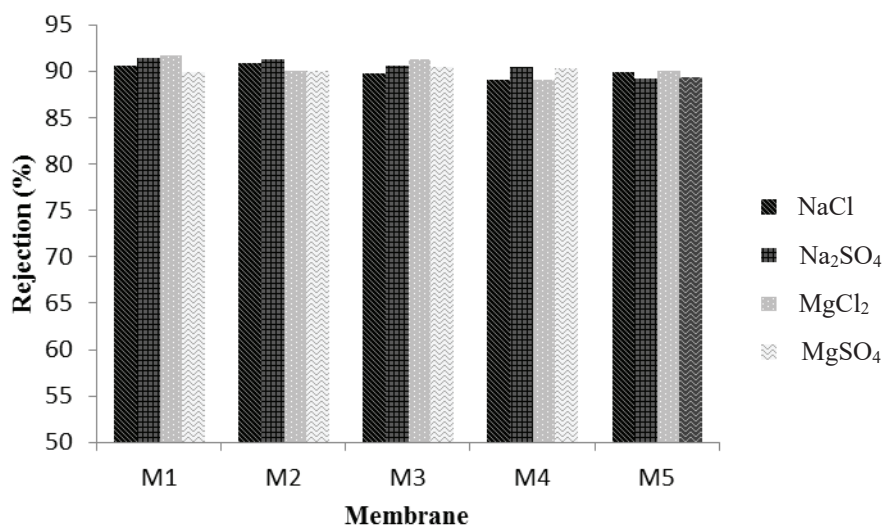
Membranes gave out increment of flux along with pressure, which increased from 3.0 bar to 5.0 bar. The M3 membrane with 1.0 wt% of chitosan had the best pure water flux performance at 46.40 L/m²h compared to the other membranes. As additive plays an important role in improving membrane properties, it enhanced pore connectivity, increased pore formation and introduced hydrophilicity to membranes. However, preparation of chitosan beyond 3 wt% into the membrane formulation is impractical due to high viscosity of the chitosan [7]. In addition, an appropriate amount of chitosan additive can increase membrane permeability and boost its performance [6].

Salt Rejection

Salt rejection test in this study used four types of salts: NaCl, Na₂SO₄, MgCl₂, and MgSO₄ at an operating pressure of 5.0 bar. The result of different amount of chitosan on membrane performance in terms of flux and salt solution rejection is illustrated in Fig. 2.



(a)



(b)

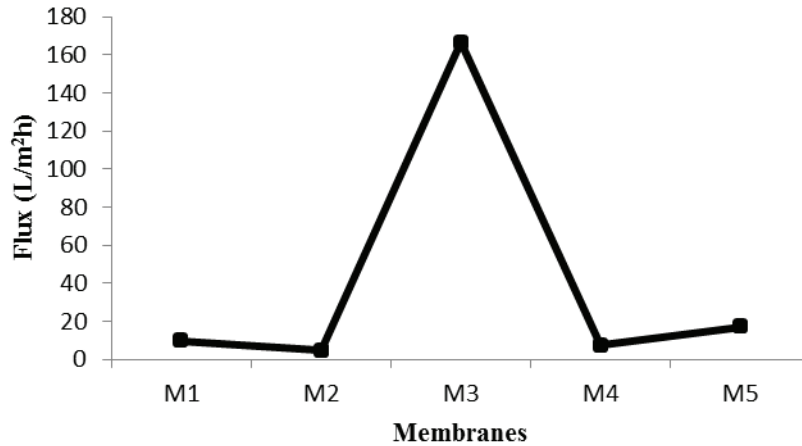
FIGURE 2. a) Salt permeation flux performance on different membrane and b) Salt rejection performance on different membrane

Among the membranes, the highest flux was resulted from M3 membrane for all types of salts tested. It was found that chitosan additives can increase membrane permeability [6]. On the other hand, the increasing amount of chitosan could make the membrane denser and less porous [8]. Nevertheless, M4 and M5 membranes had low salt permeation flux due to increasing amount of chitosan. This is because only the addition of an appropriate amount of chitosan in the membrane dope solution could enhance its permeability.

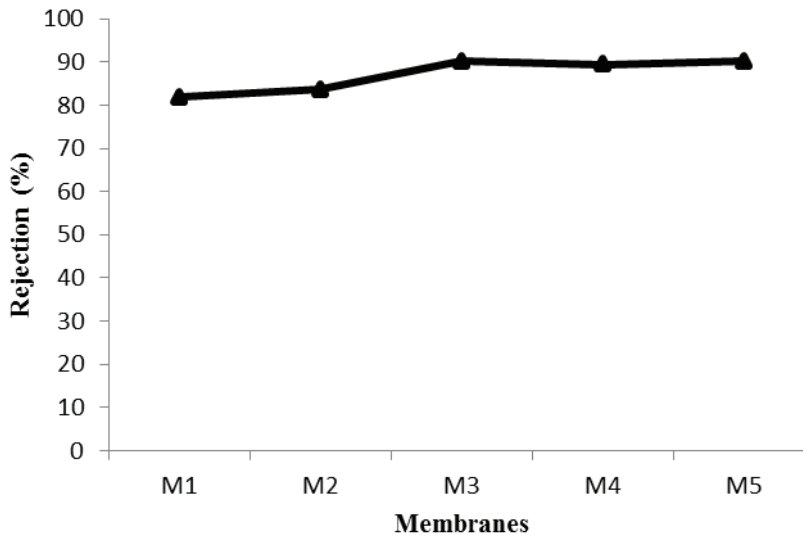
Other than that, the NaCl rejection test showed a very similar trend for all membranes with the highest peak of 90.84% and lowest peak of 89.11% recorded for M2 and M4, respectively. According to Fig. 2 (b), the developed membranes appear to be selective to both monovalent and multivalent salts. Additionally, the membranes show better selectivity towards multivalent salts rather than monovalent salts. Except for M3, other membranes had high salt rejection with low permeation flux. This may be because of M1, M2, M4, and M5 membranes had adsorbed the divalent cation on its negatively charged surface membrane and caused the reduction of the membrane's surface charge.

Oil Rejection

This test was conducted at an operating pressure of 5.0 bar. The feed entered into the testing cell had an oil concentration of about 1.5714 g/L. The resulting permeates were calculated to determine oil rejection percentage as well as permeation flux. The results are presented in Fig. 3.



(a)



(b)

FIGURE 3. a) Effect of different dope formulation to oil permeation flux and b) Effect of different dope formulation to oil rejection performance

The oil rejection test indicated that M3 membrane was plotted as the highest peak at 166.55 L/m²h. The membrane with 1.0 wt% of chitosan additive gave an excellent performance in permeation flux and during both PWP and salt rejection, as an appropriate amount of chitosan could increase membrane permeability and boost its performance [6]. The different amounts of chitosan added to the membrane dope formulation gave different result of oily wastewater permeation flux.

The excellent range of oil rejection percentage was achieved by all membranes. Every membrane achieved about 80% to 90% oil rejection. Whereas, pluronic F108 is a non-ionic surfactant that has a good effect on demulsifying crude oil as it does not leave any counter ions. In addition, the high HLB number of the surfactant contributed to its excellent demulsification abilities [9]. Nonetheless, the small difference between the membranes' rejection performance are resulted from the difference in wt% of chitosan used and the hydrophilicity of the membranes.

Membrane Characterization

FTIR spectrophotometer is a common yet powerful tool used to characterize fabricated membranes. It can identify the types of chemical bonds present in molecules through an infrared absorption spectrum. Figure 4 shows the peaks exhibited by all the membranes.

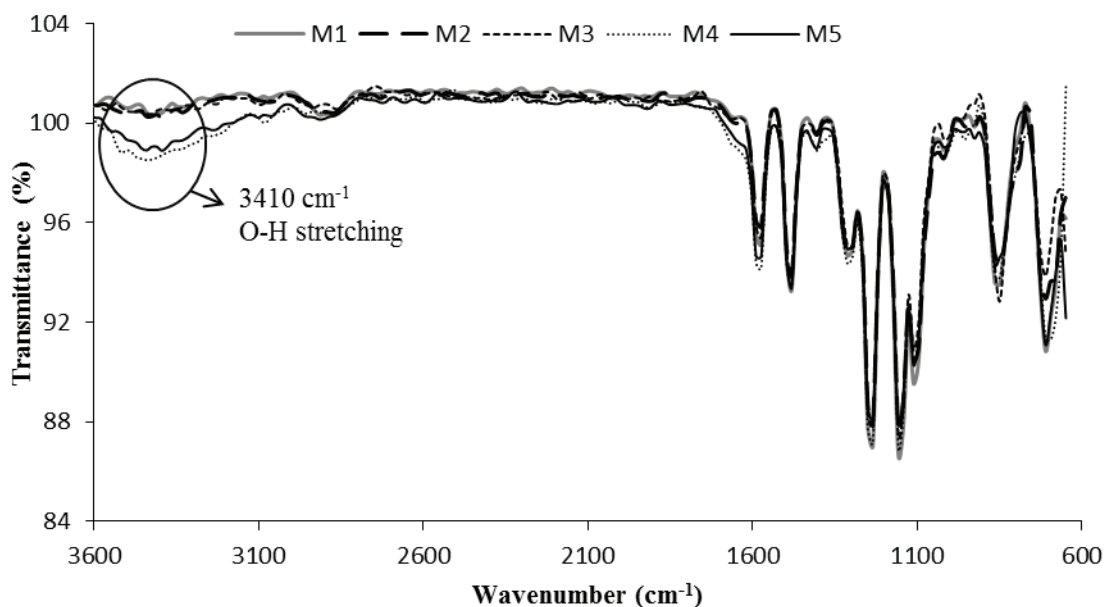


FIGURE 4. FTIR transmittance bands showing the molecular activity of the membranes

As illustrated in Fig. 4, all membranes exhibited a similar IR pattern. Strong absorption was detected at 3410 cm^{-1} , 1485 cm^{-1} , 1307 cm^{-1} , and 1106 cm^{-1} . The presence of chitosan (O-H and NH_2 stretches) on the PES membrane's surface was detected. Thus, M4 and M5 membranes gave broad high intensity O-H and NH_2 stretching compared to M1, M2, and M3 membranes. The presence of fingerprint peaks proves the existence of PES at wavelengths 1307 cm^{-1} (S=O asymmetric stretch) and 1485 cm^{-1} (aromatic stretch). The strong peak at 1106 cm^{-1} indicates the C-O-C stretch due to the blending of PES and pluronic that produced ether vibration. Therefore, the presence of different amounts of chitosan and pluronic F108 surfactant on the fabricated membrane was verified by FTIR spectroscopy.

CONCLUSIONS

This study developed membranes via phase inversion technique and tested with salt solutions and oily wastewater while FTIR spectrophotometer was used to characterize the membranes. Through this study, M3 membrane with 1.0 wt% of chitosan was proved to have the best performance. It had the highest permeation flux for PWP and good rejection percentages for both salt rejection and oil rejection tests as an appropriate amount of chitosan could increase membrane permeability and boost the membrane's performance. Moreover, all membranes had considerably high rejection percentage of salt and oily wastewater due to the presence of pluronic F108 surfactant in the dope formulation. In conclusion, chitosan additive and pluronic F108 surfactant are a good combination for membrane dope formulation as the formulation combination results in high permeation flux and good rejection percentages.

ACKNOWLEDGEMENT

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