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Analysis of Polyvinylidene Fluoride (PVDF) and Polyethersulfone (PES) Ultrafiltration Membranes Incorporated with Graphene Oxide (GO) Performance for Textile Wastewater Treatment

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Abstract: The application of membrane technology to remove pollutant dyes in the textile industry is a significant development today. The modification of membranes to improve their properties has been shown to improve the permeation flux and dye rejection efficiency of the composite ultrafiltration membrane. Objectives for this experiment to fabricate membrane with PVDF and PES membrane incorporated with GO for textile wastewater treatment, to characterize the chemical and physical properties of prepared composite membrane and to analyze the quality of treated dye wastewater treatment. The performance of membrane filtrations is mostly determined by pore structure of the membrane. There are several techniques foe membrane surface characterization. A variety of surface characterization techniques can be used to investigate the surface properties of polymer membranes. As a means of characterising the surface of a polymer membrane, the techniques of Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), atomic force microscopy (AFM), and contact angle measurement are addressed. Furthermore, a variety of tests, such as UV-VIS spectroscopy and FTIR, can aid in the identification of pure compounds. Selection of polymer and solvent in membrane preparation are influences the morphology of fabricated membrane. This research is to study the possibilities that may be realized by using a PVDF and PES as polymer membrane that also included with graphene oxide. The anti-oxidation activity of PVDF, its high thermal stability, its superior organic selectivity and great chemical and mechanical resistance led to show the efficiency to the performance to treat textile wastewater. The Advantages of PES membrane is having good mechanical property, able to conduct in high temperature (up to 200^{o}C), and outstanding resistance with chlorine and risk chemicals, as well as its malleability into various module configurations. Therefore, in this experiment, graphene oxide was used as an additive

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to modify the polyethersulfone (PES) and polyvinylidene fluoride (PVDF) membranes and prepare mixed matrix membranes (MMM). Three different ratios were used to fabricate the membrane which are 1:1, 2:1, and 1:2 of PES and PVDF. For each ratio will be added graphene oxide to see the performance through the rejection. This research is dedicated to using methylene blue as feed dye (50ppm) to study the effect of the composite ultrafiltration membrane performance. The characterization of the membrane was studied using the AFM, SEM, FTIR, contact angle, permeation flux, and dye rejection by investigating the influence of PVDF-PES and GO structure. The performance of the ultrafiltration membranes displayed a lower dye rejection which is lower than 10%. Ratio of 1:2 shown the better rejection compare to others but the others membranes don't show high improvement with the addition of 1g or 0.1% of graphene oxide. Therefore, the composite ultrafiltration membrane does not achieve the expectation for dye rejection.

Keywords: PVDF/PES Composite Membrane, Graphene Oxide, Ultrafiltration, Textile Wastewater Treatment, Water Flux, Dye Rejection

1. Introduction

Membrane technology is one of the promising technologies utilized when dealing with the above-mentioned phenomenon. Membrane technology is known for its high separation efficiency and relatively simple operational processes. Basically, membrane technology refers to the separation of certain particle sizes to pass through the membrane layer and block bigger size particles from exceeding the barrier. Even with the polymeric membrane's disadvantages, researchers are still urged to produce the same kind of membrane, but with a higher performance level. Since membrane methods can clear, concentrate and continually separate textile wastewater, they might be useful in the effluent treatment sector for dye recycling. When compared to other methods of water treatment, membrane treatment produces water of higher quality while using less chemicals. Ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) are traditional membrane processes researched in textile wastewater treatment. The choice of membrane technologies for textile effluent is dependent on costs based on a balance between water flow and solute retention [1].

Graphene oxide composites have a huge surface area and pore volume; they are able to effectively remove pollutants from the environment. Because of this, they have been put to use in the process of eliminating pollutants, particularly in industries such as the textile industry [2]. Structure of graphene oxide molecular consists of carbon, hydrogen, and oxygen. One of the most important traits of GO is that it can be produced using graphite (since it is inexpensive) using different chemical methods, yielding a high production with exceptional cost-efficiency. The second characteristic is that GO is very dispersible in water and can form stable aqueous colloids in order to assemble macroscopic structures with cheaper solution processes.

Graphene oxide (GO) has been selected to its unique properties which can enhance the antifouling properties by increasing permeate flow and selectivity. This nanomaterial has a single atom of thickness and two-dimensional structure and presents exceptional transport properties and versatility, that facilitate its combination with other modifying agents and also polymers used on membranes fabrication and surface modification In addition, GO has specific functional groups on its structure (e.g. epoxy, hydroxyl and carboxyl groups) that can increase the hydrophilicity and negative charge density of the membrane, contributing to the selective behavior that hinders the permeation of contaminants [3].

Condition parameters of dope preparation and membrane fabrication provide a significant role in determining a good structure of asymmetric membrane and consequently the membrane performance. Membrane formulation greatly influenced the UF membrane at the first stage of membrane-making. This factor can alter the membrane morphology, pore size, thickness, molecular weight cut-off and membrane surface charge. Composition of the polymer in membrane solution will affect the

performance of the resultant membrane are conceptually related to its pore size. An optimum transmission can be obtained whenever the size of the solute is smaller relative to the pore size of the membrane. Increase in the polymer concentration produced a denser membrane which led to the reduction of flux and lysozyme transmission. Based on the [4], 15wt.% seems to be an optimum polymer concentration in preparing an ultrafiltration membrane. Thus, this research is to fabricate a series of composite PVDF and PES (18wt.%) membrane with the addition of concentration of GO to measure the performance for textile wastewater treatment.

2. Experimental

2.1 Chemical and materials

1-methyl-2-pyrrolidinone (NMP, purity 99%) was purchased from Merck and used as a solvent. GO and polyvinylpyrrolidone (PVP) were supplied by Sigma Aldrich Reactive Blue 15. Polyethersulfone (PES) granule, 3 mm nominal granule size also supplied by Sigma Aldrich. Polyvinylidene fluoride (PVDF) in pellet form was supplied by Arkema (Xiamen Agency, China), and it was dried at 80 ℃ for 12 hours prior to use. Before usage, the oven was used to dry out both of the materials for a total of 12 hours, however the synthetic dye was the one that was put to use in this investigation by using Methylene blue (MB).

2.2 Preparation of dope solution

The composite PES and PVDF membrane were made by combining 18 wt.%, while the MMM was made by incorporating 0.1 wt.% of GO and 1 wt.% of PVP into 18 wt.% of PVDF in NMP. Both membranes were then used to filter water. In order to prevent the formation of precipitation, the additive was introduced gradually to the solution. After stirring each of the solutions independently for a full 24 h, a homogenous solution was finally achieved. After that, it was sonicated for 4 hours in order to get rid of any bubbles that had developed as a result of the stirring operation. The procedure for preparing the dope selection is shown in Table 2.1. To prepare a flat sheet membrane, 60g of NMP solution was first poured into the Duran Bottle. Then add 1g of PVP and 9g of PVDF into the same bottle. Magneti-bar-stir was put into the same bottle to stir the solution until fully dissolve with the help of hot plate stirrer. Recommended that the temperature of hot plate is below 70^{o}C with the speed of stirrer is 3rpm and above. After the solution in the Duran bottle fully dissolve around 24h, 9g of PES and the remaining of NMP which is 21g was added into same Duran bottle and put again on the hot plate stirrer to dissolve the solution. Different way used to prepare the solution that have additive of GO. After solution that mixed NMP, PVP and PVDF, add 9g of PES, 20.9g of remaining NMP and add 0.1g of GO. Then put the Duran bottle on the hot plate stirrer with the recommend of temperature is below 30^{o}C. Same way to prepare the flat sheet membrane with different ratio of PVDF and PES.

Table 2.1: Dope solution for preparation of composite UF membranes

Membrane	Ratio	PVDF (wt.%)	PES (wt. %)	PVP (wt. %)	GO (wt. %)	NMP (wt. %)	Total (wt.%)
a(i)	1:1	9	9	1	0	81	100
a(ii)	1:1	9	9	1	0.1	80.9	100
b(i)	2:1	12	6	1	0	81	100
b(ii)	2:1	12	6	1	0.1	80.9	100
c(i)	1:2	6	12	1	0	81	100
c(ii)	1:2	6	12	1	0.1	80.9	100

2.3 Preparation of flat sheet membrane

Once the solution was prepared, casting process can begin with put the dope solution onto a smooth and clean glass plate. The solution was then cast by a roller glass to form a membrane with the size range of the glass plate. The cast film, together with glass plate, were then immersed into a water bath to allow phase inversion to take place. Once the membrane was peeled off naturally from the glass plate,

then transferred to another water bath and immersed for 1 day to remove residual solvent then dried the membrane in the room temperature.

2.4 Membrane characterization

2.4.1 Characterization by Atomic force microscopy (AFM)

A scanning probe microscope (XE-100) was used to characterize the surface roughness of the prepared membrane. A membrane sample was cut about 1.5cm x 1.5cm and attach to the glass sample to scan the surface.

2.4.2 Characterization by Fourier transform infrared spectroscopy (FTIR)

A Fourier transform infrared spectrometer (PerkinElmer Spectrum 100 FT-IR spectrometer) was used to check the chemical structure of the membrane. It was characterized by FTIR with a resolution of $4cm^{-1}$ in the range of 600 - 4000 cm^{-1}.

2.4.3 Characterization by Scanning electron microscopy (SEM)

A scanning electron microscopy (Hitachi SU1510 SEM) was used to examine the top membrane surface morphology. Membrane samples were prepared by sputtering the coating of Titanium to increase the signal-to-noise ratio during SEM imaging and therefore produce better quality images.

2.4.4 Characterization by Contact angle

VCA Optima-AST model was used to measure the hydrophilicity of the membrane. A membrane sample was prepared about 1.5cm x 1.5cm and attach to the sample glass. When a drop of liquid is placed on a dry membrane surface, the contact angle is developing between the membrane surface and the liquid interface. To achieve optimal accuracy, at least three contact angle measurements at different locations for each membrane sample were recorded, and an average was calculated.

2.5 Pure water flux

In order to ascertain the amount of clean water that can pass through the membranes, the cross-flow membrane system was used. In light of this, each membrane was compressed at a pressure of 300 kPa or 3 bar for a period of 30 mins until a steady-state condition was reached before the flux measurement was carried out. After that, the pure water flow of the membrane was calculated using the equation below. The results are shown in units ($J_w = \frac{L}{m^2 h}$).

$$J_w = \frac{Q}{A \times \Delta T} \quad Eq.1$$

2.6 Dye rejection

A strategy employing a cross-flow study was presented as a method for determining the rate dye rejection. The equation that follows may be used to assess how well the membrane is able to separate dye molecules.

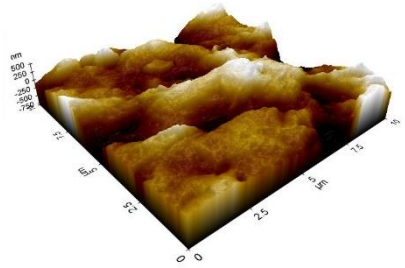
$$R = \left(1 - \frac{C_p}{C_f} \times 100\right) \quad Eq.2$$

R is the ultrafiltration rejection percentage in percent, C_p is the permeate concentration in percent, and C_f is the feed concentration in percent. R, C_p, and C_f (percent). Both the feed and the permeate samples' dye content could be detected using a UV–vis spectrophotometer at a wavelength of 664 nm. The dye solutions were created at 50 ppm which is ($\frac{3g}{L}$) concentrations.

3. Results and Discussion

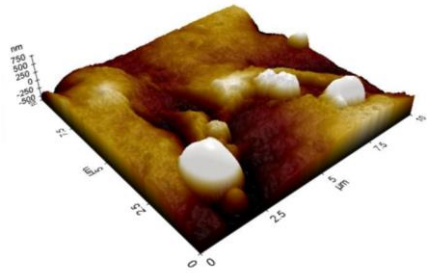
3.1 Surface roughness (AFM)

(a) PVDF: PES without GO / a(i)



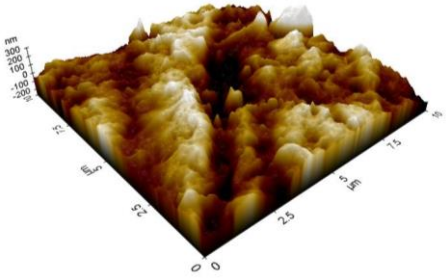
R_a =134.233 nm

(b) PVDF: PES with GO / a(ii)



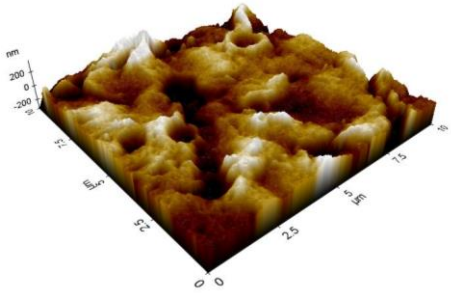
R_a =127.353 nm

(c) 2PVDF: PES without GO / b(i)



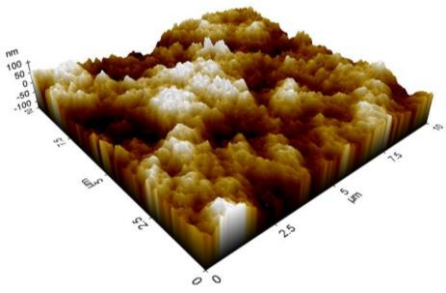
R_a =55.529 nm

(d) 2PVDF: PES with GO / b(ii)



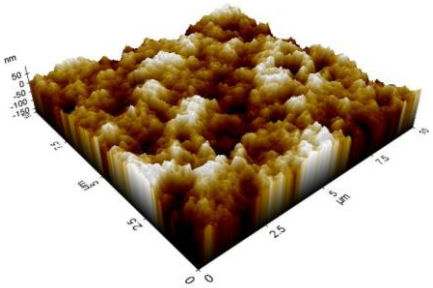
R_a =56.725 nm

(e) PVDF:2PES without GO / c(i)



R_a =23.976 nm

(f) PVDF:2PES with GO / c(ii)



R_a =21.288 nm

Figure 3.1: AFM topographic images of UF membranes (a) PVDF: PES without GO / a(i), (b) PVDF: PES with GO / a(ii), (c) 2PVDF: PES without GO / b(i), (d) 2PVDF: PES with GO / b(ii), (e) PVDF:2PES without GO / c(i), (f) PVDF:2PES with GO / c(ii)

Figure 3.1 shows the topographic AFM images of all membranes. From the analysis, it is observed that the membrane PVDF:2PES based composite UF membranes exhibits the greatest mean surface roughness value compared to other fabricated membranes. The surface membrane roughness change when the ratio of PES or PVDF is increase. But between the PVDF:2PES with GO and PVDF:2PES without GO, with GO has the greatest surface roughness compared to without GO. It is because it could be due to the fast exchange between solvent and non-solvent during the phase inverse process that

impacts the porosity in the membrane [5]. With the concentration of PES increased (from 6g to 12g), more compact and thicker of ultrafiltration membrane layer is formed, resulting in rougher surface of the composite UF membranes.

3.2 Organic functional groups (FTIR)

Infrared (IR) spectroscopy is an absorption method widely used in both qualitative and quantitative analyses. The infrared region of the spectrum includes electromagnetic radiation that can alter the vibrational and rotational states of covalent bonds in organic molecules. Shown in Figure 3.2 is the IR spectrum for membrane b (i) which are the present of PES and PVDF. Figure 3.2 shows the FTIR spectrum of PVDF and PES. There are several characteristics bands of PES membrane that can be identified including aromatic C=C band at 1450 cm^{-1} and 1580 cm^{-1}, aromatic ether (-C-O-C) at 1240 cm^{-1} and asymmetric/symmetric stretching of O=S=O at 1320 cm^{-1} and 1162 cm^{-1} [6].

The IR spectra of PVDF are also shown in the Figure 3.2. PVDF exhibited distinct characteristics peaks assigned to -CH_2 bending (1400 cm^{-1}) and amorphous phase absorption (876 and 839 cm^{-1}).

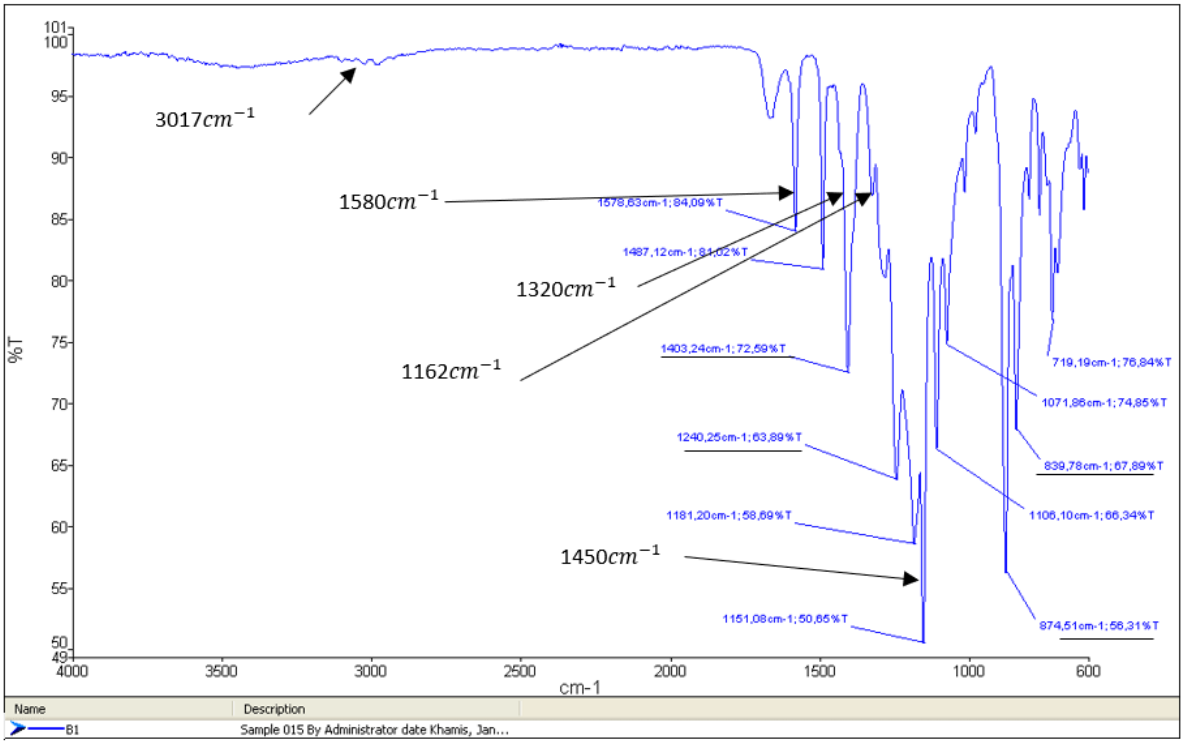


Figure 3.2: IR spectrum for 2PVDF: PES without GO / b (i)

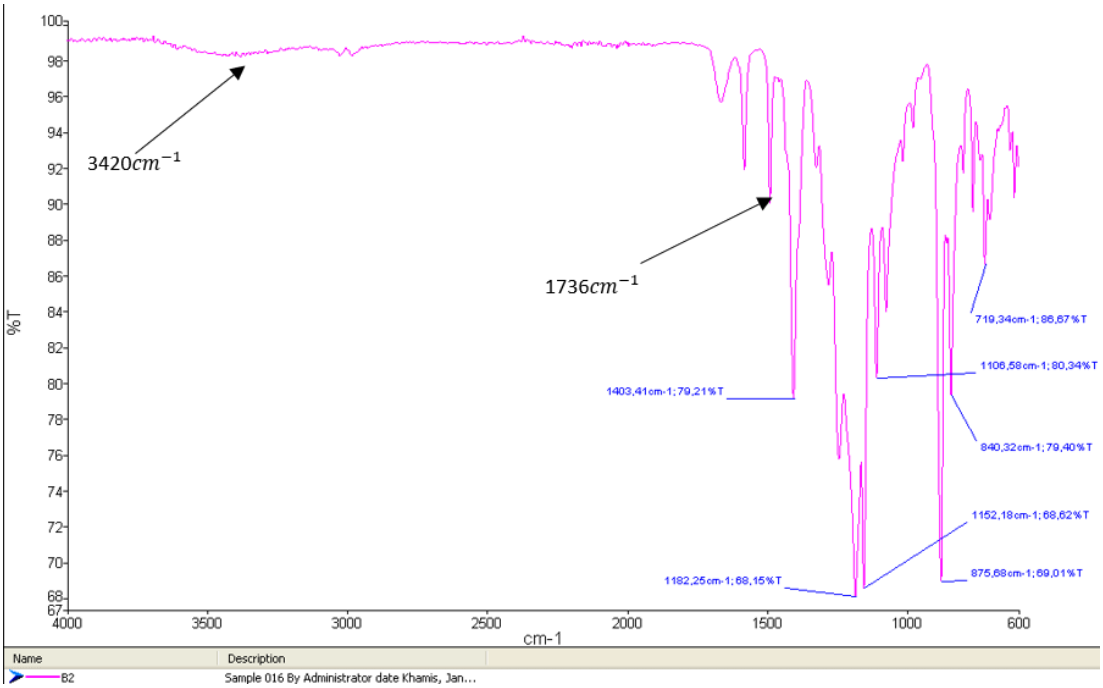


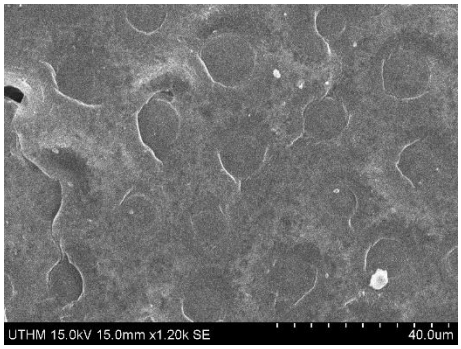
Figure 3.3: IR spectrum for 2PVDF: PES with GO / b (ii)

Figure 3.3 shows the graph IR spectrum for b (ii) which is membrane 2PVDF: PES with GO / b (ii). The FTIR spectrum of GO revealed characteristic peaks at around 3420 and 1736 cm^{-1} corresponding to O-H and C=O stretching frequencies of the -COOH group respectively. The peaks corresponding to aromatic C=C bending, phenolic C-O stretching and epoxy C-O-C stretching were identified at 1637, 1222 and 1050 cm^{-1} respectively.

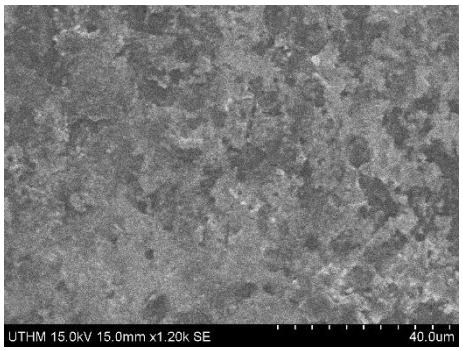
3.3 Surface morphology (SEM)

Figure 3.4 presents the SEM images of top surface of each membrane made with different ratio of PVDF and PES. It is seen that the pores of PVDF: PES without GO / a(i) were evenly distributed throughout the membrane surface and could be easily observe when examined at a magnification of 1200. Due to the decrease in pore size of others membrane surface, these pores were unable to be seen at the same magnification. The decrease in pore diameter of membrane could be explained by the delayed solvent of NMP and non-solvent (water from the coagulation bath) exchange rate during the phase inversion process which resulted from higher viscosity of dope solution used [7].

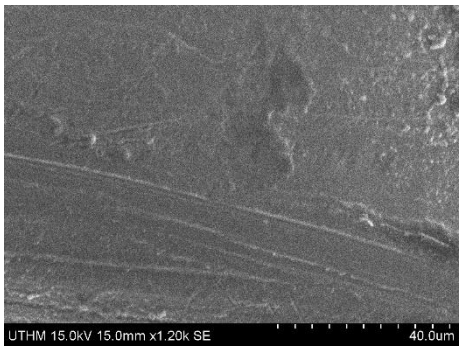
(a) PVDF: PES without GO / a(i)



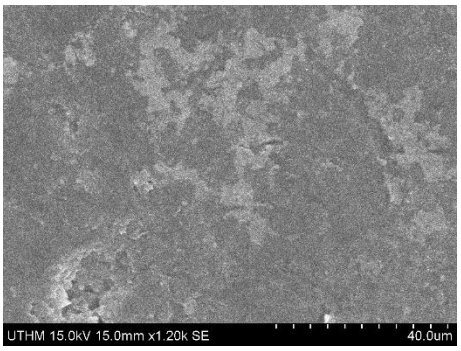
(b) PVDF: PES with GO / a(ii)



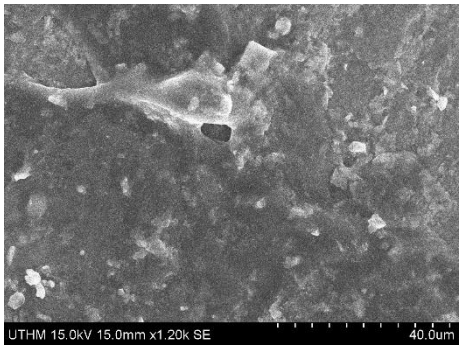
(c) 2PVDF: PES without GO / b(i)



(d) 2PVDF: PES with GO / b(ii)



(e) PVDF:2PES without GO / c(i)



(f) PVDF:2PES with GO / c(ii)

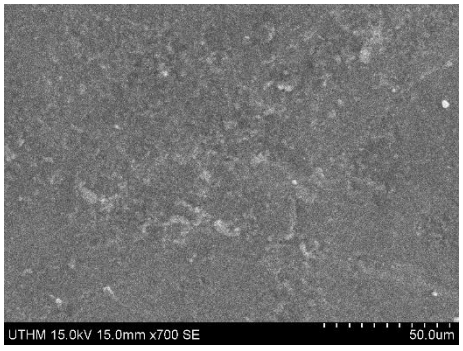


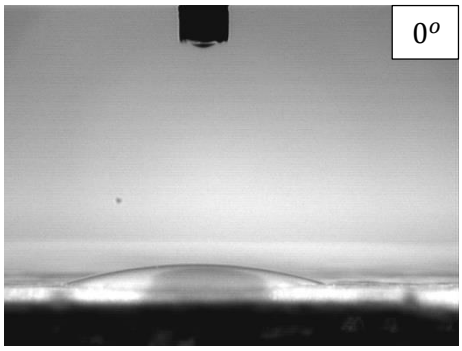
Figure 3.4 SEM images of top surface UF membranes (a) PVDF: PES without GO / a(i), (b) PVDF: PES with GO / a(ii), (c) 2PVDF: PES without GO / b(i), (d) 2PVDF: PES with GO / b(ii), (e) PVDF:2PES without GO / c(i), (f) PVDF:2PES with GO / c(ii)

3.4 Surface wettability of the membranes (Contact angle)

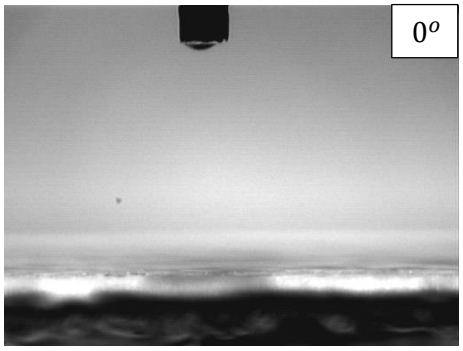
As shown in the Figure 3.5, the contact angle of the membranes reported lower than 90^{o} which is hydrophilic surface, a (i) and a (ii) shown that there is no contact angle because of several defect of membrane. Contact angle of the membrane with no addictive of graphene oxide is small compared to the membrane that have addictive with the graphene oxide such as membrane c. Membrane c (i) has 55.40^{o} while c (ii) has 64.70^{o}. Different with membrane b where the contact angle of b (i) is higher than b (ii) which are b (i) has 74.70^{o} and b (ii) has 68.70^{o}. It should be with the presence of PVDF, the contact angle of the membranes should become hydrophobic surface because the presence of PVDF which indicates of the crystalline nature. A large quantity of spherical beads with diameters ranging from 0.3um to 1.0um adhered to the membrane surface. The bicontiuous structure and the presence of the

spherical beads significantly improved the hydrophobicity of the PVDF membrane surface. But with some factors have caused membrane defects. One of the factors is concentration of hydrophilic (PES and GO) material is higher than hydrophobic (PVDF). With the addictive of the GO which is high hydrophilic nature promote high potential to adsorb and store water molecules due to the oxygen-containing functional groups on its hydrophilic surface.

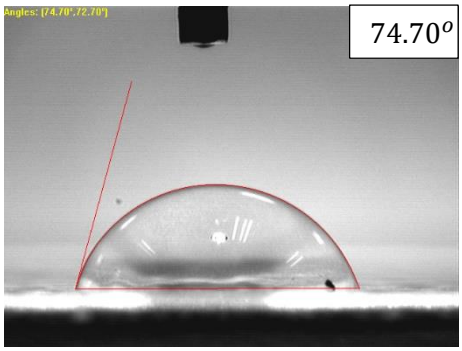
(a) PVDF: PES without GO / a(i)



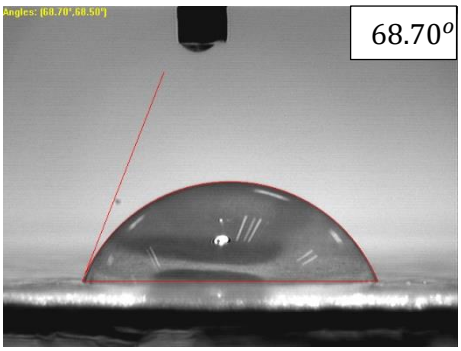
(b) PVDF: PES with GO / a(ii)



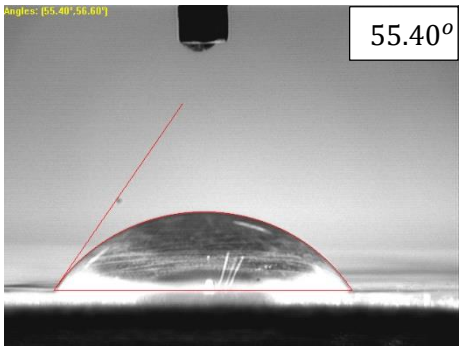
(c) 2PVDF: PES without GO / b(i)



(d) 2PVDF: PES with GO / b(ii)



(e) PVDF: 2PES without GO / c(i)



(f) PVDF: 2PES with GO / c(ii)

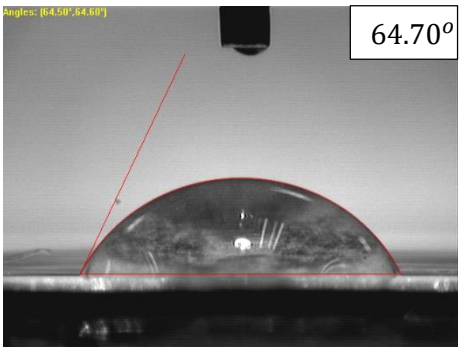


Figure 3.5: Contact angle water droplet on UF membrane (a) PVDF: PES without GO / a(i), (b) PVDF: PES with GO / a(ii), (c) 2PVDF: PES without GO / b(i), (d) 2PVDF: PES with GO / b(ii), (e) PVDF:2PES without GO / c(i), (f) PVDF:2PES with GO / c(ii)

3.5 Permeate flux

Based on the Table 3.1 shown the flux of the pure water and dye waste water. With the respect to water flux and surface of the membrane, it is also found that both properties were influenced by the polymer concentration used in preparing substrates. Table below shows the effect of PVDF to PES concentration with or without GO on the membrane properties with respect to pure water permeability

and dye permeability. Refer to discussion on contact angle, the value for permeate flux on the UF membrane a(i) and a(ii) was reasonable because of the higher rate value. The membrane shown same trend for the pure water flux and the dye wastewater flux. With the addition of concentration 2PVDF to PES without GO, the trend of pure water flux decreases to 7502.89 L/m^2h but increases when the membrane added with GO to 9107.63 L/m^2h. For the flux of dye wastewater, the trend shown the opposite where the value permeate flux b(ii) was lower than b(i) same goes to UF membrane c. With the increasing of polymer concentration, revealing that the flux reduction in polymer membrane could also be due to the decrease in surface hydrophilicity.

Table 3.1: Data of permeate pure water flux and dye wastewater flux

Membrane	Ratio PVDF to PES concentration	Flux(L/m^2h)	
		Pure water	Dye wastewater
a(i)	1: 1	20834.20	13559.69
a(ii)	1: 1	21014.58	9335.32
b(i)	2: 1	7502.89	1094.31
b(ii)	2: 1	9107.63	865.30
c(i)	1: 2	17153.25	8010.51
c(ii)	1: 2	19653.32	1019.61

The graph in the Figure 3.6 and Figure 3.7 shown the pure water flux against the ratio of PVDF to PES concentration without and with GO and dye wastewater flux against the ratio of PVDF to PES concentration without and with GO The trend of both graphs shown not the same trend because of the different feed solution which are pure water and dye solution. Both of solution have different concentration and it might effect of the permeate flux result.

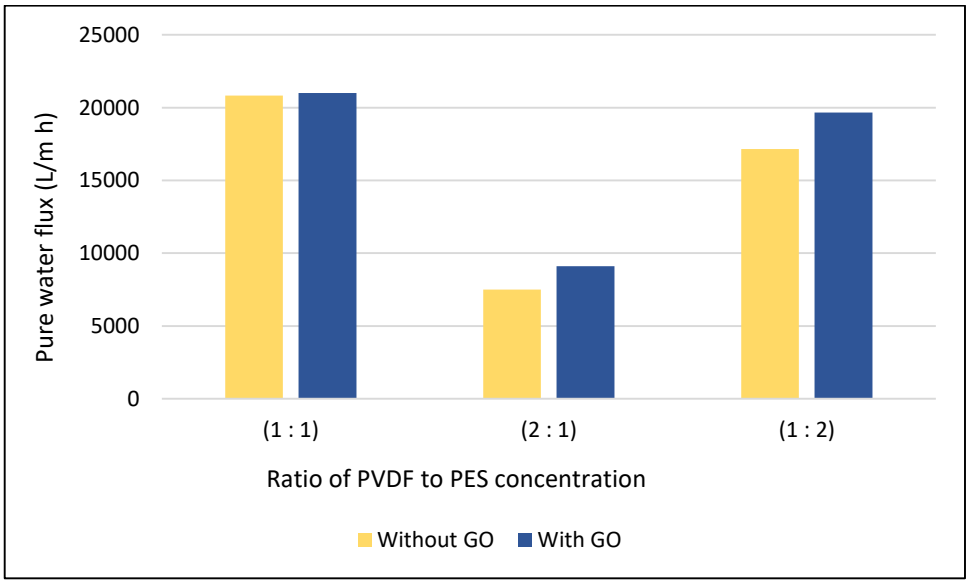


Figure 3.6: Pure water flux (L/m^2h) against Ratio of PVDF to PES concentration without and with GO

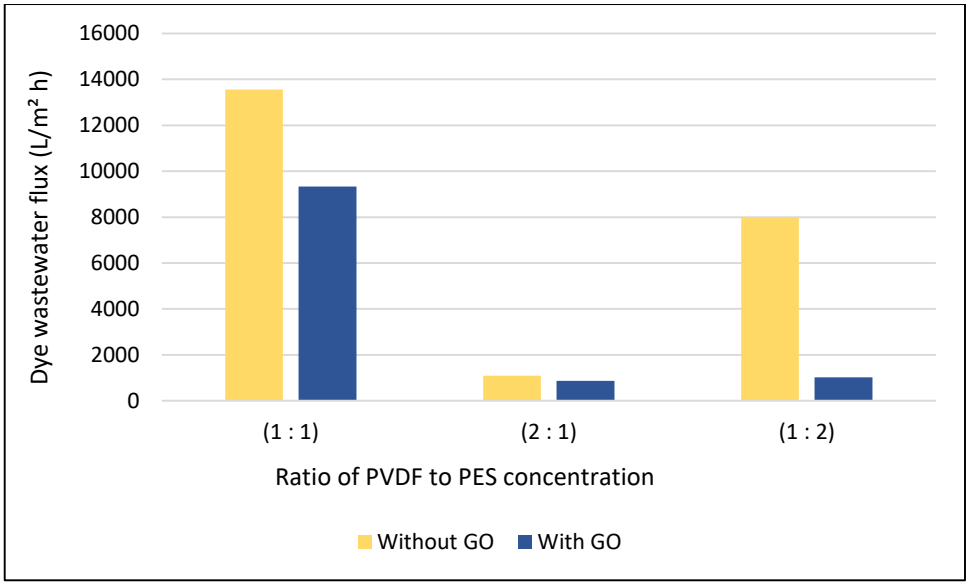


Figure 3.7: Dye wastewater flux (L/m²h) against Ratio of PVDF to PES concentration without and with GO

3.5 Dye rejection

Based on the Table 3.2, membrane that most have dye rejection are membrane PVDF: 2PES with GO / c (ii) which is 8.54 %. Followed by c (i), b (ii), a (ii), b (i) and a (i). Percent of dye rejection for the membrane are 8.35 %, 7.82 %, 7.32 %, 6.88%, and 1.54 % respectively. Figure 3.8 below shown the trend between the dye rejection and the ratio of PVDF to PES concentration without and with GO. With the addition of GO incorporated in the membrane helped to improve the surface hydrophilicity which further promoted the interactions between the membrane surface and the solution. Moreover, the addition of this organic material helps to increasing the dye rejection. With the high concentration of GO and the ratio of PVDF to PES in producing membrane, it will be resulted high dye rejection on dye wastewater treatment. Membranes with higher water flux exhibited a lower rejection, supporting the trade-off relationship between flux and rejection.

Table 3.2: Data of dye rejection with the ratio of PVDF to PES concentration

Membrane	Ratio of PVDF to PES concentration	Dye rejection, R (%)
a(i)	1: 1	1.54
a(ii)	1: 1	7.32
b(i)	2: 1	6.88
b(ii)	2: 1	7.82
c(i)	1: 2	8.35
c(ii)	1: 2	8.54

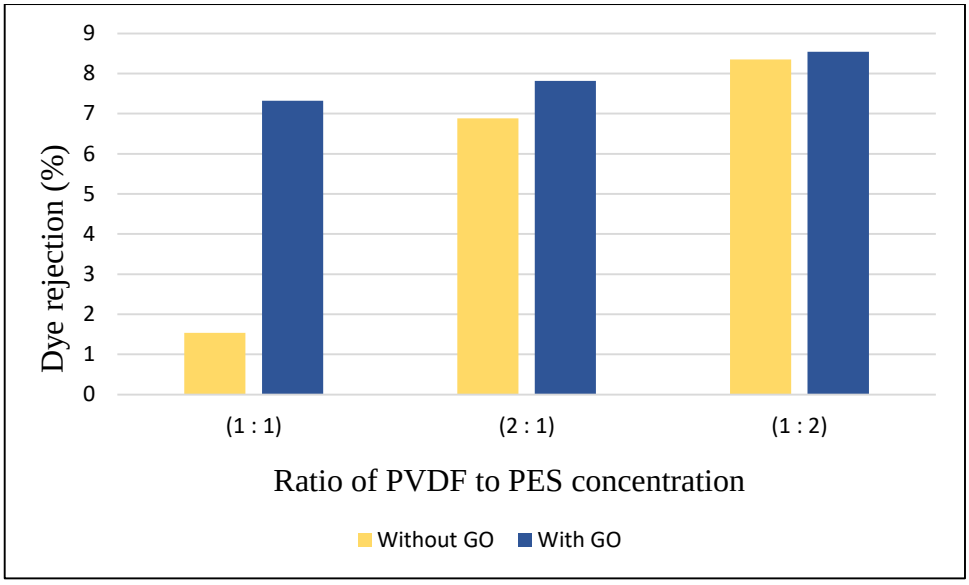


Figure 3.8: Dye rejection (%) against ratio of PVDF to PES concentration without and with GO

These results obtained were mainly influenced by the varied morphology of layer formed over the different ratio of polymer. The highest rejection reported in ratio 1:2 of PVDF and Pes is most likely due to significant increase in selective layer thickness, creating additional resistance for water transport through the membrane. Based on the AFM and SEM analysis, convinced that the physical characteristics of membrane layer such as layer thickness and surface roughness could be obviously altered using different ratio which as a result affected the water and dye permeation rate. It must be emphasized that the findings obtained in this study might differ if the substrate or ratio is made of another type of polymer and/or with the presence of the additives.

4. Conclusion

The objective of this study to fabricate membrane with a PVDF and PES membrane incorporated with graphene oxide (GO) for textile wastewater treatment was found successful but unsuccessful to gain a good result in characterize the chemical and physical properties of composite membrane and to analyst the quality of treated dye wastewater treatment. This experiment was divided into 3 different ratios 18 wt.% of NMP and PES. For the first ratio was 1:1 followed by 2:1 and 1:2 of NMP and PES solution and each of the ratio was added with 0.1g of graphene oxide. There are six total of different composite membrane to test the performance of textile wastewater treatment. According to the performance of dye rejection by using composite ultrafiltration membrane, a membrane with the ratio 1:1 without graphene oxide (PVDF: PES without GO/a(i)) showed less rejection compared to other membranes without GO which is 1.54%. Followed by the ratio (2PVDF: PES without GO / b (i)) and (PVDF: 2PES without GO / c (i)) which the value is 6.88% and 8.35% respectively. To form a better membrane, graphene oxide was used as an additive to NMP and PES. GO are capable of forming a perfect barrier when dealing with liquids. It can effectively separate the organic solvent from water to an exceptional level. With the same ratio, GO was added to see the capability of the composite ultrafiltration to test the rejection of dye wastewater. With the addition of GO, the dye rejection showed the value of a small change with the same order of membrane which are 7.32%, 7.8% and 8.54% respectively. This analysis shows that all six membranes are not capable to treat the dye wastewater because the rate of rejection is below 10%. Unsuccessfully to treat the quality of dye wastewater because of many factors, especially when fabricating the membranes. The main factor can be observed when the phase inversion process of the membrane is submerged in a tab water bath. The combination of phase separation and mass transfer affects the membrane structure. During the immersion phase inversion process, the kinetic and thermodynamic properties of the casting solution are the two key aspects that influence the morphology and properties of the resulting membranes. When the membranes

are defective in the beginning, the next process will be affected when the membrane is used to filtrate the dye solution. All the composite ultrafiltration membrane was testing their characteristic. From the contact angle and SEM test, it can be seen that the membranes were defects because the contact angle result shows that there is no contact angle for membrane PVDF: PES without GO and with GO. Other than that, the SEM has shown the large pore size from the membrane that can look like a hole. It can be concluded that probability the membrane is leaking and it cannot be seen with the contact angle equipment.

Last but not least, unsuccessful of this experiment was because of the cross and tangential flow test cells which are the equipment that tests the performance of the flat sheet membrane. From the observation of the equipment, it has leaked in several parts. It might be affecting the flow of the dye solution. When the part where the membrane is placed has leaking, so the membrane will not fully function to filtrate the dye solution and resulting in low rejection of dye.

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References

[1] Bubert, H., Rivière, J. C., Arlinghaus, H. F., Hutter, H., Jenett, H., Bauer, P., & Friedbacher, G., Surface and Thin-Film Analysis, Ullmann's Encyclopedia of Industrial Chemistry, 2000.

[2] Johari, N. A., Yusof, N., Lau, W. J., Abdullah, N., Salleh, W. N. W., Jaafar, J., . . . Ismail, A. F. (2021). Polyethersulfone ultrafiltration membrane incorporated with ferric-based metal-organic framework for textile wastewater treatment. Separation and Purification Technology, 270, 118819. https://doi.org/10.1016/j.seppur.2021.118819

[3] Akhtar, K., Khan, S. A., Khan, S. B., & Asiri, A. M. (2018). Scanning Electron Microscopy: Principle and Applications in Nanomaterials Characterization. Handbook of Materials Characterization, 113–145. https://doi.org/10.1007/978-3-319-92955-2_4

[4] Sofiah, H., Nora`aini, A., & Marinah, M. (2010). The Influence of Polymer Concentration on Performance and Morphology of Asymmetric Ultrafiltration Membrane for Lysozyme Separation. Journal of Applied Sciences, 10(24), 3325–3330. https://doi.org/10.3923/jas.2010.3325.3330

[5] Ammar, A., Al-Enizi, A. M., Al-Maadeed, M. a. A., & Karim, A. (2016). Influence of graphene oxide on mechanical, morphological, barrier, and electrical properties of polymer membranes. *Arabian Journal of Chemistry*, *9*(2), 274–286. https://doi.org/10.1016/j.arabjc.2015.07.006

[6] Kouhestani, F., Torangi, M. A., Motavalizadehkakhky, A., Karazhyan, R., & Zhiani, R. (2019). Enhancement strategy of polyethersulfone (PES) membrane by introducing pluronic F127/graphene oxide and phytic acid/graphene oxide blended additives: preparation, characterization and wastewater filtration assessment. *Desalination and Water Treatment*, *171*, 44–56. https://doi.org/10.5004/dwt.2019.24769

[7] Dong, X., Lu, D. S., Harris, T. a. L., & Escobar, I. C. (2021). Polymers and Solvents Used in Membrane Fabrication: A Review Focusing on Sustainable Membrane Development. *Membranes*, *11*(5), 309. https://doi.org/10.3390/membranes11050309

[8] Akhtar, K., Khan, S. A., Khan, S. B., & Asiri, A. M. (2018). Scanning Electron Microscopy: Principle and Applications in Nanomaterials Characterization. Handbook of Materials Characterization, 113–145. https://doi.org/10.1007/978-3-319-92955-2_4

[9] Johari, N. A., Yusof, N., Lau, W. J., Abdullah, N., Salleh, W. N. W., Jaafar, J., . . . Ismail, A. F. (2021). Polyethersulfone ultrafiltration membrane incorporated with ferric-based metal-organic framework for textile wastewater treatment. Separation and Purification Technology, 270, 118819. https://doi.org/10.1016/j.seppur.2021.118819

[10] Kadhim, R. J., Al-Ani, F. H., Al-shaeli, M., Alsalhy, Q. F., & Figoli, A. (2020). Removal of Dyes Using Graphene Oxide (GO) Mixed Matrix Membranes. Membranes, 10(12), 366. https://doi.org/10.3390/membranes10120366

[11] Adane, T., Adugna, A. T., & Alemayehu, E. (2021). Textile Industry Effluent Treatment Techniques. Journal of Chemistry, 2021, 1–14. https://doi.org/10.1155/2021/5314404

[12] Patel, A., Arkatkar, A., Singh, S., Rabbani, A., Solorza Medina, J. D., Ong, E. S., Habashy, M. M., Jadhav, D. A., Rene, E. R., Mungray, A. A., & Mungray, A. K. (2021). Physico-chemical and biological treatment strategies for converting municipal wastewater and its residue to resources. Chemosphere, 282, 130881. https://doi.org/10.1016/j.chemosphere.2021.130881

[13] Natália Cândido Homem, Natália de Camargo Lima Beluci, Sara Amorim, Rui Reis, Angélica Marquetotti Salcedo Vieira, Marcelo Fernandes Vieira, Rosângela Bergamasco, Maria Teresa Pessoa Amorim (2019), Surface modification of a polyethersulfone microfiltration membrane with graphene oxide for reactive dyes removal, Applied Surface Science, Volume 486, Pages 499-507, (https://www.sciencedirect.com/science/article/pii/S0169433219313029

[14] Parvin, F., Islam, S., Zakia Urmy, & Akm Saiful Islam. (2020, July 7). A Study on the Solutions of Environment Pollutions and Worker's Health Problems Caused by Textile... ResearchGate; unknown. https://www.researchgate.net/publication/355778898_A_Study_on_the_Solutions_of_Environment_Pollutions_and_Worker's_Health_Problems_Caused_by_Textile_Manufacturing_Operations