

Antifouling Modified Membrane For Effective Oil-Water Separation

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ABSTRACT

Today, numerous businesses produce significant amounts of wastewater that is greasy. Separating the stable emulsified oil particles from water is the fundamental difficulty in treating oily wastewater. Generally speaking, polymeric membranes are essential in these procedures due to how simple and inexpensive modern separation processing is, as well as how flexible they are. The phase inversion method utilized in this study to create a poly vinyl alcohol (PVC) membrane is demonstrated. In order to increase both permeation flow and fouling resistance, PVC has been modified by the addition of polymeric additives like polyvinylpyrrolidone (PVP). Additionally, in this paper, we compare lab chemical membrane and commercial chemical membrane on the basis of their fluxes, rejection, and characterization. The enhanced PVC/PVP membranes were characterized and evaluated using mechanical strength, porosity, scanning electron microscopy (SEM), Fourier Transform Infrared (FTIR), and water contact angle measurement. The membranes were subsequently tested at a lab size in a cross-flow system with synthetic greasy wastewater as the input.

Keywords: Oil/water separation, UF membrains, Membrane modification

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1 INTRODUCTION

In reality, the release of oily wastewater into the environment has a number of harmful effects, including the contaminating of surface water, ground, marine, and soil pollution, as well as air pollution brought on by the evaporation of oil into the atmosphere and the presence of hydrocarbons. [1, 2]. Oily wastewaters have oil particles that fall into one of three sizes categories. Free oil-water mixtures (>150 mm), oil-water dispersions (20-150 mm), and oil/water emulsions (20 mm) can all be classified as oil/water mixtures depending on the size of the dispersed phase.[3]. Utilizing membranes as one of the advanced separation methods since The employment of membrane technology in numerous industrial fields, including the manufacture of

ultra-pure water, water desalination, product recycling, and wastewater treatment, has garnered significant interest during the past 30 years. [4]. Ultrafiltration (UF), one of many membrane technologies, has been known as an astonishing technique in the wastewater systems of refineries due to its capacity to extract emulsified oil droplets and other

organic contaminants. Typically, its pore sizes range from 2 to 50 nm. [5]. Internal adsorption, pore-blocking deposition, a cake-like layer of deposition on the membrane surface, and other factors can all contribute to membrane fouling. [6]. Carbon membranes, ceramic membranes, and polymer membranes are just a few of the several types of membranes that can be categorized. In general, polymeric membranes play a crucial role in those procedures because of their simple, low-cost manufacturing and excessive flexibility [7].

Antifouling membranes have been prepared using three methods: organic nanoparticles being incorporated into the membrane matrix [8, 9], deformation of the membrane's surface [10, 11], and combining various polymers [12, 13]. One of these strategies is blending polymers together since it is the most practical and convenient technique to improve the antifouling capabilities and performance of polymeric membranes. It is also the most practical from an operational and financial standpoint [14, 15]. From a financial and operational perspective, it is also the most sensible. [14, 15]. Polyvinyl chloride (PVC) has gained a lot of attention recently for the creation of oily wastewater treatment membranes due to its intriguing physical, chemical, and thermal stabilities, superior mechanical strength, long lifespan, low cost, and solubility in a variety of solvents like tetrahydrofuran (THF), dimethylformamide (DMF), N-methyl pyrrolidone (NMP), and N,N-dimethylacetamide (DMAc) [16, 17]. PVP additive is added to PVC-based polymers to enhance the membranes' properties. Actually, the membranes' permeability is increased by the addition of a PVP additive with a very comparable pore size distribution [18]. An increase in pore density, a reduction in the dense layer's effective thickness brought on by macrogaps in the support layer, and an increase in the hydrophilicity of the membrane and pore surfaces could all be to blame for this. Given the low cost and antifouling properties of membranes, the study's objective is to offer a practical method for oil-water emulsion separation.

2. EXPERIMENTAL WORK

2.1. Materials, Membrane preparation and Oil/water emulsion preparation.

Table (2.1): Properties of chemical Used

Chemicals	Compound	Mwt.
Polyvinyl chloride (PVC)	C_2H_3CL	3000-4000
Polyvinyl Pyrrolidone (PVP)	C_6H_9NO	24000
N, N-Dimethyl formamide (DMF)	C_2H_7NO	73.09
Sodium laury sulphate (SLS)	$NaC_{12}H_{25}SO_4$	288.38
Citric acid	$C_6H_8O_7$	36
Sodium hydroxide	$NaOH$	40

PVP as a pore former in a composition of 3 wt% with Mwt equal to 24000 g/mol and PVC as the base polymer in a composition of 17 wt% with Mwt equal to 3000–4000 g/mol were used to create modified PVC membranes. Flat sheet PVC membranes are modified by using water as a non-solvent during the phase inversion process. In order to make the casting solution, PVC and the necessary additive were dissolved in N, N-Dimethylformamide (DMF), which made up 80% of the mixture. A separate solvent for pore creation is not used in the process of making membranes. The casting solution was constantly agitated for 24 hours until a clear, homogeneous solution was achieved. The solution was then cast onto a glass plate for a thickness of around 200 μ m using an Elicometer thin film applicator. In order to refill the residual DMF, the glass plate was then immediately immersed for 24 hours in a distilled water bath that was kept at 20°C. A certain portion of the modified PVC membrane had to be removed in order to conduct the cross-flow UF experiments employed in this study. The phase inversion method is shown in figure (2.1). A stable emulsion concentration of 2000 ppm of the oil-water mixture was produced in the lab using distilled water and commercial-grade olive oil. First, 2 g of olive oil and 1 g of the emulsifier anionic surfactant SDS were mixed with 500 ml of distilled water. The solution was diluted to a volume of 1000 ml in a volumetric flask. The mixture was swirled for two hours at a speed of 750 revolutions per minute using a magnetic stirrer tank till the milky white oily water was found to be stable because the turbidity of the emulsion's surface was found to be 165 NTU and its bottom was almost discovered to be 163 NTU. The stable oil-water emulsion was then kept at room temperature to maintain the oil-to-water ratio during each cycle of the filtration process.

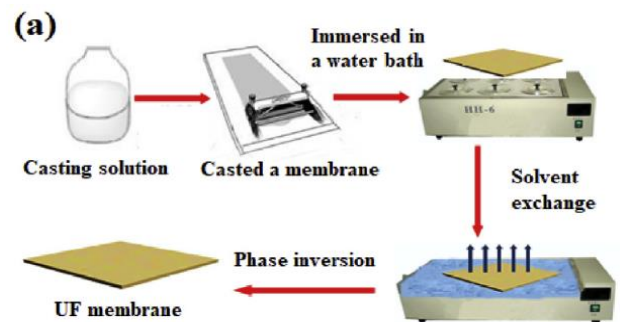


Figure 2.1: Schematic diagram for fabrication and enhancing UF membranes by phase inversion method

2.2. Characterization of modified PVC membrane

In order to assess how hydrophilic the manufactured membranes were, contact angle (CA) was utilized to analyze the contact angle. The incident and retreating angles were determined using water droplets placed in five distinct positions to find the equilibrium water contact angle. The five data were averaged to find each membrane's water contact angle. The top surface morphology and cross-section

morphology of additives added to industrial and research-grade PVC membranes were seen using FESEM. FTIR stands for Fourier transform infrared spectroscopy. In order to determine the functional group, infrared spectra have been recorded using Fourier transform infrared spectroscopy (FTIR). Strength mechanically and porosity. The mechanical strength of the created membranes was evaluated using a tensile testing equipment. Porosity might also be determined by testing the membrane sample's permeability and examining the oil permeate flows and rejections to see how effectively the membrane functioned.

2.3. Experimental setup and methodology

We'll provide examples of the equipment setup, experimentation process, and operational circumstances.

2.3.1. Experimental set-up

Using a lab-scale cross flow system, we examine pure water flux (PWF) and oily wastewater filtration. Since the filtering cell has a surface area of 17.349 cm², the system comprises of an ultrafiltration (UF) membrane, a feed reservoir, a pump, and a pressure gauge to ensure that the pressure is maintained at 1 bar throughout the entire procedure. Figure (2.2) illustrates the schematic of the cross-flow system used in this paper.

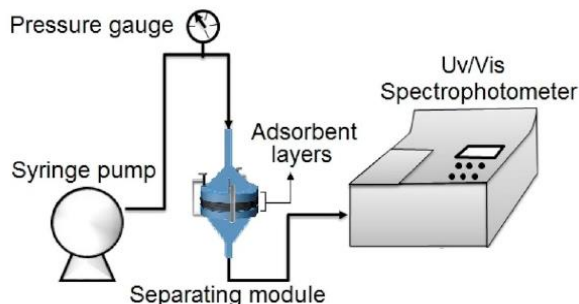


Figure 2.2: Schematic representation of cross-flow UF.

2.3.2 Experimental methodology

The emulsion is first made by adding 1 gm of sodium lauryl sulphate to 2000 ppm of oil concentration and stirring it with a magnetic stirrer at 750 rpm for two hours, or until it becomes homogeneous. The turbidity on top of the emulsion, which we used to verify for homogeneity, was measured at 165 NTU; the turbidity on the bottom, which was almost as high, was found to be 163 NTU. We use a peristaltic pump to pressurize the emulsion at a constant pressure of 1 bar. The feed is then passed over a cross-flow UF membrane with a surface area of approximately 17.349 cm², and we collect the permeate.

2.4. Fouling effect, chemical cleaning and coating of membrane.

We study membrane fouling because it happens when pores in an emulsion are partially or totally closed by the adsorption of its continuous or dispersed phases, or when concentration polarization causes one or more phases to accumulate on the membrane surface. This incident results in a significant decrease in flux and worsens the hydraulic system's overall flow rate. To solve fouling problems, we made chemical cleaning for both types of membranes with simple acid as citric acid and simple base as sodium hydroxide with concentration 0.15M and 0.1M respectively. In this study we show the positive results of cleaning on fluxes and rejection of oil. Another way to solve fouling effects is coating of membrane with three different mechanisms with TA, PVP and Ferric chloride.

3. RESULTS AND DISCUSSIONS

3.1 Characterization of the membranes

The enhanced PVC/PVP membranes were characterized and evaluated using the mechanical strength, porosity, SEM, FTIR, and water contact angle measurement.

3.1.1 Mechanical strength

After determining the force and extension from the device, we evaluate the tensile strength. The tensile strength for lab chemical membrane was approximately 42.12766 MPa, whereas the tensile strength for commercial chemical membrane was around 32.75461 MPa. The mechanical characteristics of chemical membranes used in laboratories and in commerce are shown in table (3.1).

Table (3.1): Mechanical properties for two types of membrane

Type of membrane	Force (N)	Extensio n (mm)	Thickness (mm)	Average thicknes s (mm)	Tensile strength s (MPa)
Lab chemical membrane	99	14.66	0.087 0.093 0.102	0.094	42.13
Commercial chem membrane	119.8	19.06	0.147 0.143 0.149	0.015	32.76

3.1.2 Porosity measurement

We calculated the porosity of a lab membrane and a commercial membrane, and we discovered that the lab membrane's porosity was 0.2755 while the commercial membrane's porosity was 0.0937, Since the permeability of

both types of membranes could be estimated using the following equation, $135.5/\text{time (sec)}$, the table (3.2) illustrates their porosity. Since P is permeability, C is constant equal to 2, and r^2 equals 6.25 cm^2 from the device, time is estimated straight from the equipment and porosity is calculated using equation PC/r^2 .

Table (3.2): Porosity of membranes

Type of membrane	Time (sec)	Vol (ml)	Permeability	Porosity Q_c
Lab	157.4	110	0.86086	0.2755
Commercial	462.7	120	0.2928	0.0937

3.1.3 Contact angle (CA)

We examined the contact angles for both pristine PVC lab and PVC commercial chemical membranes and discovered that the lab chemical membrane had a lower contact angle than the latter, coming in at 66.9° as opposed to 86.4° for the latter. We conclude that hydrophilicity of lab chemical is more than commercial chemical membrane as shown in figure (3.1).

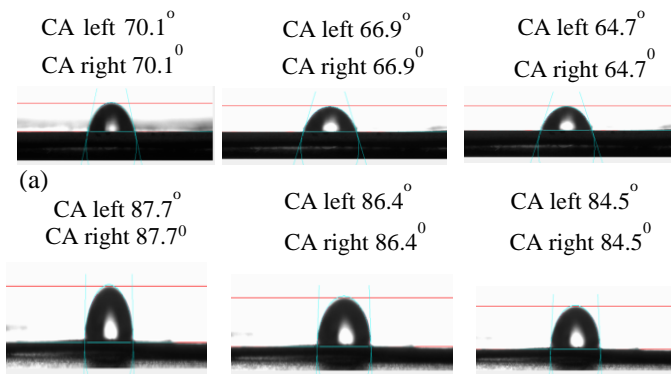


Figure 3.1: Illustrate contact angle for both (a) lab and (b) commercial chemical membrane from different three angles.

When coated membrane with different three mechanisms, we found that contact angle is lower than pristine membranes. Since the first way of coating lab chemical membrane (M_1 lab) with TA and ferric chloride the contact angle was found about 44.3° but for commercial chemical membrane (M_1 commercial) was found 57.7° , second way of coating lab chemical membrane (M_2 lab) was found about 61.3° but commercial chemical membrane (M_2 commercial) was 54.1° . The Last mechanism for coating with TA, PVP and ferric chloride, CA for lab chemical membrane (M_3 lab) was 51.4° but for commercial chemical membrane (M_3 commercial) was found 68.9° .

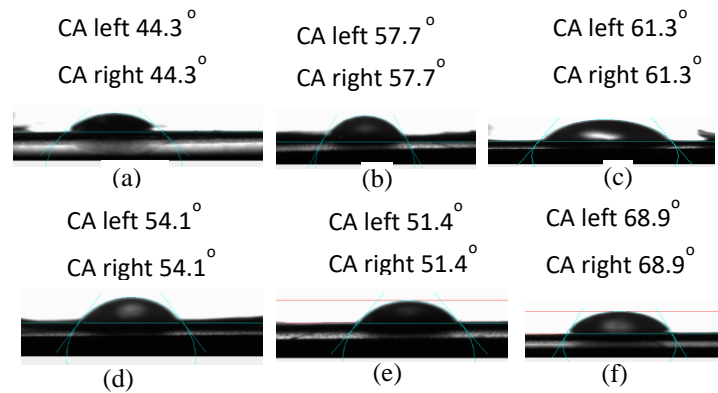


Figure 3.2: Illustrate contact angle for both (a) M_1 Lab, (b) M_1 commercial membrane, (c) M_2 lab, (d) M_2 commercial, (e) M_3 lab, (f) M_3 commercial.

Since M_1 lab refers to first way for lab coated membrane, M_1 commercial is the first way for coating commercial membrane, M_2 lab refers to second way for lab coating membrane, M_2 commercial refers to second way for commercial coated membrane, M_3 lab the third way for coating lab chemical membrane and M_3 commercial third way for commercial coated membrane.

3.1.4 Scanning electron microscopy (SEM)

The mineralized membrane's cross-sectional scanning electron microscopy (SEM) image shows a finger-like porous structure with an even distribution of components throughout the membrane, since commercial chemical membrane has less voids than lab chemical membrane, also for top surface structure the commercial is more closed opening than lab so the results for lab chemical membrane is slightly better than commercial chemical membrane as shown in figure (3.3).

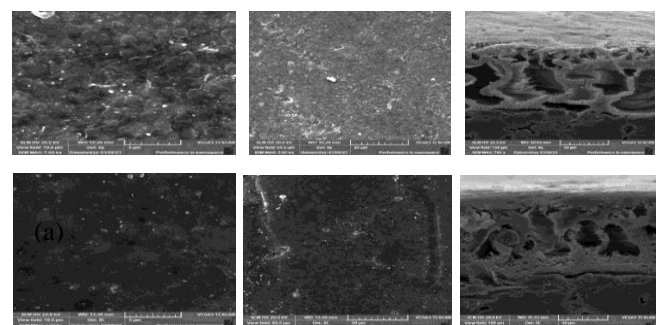
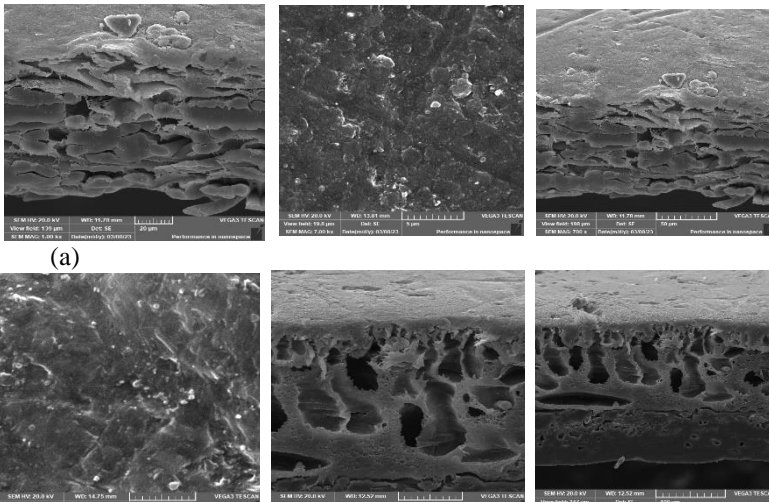
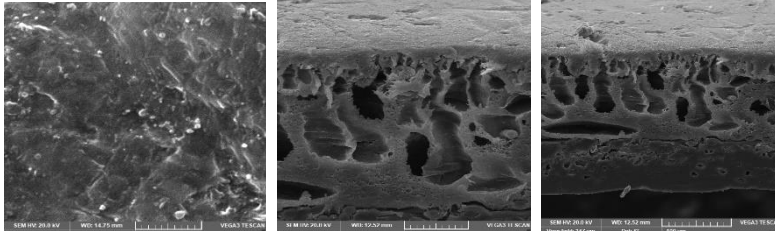


Figure 3.3: SEM analysis for both (a) lab chemical membrane and (b) commercial chemical membrane from different scales illustrating surface and cross section morphology.



(a)



(b)

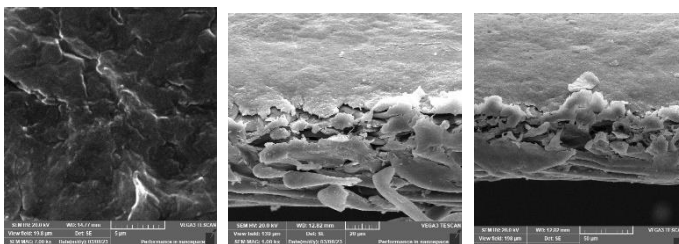
Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Chlorine	17	56	2.13	51.42	28.36	0.34	16.01
Carbon	6	2	1.54	37.13	60.44	4.81	313.01
Oxygen	8	1	0.34	8.25	10.08	1.45	425.58
Iron	26	7	0.13	3.20	1.12	0.08	57.50
Sum	4	14	100.00	100.00	100.00		

(c)

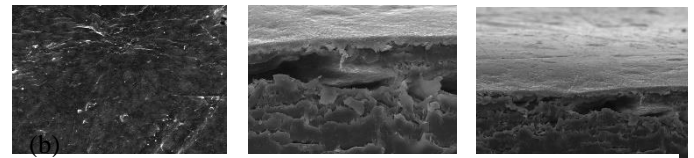
Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Chlorine	17	139	2.82	55.76	32.36	0.31	10.93
Carbon	6	3	1.53	30.25	51.81	3.91	255.28
Oxygen	8	3	0.59	11.63	14.96	1.49	253.27
Iron	26	11	0.12	2.36	0.87	0.06	51.87
Sum	5	06	100.00	100.00	100.00		

(d) Figure 3.4: SEM analysis for both (a) M₁ lab membrane, (b) M₁ commercial membrane, (c) EDX for M₁, (d) EDX for M₁.

We note when we coated membrane with tannic acid and ferric chloride that the structure of surface and cross surface morphology is enhanced so the flux of permeate is increased, also rejection of oil increased. EDX for both lab chemical membrane and commercial chemical membrane show the percentage of chemical component as shown in figure (3.4).



(a)

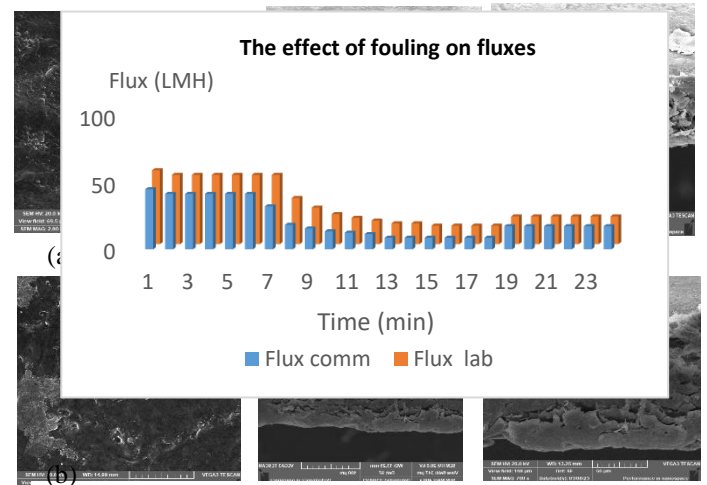


Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Carbon	6	762	27.54	62.77	80.04	6.77	24.58
Chlorine	17	4066	11.48	26.16	11.30	0.47	4.10
Oxygen	8	71	2.12	4.83	4.62	1.24	58.63
Nitrogen	7	13	1.32	3.00	3.29	1.60	121.61
Hafnium	72	61	0.74	1.68	0.14	0.13	17.07
Tantalum	73	20	0.24	0.56	0.05	0.08	33.40
Sodium	11	73	0.21	0.48	0.32	0.06	28.09
Phosphorus	15	81	0.19	0.44	0.22	0.05	26.08
Iron	26	6	0.03	0.08	0.02	0.01	43.13
Sum	43.87		100.00	100.00			

(c)

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Carbon	6	369	3.81	67.99	84.66	1.20	31.49
Chlorine	17	3486	1.51	26.92	11.35	0.08	5.62
Oxygen	8	33	0.23	4.08	3.82	0.21	90.49
Tellurium	52	5	0.04	0.69	0.08	0.02	46.49
Iron	26	3	0.02	0.32	0.09	0.01	60.80
Sum	5.61		100.00	100.00			

(d)

Figure 3.5: SEM analysis for both (a) M₂ lab membrane, (b) M₂ commercial membrane, (c) EDX for M₂, (d) EDX for M₂.Figure 3.6: SEM analysis for both (a) M₃ lab membrane, (b) M₃ commercial membrane, (c) EDX for M₃, (d) EDX for M₃.

(b)

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Carbon	6	369	3.81	67.99	84.66	1.20	31.49
Chlorine	17	3486	1.51	26.92	11.35	0.08	5.62
Oxygen	8	33	0.23	4.08	3.82	0.21	90.49
Tellurium	52	5	0.04	0.69	0.08	0.02	46.49
Iron	26	3	0.02	0.32	0.09	0.01	60.80
Sum	5.61		100.00	100.00			

(c) M₃ commercial membrane, (d) EDX for M₃, (d) EDX for M₃.

3.1.5 Fourier transform infrared spectroscopy (FTIR)

For the clean PVC for lab and commercial use, a moderately intensified wideband between 1730 and 2853 cm^{-1} can be seen as shown in figure (3.7), coated membranes exhibited a wavelength between 1744 and 2853 cm^{-1} , which was attributed to the hydroxyl groups in TA-Fe's O-H stretching vibration. Due to the O-H group's hydrophilic properties, PVC coated had a much lower contact angle (44.3) than neat PVC (64.7^(a)).

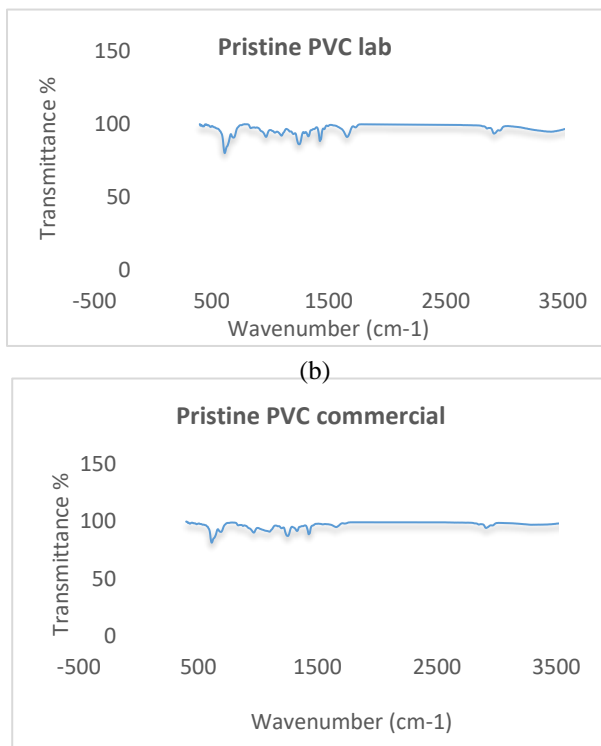


Figure 3.7: FTIR analysis for both (a) lab and (b) commercial membranes.

3.2 Fouling effect

For lab chemical membrane the pure water in initial state before the process was found to be 55.3346 LMH and after fouling occurs the PWF reached to 20.7505 LMH as shown in figure (3.8). but for commercial membrane the flux of pure water in initial state before the process was found about 44.9594 LMH but it reached after fouling to 17.2921 LMH.

Figure 3.8: Comparison between fouling for both types of membrane

3.3 Chemical cleaning

Compare between fluxes of both Lab and chemical membrane after cleaning.

3.3.1 Chemical cleaning with simple acid as citric acid $\text{C}_6\text{H}_8\text{O}_7$ (0.15M)

For commercial chemical membrane flux reached after chemical cleaning with citric acid to 24.20889 LMH, but lab chemical membrane after chemical cleaning with citric acid reached to 31.125 LMH as shown in figure (3.9).

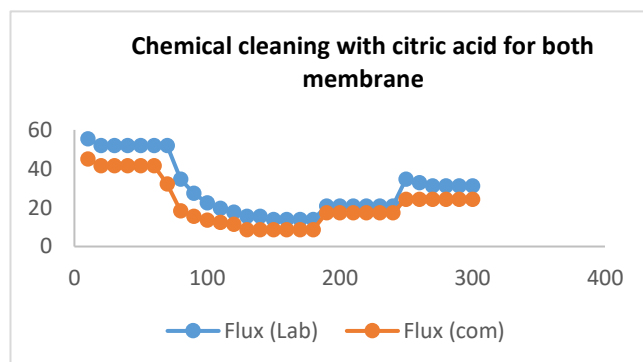


Figure 3.9: Chemical cleaning with citric acid for both lab and commercial chemical membrane

3.3.2 Chemical cleaning with simple base as sodium hydroxide (0.1M)

For commercial chemical membrane flux reached after chemical cleaning with sodium hydroxide to 27.6673 LMH, but lab chemical membrane after chemical cleaning with sodium hydroxide reached also to 27.7 LMH as shown in figure (3.10).

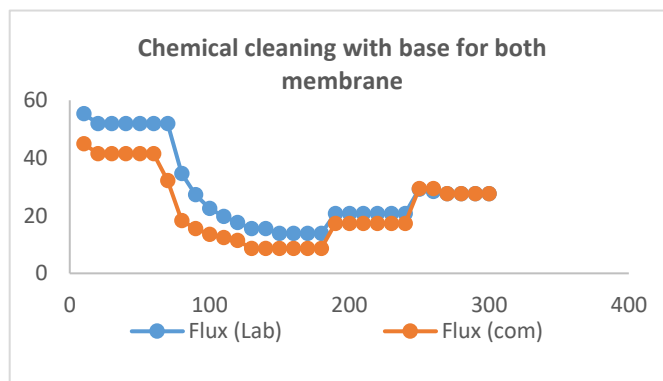


Figure 3.10: Chemical cleaning with sodium hydroxide for both lab and commercial chemical membrane.

3.4. Membrane coating results

We study the effect of coating in membrane performance and we made coating for six membranes, M_1 lab and M_1 commercial [PVC/TA-Fe^{III}] coated membranes respectively, M_2 lab and M_2 commercial [PVC/TA-PVP-Fe^{III}] coated membranes respectively and also M_3 lab and M_3

commercial [PVC/TA-PVP-Fe^{III}] coated membranes respectively but in another way for coating.

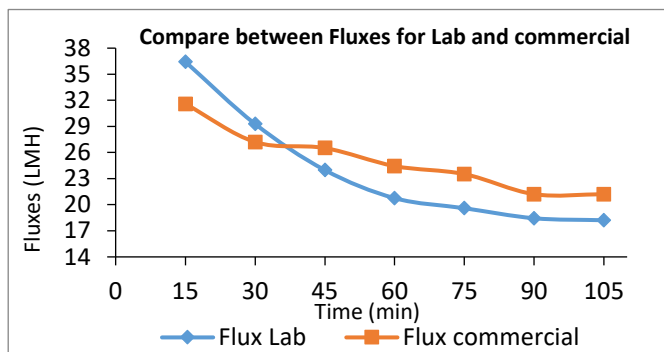
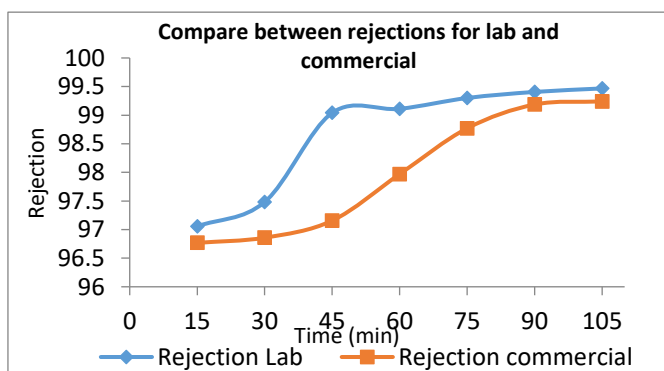
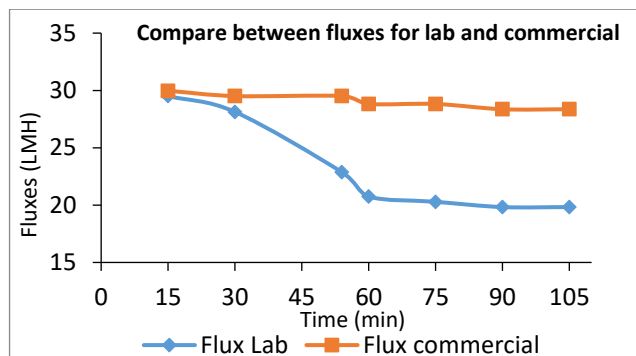


Figure 3.11: The results between (a) M₁ lab and (b) M₁

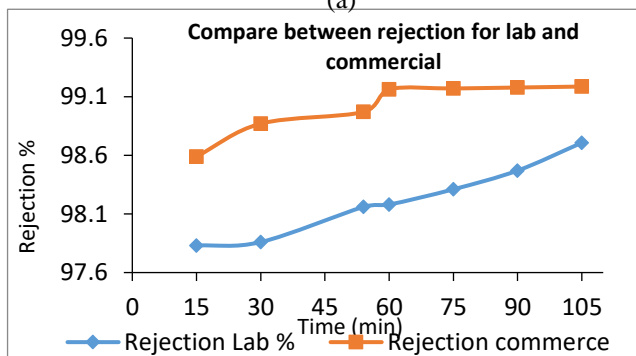


commercial membranes for TA-Fe^{III} [PVC/TA-Fe^{III}] coated membranes.

Figure (3.12): The results between (a) M₂ lab and (b) M₂ commercial membranes for [PVC/TA-PVP-Fe^{III}] coated membranes.

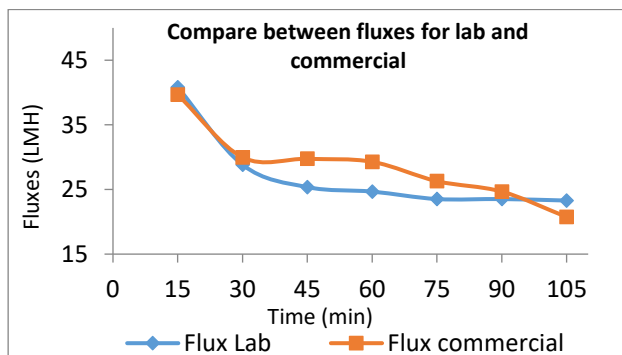


(a)

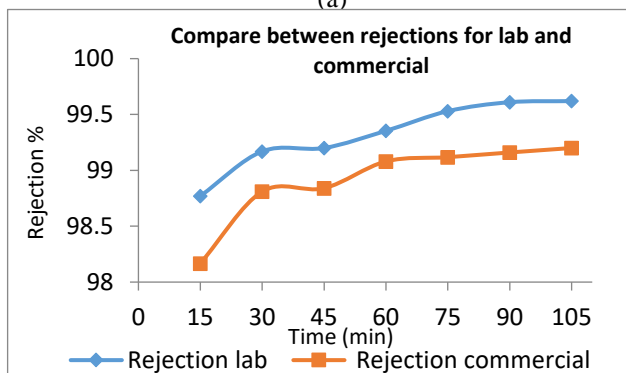


(b)

Figure (3.13): The results between (a) M₃ lab and (b) M₃ commercial membranes for [PVC/TA-PVP-Fe^{III}] coated membranes.



(a)



(b)

3.5. Fouling effect for coated membranes (M₁ lab & M₁ commercial membranes) [PVC/TA-Fe^{III}], (M₂ lab & M₂ commercial membranes) [PVC/TA-PVP-Fe^{III}] and (M₃ lab & M₃ commercial membranes) [PVC/TA-PVP-Fe^{III}].

We study the performance of membrane after coated with TA & Fe^{III} materials and we found that (M₁ lab coated membrane), the decline of PWF was from 77.81 LMH before fouling to 31.13 LMH after doing fouling by percentage of 59.99%. But before coating the PWF was about 51.876 LMH before fouling to 20.7505 LMH after doing fouling so after coating, the flux of membrane was enhanced by 33.34%. For (M₁ commercial coated membrane) the decline of PWF was from 70.89 LMH before fouling to 41.5 LMH after doing fouling by percentage of 41.46 %. But before coating the PWF was about 44.9594 LMH before fouling to 17.2921 LMH after doing fouling, so after coating the flux of membrane was enhanced by 58.33% as shown in figure (5.28). When coating with TA & PVP & Fe^{III} for PVC based-lab chemical membrane (M₂ lab), the decline of PWF reached from 69.17 LMH before fouling to 41.5 LMH after fouling by percentage of 40%. But before coating the PWF was about 51.876 LMH before fouling to 20.7505 LMH after doing fouling so after coating, the flux of lab chemical membrane was enhanced by 50%. On the other hand when coating with TA & PVP & Fe^{III} for PVC based-commercial chemical membrane (M₂ commercial), the decline of PWF reached from 69.17 LMH before fouling to 22.48 LMH after fouling by percentage of 67.5%. But before coating the PWF was about 44.9594 LMH before fouling to 17.2921 LMH after doing fouling so after coating, the flux of commercial chemical membrane was enhanced by 23% ...When coating with TA & PVP & Fe^{III} for PVC based-lab chemical membrane (M₃ lab), the decline of PWF was from 69.2 LMH before fouling to 32.51 LMH after doing fouling by percentage of 53%. But before coating the PWF was about 51.876 LMH before fouling to 20.7505 LMH after doing fouling so after coating, the flux of membrane was enhanced by 36.17%. For (M₃ commercial) the decline of PWF was

from 93.4 LMH before fouling to 41.84 LMH after doing fouling by percentage of 55.2 %. But before coating the PWF was about 44.9594 LMH before fouling to 17.2921 LMH after doing fouling, so after coating the flux of membrane was enhanced by 58.67% as shown in .

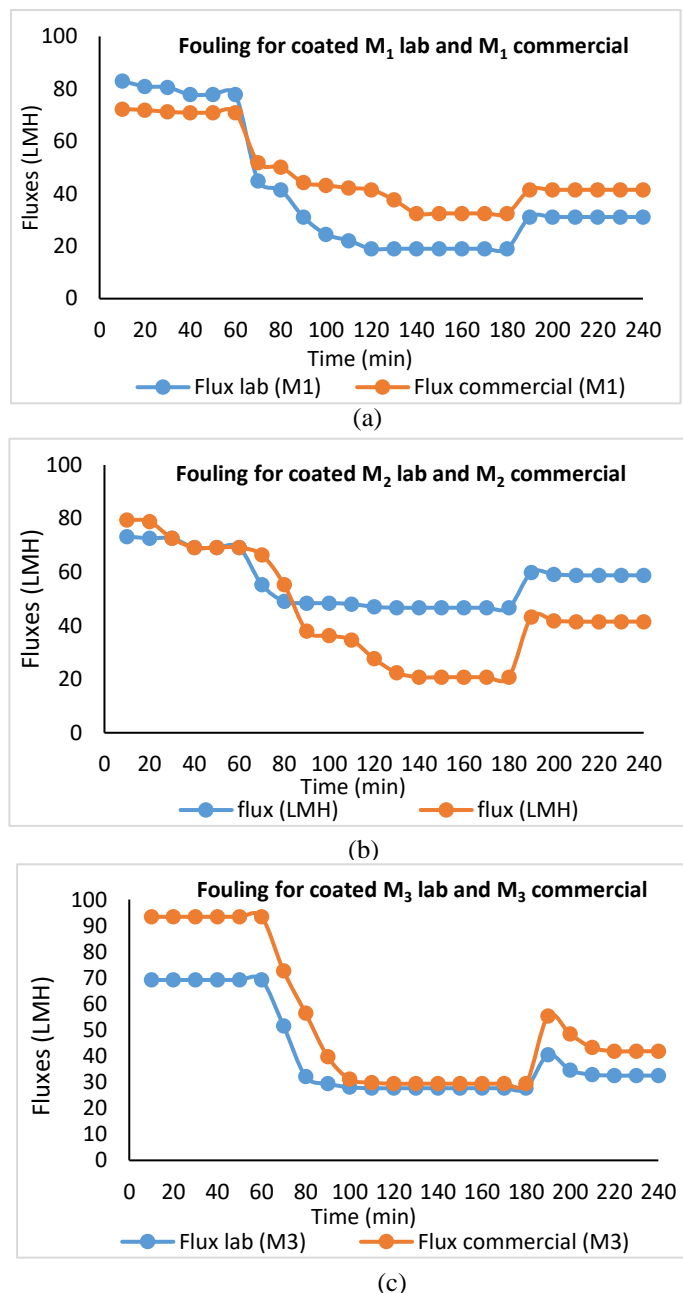


Figure 3.14: Show the effect of fouling for lab and commercial membranes for three different coated membranes

4. CONCLUSION

Phase inversion was used in this study to create commercial and lab chemical PVC/PVP membranes for the

treatment of oily waster. Due to its high hydrophilicity, PVP has been used to solve a number of PVC-related issues, including the fouling of PVC membranes and matrix aggregation. In this research, the following points have been investigated:

(1) Compared the performance between lab chemical membrane (M_L) and commercial chemical membrane (M_C), pure water flux decreased by 60.9% and 46.67% for M_L and M_C , respectively

(2) The standard emulsion concentration used was about 2000 ppm. The concentration of oil is reduced by 93.75% and 92.806% for M_L and M_C used, since it is known as rejection percentage.

(3) By studying the effect of fouling and chemical cleaning since For M_L the pure water in initial state before the process was found to be 55.3346 LMH and after fouling occurs the PWF reached to 20.7505 LMH, but for M_C the flux of pure water in initial state before the process was found about 44.9594 LMH but it reached after fouling to 17.2921 LMH.

(4) Chemical cleaning with simple acid as citric acid $C_6H_8O_7$ (0.15M), for M_C flux reached after chemical cleaning with citric acid to 24.20889 LMH, but M_L after chemical cleaning with citric acid reached to 31.125 LMH, also with cleaning with simple base as sodium hydroxide (0.1M), since for M_C flux reached after chemical cleaning with sodium hydroxide to 27.6673 LMH, but M_L after chemical cleaning with sodium hydroxide reached also to 27.6673 LMH.

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Today, numerous businesses produce significant amounts of wastewater that is greasy. Separating the stable emulsified oil particles from water is the fundamental difficulty in treating oily wastewater. Generally speaking, polymeric membranes are essential in these procedures due to how simple and inexpensive modern separation processing is, as well as how flexible they are. The phase inversion method utilized in this study to create a poly vinyl alcohol (PVC) membrane is demonstrated. In order to increase both permeation flow and fouling resistance, PVC has been modified by the addition of polymeric additives like polyvinylpyrrolidone (PVP). Additionally, in this paper, we compare lab chemical membrane and commercial chemical membrane on the basis of their fluxes, rejection, and characterization. The enhanced PVC/PVP membranes were characterized and evaluated using mechanical strength, porosity, scanning electron microscopy (SEM), Fourier Transform Infrared (FTIR), and water contact angle measurement. The membranes were subsequently tested at a lab size in a cross-flow system with synthetic greasy wastewater as the input.

Keywords: Oil/water separation, UF membranes, Membrane modification.