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PERFORMANCE OF THIN FILM COMPOSITE REVERSE OSMOSIS MEMBRANE IN TREATMENT OF WASTEWATER CONTAMINATED WITH HEAVY METALS AND ORGANICS

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Abstract- Reverse osmosis (RO) membranes are the predominant technique of water desalination due to their low energy demands and unit water expense. Fouling is one of the difficulties associated with the utilization of RO membranes because it increases operational expenses owing to regular cleaning, shorter membrane lifetime, and reduction in permeate flux. In this work, polluted water samples were utilized as feedwaters to investigate the effectiveness of the TFC-PA RO membrane in the desalination of waterways carrying various pollutants. Pollutant effects on membrane fouling development were also investigated. Results from the experiments showed that the membrane effectiveness in relation to permeate flow, pollutant rejection, and water recovery was reduced after the addition of the individual pollutants to the RO feedwater. Additionally, as demonstrated by SEM images, the pollutants were found to be irregularly deposited on the membrane surface, which formed a scaling layer that completely covered the surface when only salts and heavy metals were present in the feedwater. However, when all contaminants were in the feedwater, SEM images exhibited that the surface of the membrane was totally covered with a sludge-like layer of organic and inorganic foulants. In general, heavy metals (Cu^{+2} and Cr^{+3}) and organic pollutants (oil) were found to have a negative effect on membrane performance due to the high fouling formation, which induced water recovery to drop by 33.6 and 36.8%, respectively. Therefore, a pretreatment for heavy metals and organics present in the feed of RO plants is suggested to prevent fouling, enhance membrane efficiency, and lengthen membrane life.

Keywords: Reverse Osmosis, Desalination, Permeate Flux, Salt Rejection, Fouling.

1. INTRODUCTION

One of the most significant issues facing the planet at the moment is the ongoing rise in water usage, despite the fact that the world's supplies of drinkable water are finite, as well as the pollution of water sources by wastewater from industries and municipalities [1, 2]. As a result, desalination may be used to produce potable water and therefore help reduce water shortages. Desalination is primarily accomplished through the use of RO membrane water filtration. Among the current desalination technologies, RO has the lowest energy usage and water production costs [3]. The most commonly utilized membrane in the RO process is the thin-film composite polyamide TFC-PA, which is generated via interfacial polymerization [4]. This membrane has an active layer made of polyamide that has a thickness in the range of 0.01-0.2 µm and is supported by layers of polysulfone (50 µm) and polyester fabric (100-150 µm) [5].

Despite that the reverse osmosis (RO) process being recognized as a power-saving and cost-efficient technique, it faces significant challenges. Fouling is one of the issues associated with employing RO membranes in water desalination [6]. Fouling of a membrane refers to the accumulation of particles that have been retained on the surface of the membrane [7]. Fouling is categorized into four types: organic fouling, inorganic fouling, particulate fouling, and biofouling [8]. Fouling minimizes the membrane active area and rises membrane resistance. As a consequence, the filtration rate decreases, and hence membrane productivity diminishes. It further raises operational expenses owing to greater periodic cleaning, reduced membrane longevity, and permeate flow loss [9]. Membrane autopsy is a successful technique for identifying foulants to determine the nature of fouling and adequately prevent it [6]. The membrane autopsy approach includes of membrane dissection, visual evaluation of the membrane surface, and examination of other components to detect any damage.

Scanning electron microscopy (SEM) image is the most widely utilized method in autopsy investigations because they provide a comprehensive micrographic overview of the membrane surface and assist in identifying the type of deposits [10]. Another autopsy technique is the Atomic Force Microscope (AFM) spectrum, which is performed to determine the roughness of the membrane surface [11]. Autopsy studies of fouled membranes have been conducted to determine the kind and extent of fouling.

For example, Koyuncu and Wiesner [12] employed membrane autopsy to study the formation and content of foulants. They discovered that $CaCO_3$ and $CaSO_4$ were the most prominent pollutants on the membrane surface. Also, variations in organic substance concentration were shown to affect the crystal structure of calcium carbonate, enhancing its precipitation over the membrane surface. In another research, Karime, et al. [13] performed an autopsy on a spiral wound membrane. According to autopsy findings, the principal foulants were polysaccharide, clay, SiO_2, $CaSO_4$, $CaSiO_3$, $AlPO_4$, and Fe_3O_4. In another study, Kim, et al. [14] examined the degree and distribution of fouling in spiral wound RO membranes using membrane autopsy. The study revealed that fulvic acid was the primary cause of fouling. A slight degree of inorganic fouling caused by Fe and Al was also noticed. In different study, Trinanes, et al. [15] studied the effectiveness of membrane autopsy in estimating the intensity of fouling. They discovered that biofouling was mainly prompted by diatoms, pseudomonas, and polysaccharides. On the other hand, inorganic scaling was observed to be predominantly induced by sulphate, aluminum, calcium, and silica.

In this study, the performance of the TFC-PA RO membrane in treating simulated feedwater containing salts, heavy metals and organic matters was studied. An autopsy was performed on the membranes to discover the primary causes of fouling and performance decline. Also, the effect of the pollutants on fouling formation was investigated using SEM and AFM tests.

2. MATERIALS AND METHODS

2.1. Materials

The chemicals used in this work were sodium chloride (NaCl), calcium chloride (CaCl2), magnesium chloride ($MgCl_2$), copper chloride ($CuCl_2.2H_2O$), and chromium chloride ($CrCl_3.6H_2O$) 98%, with a purity of 99.5%, 99%, 98%, 99%, and 98%, respectively. All were obtained from Thomas Baker Chemicals, Mumbai, India. The organic substance in this study was represented by kerosene oil. An ultrapure water purification system in the Environment and Water Directorate, Ministry of Science and Technology, Iraq was utilized to produce the deionized (DI) water that was used in the experiments. The membrane employed in this research was a TFC-PA SEPA CF cell RO membrane ordered from Sterlitech Corporation in Washington, United States.

2.2. RO Performance Test

In this work, all of the experiments were performed with a SEPA CF Cell (purchased from Sterlitech, United States). The RO Cell was manufactured to resist a maximum applied pressure of 6895 kPa (68.95 bar) and carry a flat sheet membrane with a dimension of 19×14 cm. Consequently, its effective area was 140 cm^2. Detailed info regarding the experiment device setup could be found in the literature [16]. The SEPA CF Membrane system adopted in the experiment is shown in Figure 1.

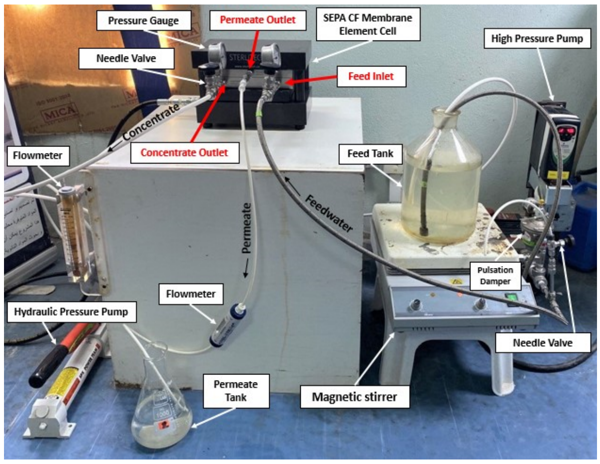

Figure 1. SEPA CF Membrane system used in the experiment.

Feedwaters were prepared by dissolving the requisite amounts of salts, heavy metals, and oil in 5 liters of deionized (DI) water while maintaining a constant total osmotic pressure (Π) of 1.685 bar. For the oil, a Laboratory Emulsifier (SRH-S450, SIEHE Industry) spins the mixture at 10,000 rpm to emulsify it. At a medium speed, a magnetic stirrer (ISOLAB, Laborgerate GmbH) was utilized to guarantee the homogeneity of the solution. Using the high-pressure pump, 5 liters of feedwater was pushed at a consistent flow of 2.5 L/min from the feedwater tank via the RO membrane cell. The applied pressure during the experiments was sustained at 2068 kPa (20 bar) by using the needle valve. The permeate flux was calculated every 30 minutes over the three-hour duration of each experiment. The TDS concentration of the permeate and concentrate was also calculated by a lab multi-meter (HQ430d, Flexi, Hach Company). After that, two 100 ml samples from the permeate tank were obtained to analyze the concentration of specific ions of salts, heavy metals, and oil in the permeate by utilizing the atomic absorption spectrophotometer (Shimadzu, AA-6800). At the conclusion of each test, the polluted membrane was carefully removed from the RO cell. Then, a piece of the membrane was cut and sent for SEM and AFM examination. Afterwards, the permeate flux across the membrane was measured utilizing Equation (1) [17].

$$J_w = \frac{V}{A \times t} \quad (1)$$

where, J_w is the permeate water flux (L/m^{2}h), V is the permeate water volume (L), A is the effective area of the membrane (m^2), and t is the accumulation time (h). The rejection percentage (R%) was calculated by Equation (2) [18].

$$R = \frac{C_f - C_p}{C_f} \times 100 \quad (2)$$

where, C_p is the salts concentration in the permeate (mg/L) and C_f is the salts concentration in the feed (mg/L). The proportion of water recovered (r%) was computed by employing Equation (3) [19].

$$r = \frac{V_p}{V_f} \times 100 \quad (3)$$

where, V_p is the permeate volume (L) and V_f is the feedwater volume (L). Additionally, the feedwater osmotic pressure (π) may be calculated using the Van't Hoff relation, as shown in Equation (4) [20].

$$\pi = iCRT \quad (4)$$

where, i is the number of ionic dissociations, C is the concentration of ion (mole/L), R is the constant of ideal gas (L.bar/K.mole), and T is the absolute temperature (K).

The steps for conducting the experiment described earlier were carried out in a total of three separate runs. Tables 1 and 2 list the feedwater contents, concentrations and osmotic pressures of individual constituents, total TDS, and total osmotic pressure of the runs that were performed.

Table 1. Analysis of the feedwater components and total TDS for each run

Concentrations (mg/L)			
item.	Run 1	Run 2	Run 3
NaCl	1640	1600	1600
$CaCl_2$	200	200	200
$MgCl_2.6H_2O$	200	200	200
$CuCl_2.2H_2O$	0	50	50
$CrCl_2.6H_2O$	0	50	50
Oil	-	-	500
total TDS	2040	2100	2100

Table 2. Summary of the feed contents osmotic pressure and the total osmotic pressure of runs

Osmotic Pressure (bar)			
Item.	Run 1	Run 2	Run 3
NaCl	1.395	1.354	1.354
$CaCl_2$	0.134	0.134	0.134
$MgCl_2.6H_2O$	0.156	0.156	0.156
$CuCl_2.2H_2O$	0	0.022	0.022
$CrCl_2.6H_2O$	0	0.019	0.019
Oil	-	-	-
total Osmotic pressure (bar)	1.685	1.685	1.685

2.3. Characterization Methods

The foulant layers were visually characterized by scanning electron microscopy image (Fesem Mira3 Tescan, France). Atomic Force Microscopy (AFM) analysis used to measure the membrane's roughness (Angstrom Advanced Inc., USA). For testing purposes, wet RO membrane samples were completely washed with DI water. In addition, the various fouling membranes were meticulously sliced to maintain the material's original composition in its deposited state. The samples were then placed in an enclosed case and sent for testing.

3. RESULTS AND DISCUSSION

3.1. RO Performance Results

Several runs were performed to investigate the performance of the TFC-PA RO membrane in the treatment of the contaminated wastewater samples. Figure 2 shows the evolution of the permeate flux with time for runs 1, 2, and 3. Overall, the permeate flux of all runs dropped with the run time. In run 1, where the feedwater contained only salts (NaCl, $CaCl_2$, and $MgCl_2$), showed the highest permeate flux, which ranged from 119.6 to 104.96 L/m^2h.

Whereas in run 2, where both salts and heavy metals ($CuCl_2.2H_2O$ and $CrCl_3.6H_2O$) were presented in the feedwater, the range of permeate flux was from 80.33 to 29.56 L/m^2h. On the other hand, run 3, which was performed with the addition of all pollutants to the feedwater, including salts, heavy metals, and oil, showed the lowest permeate flux, which ranged from 48.51 to 17.67 L/m^2h. There was less scale deposition on the membrane surface in run 1, as shown in Figure 7, which explains the higher permeate flow observed in that run.

However, in run 2, the permeate flux went down because heavy metal ions (Cu^{+2} and Cr^{+3}) were added to the feedwater. This made it more likely that scale would form on the surface of the membrane because of inorganic fouling and decreased the filtration area, which led to a noticeable drop in the permeate flux. In addition, the drop in flux in run 3 was probably caused by the presence of oil in the feedwater, which led to the development of organic fouling on the membrane surface, resulting in a decrease in the effective membrane area and a corresponding decline in permeate flux. The drop in permeate flux was a result of fouling buildup on the surface of the membranes, which reduced the available filtration area [21]. Furthermore, when both organic and inorganic pollutants were existed in the feedwater (run 3), the cohabitation of organic foulants significantly boosted the permeate flux reduction [22].

Rejection percentages for each run are shown in Figure 3. Run 1 had the greatest rejection rate compared to runs 2 and 3. The low rejection percentage of run 2 is attributed to the addition of heavy metal ions to the feedwater, which increased the concentration of pollutant in the permeate channel due to the increased passage of pollutants ions through the membrane, leading to a lesser rejection. The rejection was reduced as the concentration of the feedwater pollutant grew owing to the higher concentration difference across the membrane and the diffusion of pollutant ions through the membrane [23]. This agrees with the outcomes of run 2. On the other hand, in run 3, the large molecular weight of oil and its limited solubility in water rose the rejection percentage of pollutants. This confirms that molecular weight plays a crucial role in the rejection of contaminants [24].

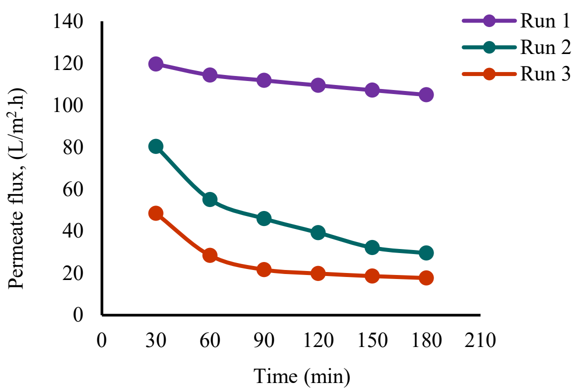

Figure 2. Permeate flux of the TFC- RO membrane of the performed runs

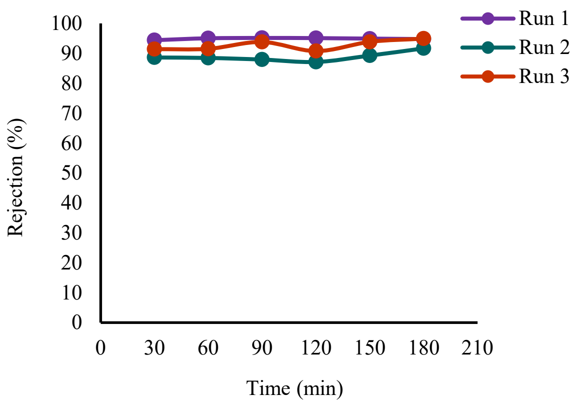

Figure 3. Rejection percentages of the membranes

Figure 4 demonstrates the removal rates of the major ions. According to the results, the membrane exhibited low rejection rates in run 1, in which the membrane rejected 95.4%, 70%, and 94% of Na^{+}, Ca^{+2}, and Mg^{+2}, respectively (Figure 4a). The increased rejection of sodium salts over divalent salts might be attributed to the higher diffusivity of Ca^{+2} and Mg^{+2} ions across the membrane [16]. However, in run 2, the tested membrane removed Na^{+}, Ca^{+2}, Mg^{+2}, Cu^{+2}, and Cr^{+3} at rates of 88.7%, 77.5%, 90.5%, 93.4%, and 99.6%, respectively (Figure 4b). On the other hand, run 3 displayed the largest rejection percentages of all ions, which were 91.9%, 84%, 96%, 97.6%, 99.8%, and 99.9% of Na^{+}, Ca^{+2}, Mg^{+2}, Cu^{+2}, Cr^{+3}, and oil, respectively (Figure 4c).

Furthermore, the findings showed that adding the heavy metal ions Cu^{+2} and Cr^{+3} to the feedwater increased the rejection rate of Ca^{+2} ions while decreasing the rejection of Na^{+} and Mg^{+2} ions (Figure 4.b). In contrast, in run 3, the presence of oil in the feedwater enhanced the proportion of rejected salts and heavy metal ions. According to Indika, et al. [25] more divalent ions stick to the surface of the membrane and cover a significant portion of the surface as the ion concentration in the feedwater rises.

The percentages of water that were recovered during runs 1, 2, and 3 are illustrated in Figure 5. In general, better water recovery is associated with higher fluxes in all runs. As an example, run 1, which had the largest permeate flux, exhibited the highest recovery percentage (58.4%) compared to runs 2 and 3, which had lower recovery percentages (24.8 and 21.6%). The low rates of water recovery suggested that by adding various impurities to the feedwater, additional fouling substances were deposited on the membrane surface. Thus, the overall filtration area was reduced, resulting in a decline in permeate flow and, consequently, a reduction in water recovery [21].

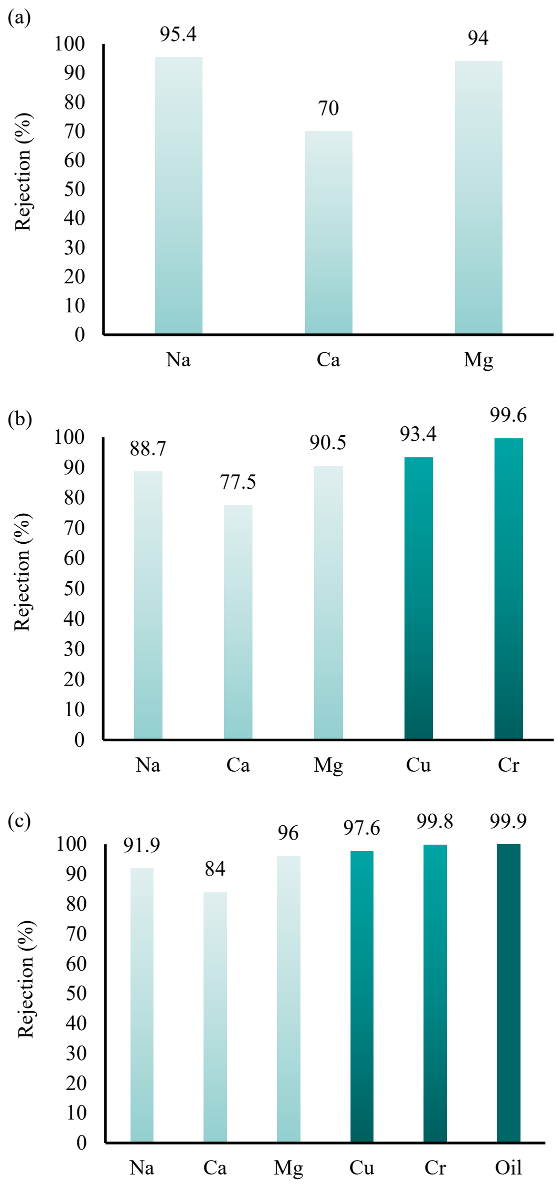

Figure 4. Removal percentages of the individual ions for; (a) Run 1, (b) Run 2, (c) Run 3

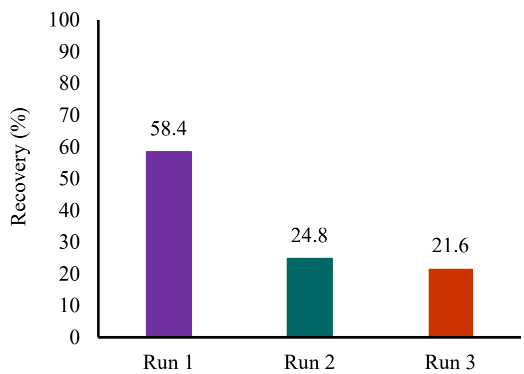

Figure 5. Water recovery percentages of the performed run

3.2. Fouling Characterization

3.2.1. Scanning Electron Microscope (SEM) Images

To study the effect of salts, heavy metals, and oil on fouling formation on the surface of the TFC-PA RO membrane, several top and cross-sectional SEM images with a magnification of 50 kx were conducted for the clean and fouled membranes of runs 1, 2, and 3. Figure 6 represents the top and cross-sectional SEM images of a clean membrane. According to the images, the TFC-PA RO membrane has a highly porous and rough surface shape with ridges and valley zones, which is essential to the RO operation's productivity [26]. Higher surface roughness enhances the membrane performance by increasing the filtration area of the membrane, which allows more permeate water to pass through [20].

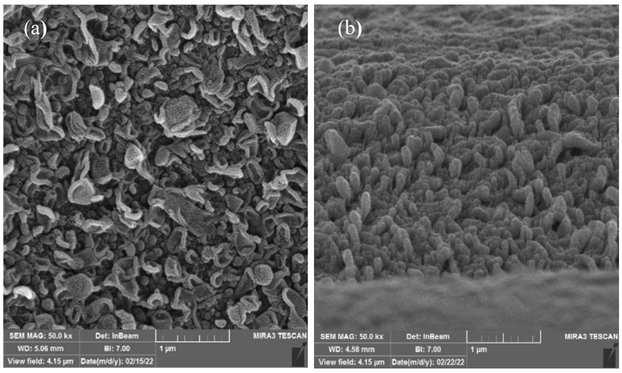

Figure 6. SEM images for a clean membrane for (a) Top and (b) Cross-section

The top and cross-sectional SEM images of the fouled membrane for run 1 are shown in Figure 7. The SEM images showed that the fouling covered a small portion of the membrane surface area. Fouling deposition has an effect on permeate flux [27]. This was obviously seen in the permeate flux of run 1, which was much greater than runs 2 and 3. In addition, run 1 had a greater percentage of rejection than the other runs. This indicates that scale development also influences membrane rejection.

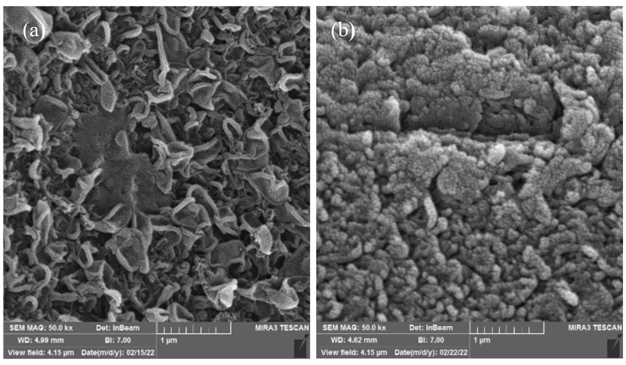

Figure 7. SEM images of the polluted membrane in run 1 (Salts) for; (a) Top, (b) Cross-section

Figure 8 depicts a characteristic fouled membrane surface, which consists of a scale layer with an apparent crystalline shape caused by inorganic material (salts and heavy metals) in the feedwater of run 1. Cross-sectional SEM images demonstrated that the deposition of heavy metal pollutants on the surface of the membrane, which fully covered the membrane surface with a fouling film, led to a reduction in permeate flux and physical degradation of the membrane (Figure 8.b). This assumption was supported by the fact that the initial permeate flux of run 2 was lower than that of run 1, owing to degradation of the membrane surfaces in run 2 caused by significant scale development as a result of inorganic fouling, which had an adverse influence on membrane efficiency. In general, the foulants were irregularly deposited over the membrane surface. Irregular scaling can be seen in the cross-sectional SEM image, which shows a significant difference in deposit thicknesses (Figure 8.b).

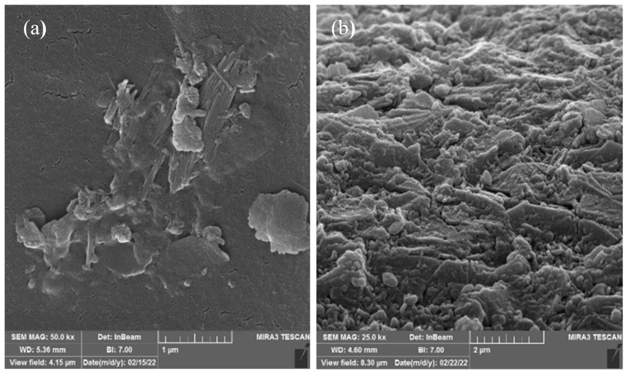

Figure 8. SEM images of the used TFC-PA RO membrane in run 2 (Salts and heavy metals) for; (a) Top, (b) Cross-section

Additionally, the SEM images of the fouled membrane for run 3 indicated that the membrane surface is completely covered with a thick sludge-like layer of foulants comprising a combination of organic and inorganic contaminants (Figure 9). The existence of cations in the feedwater, particularly salts and heavy metal ions (Na^{+}, Ca^{+2}, Mg^{+2}, Cu^{+2}, and Cr^{+3}), could have enhanced fouling. Because the presence of different cations in the feedwater strengthened non-electrostatic interactions among different foulants as well as between foulants and the membrane itself [28]. This could explain the considerable drop in permeate flux seen in run 3, which demonstrated that permeate flux decreased as fouling deposition on the membrane surface developed [29].

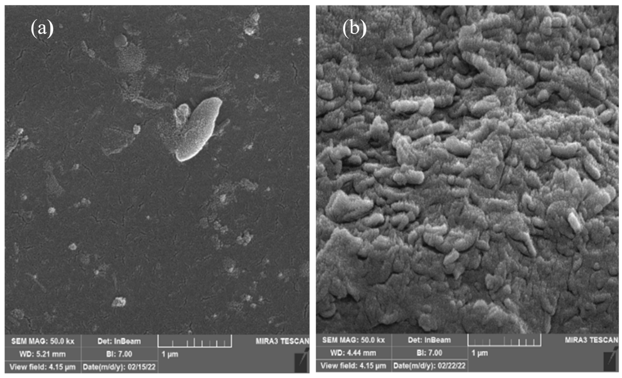

Figure 9. SEM images of the fouled RO membrane in run 3 (Salts, heavy metals, and oil) for; (a) Top, (b) Cross-section

3.2.2. Atomic Force Microscopy (AFM) Images

The AFM test results revealed that the TFC-PA RO membrane had a highly rough surface shape. Fouling is likely to rise as a result of the membrane's high surface roughness [20]. The AFM images of the clean and polluted membranes for the accomplished runs are shown in Figure 10. In addition, the data from the AFM test of the runs that were done are given in Table 3.

Table 3. Analysis of AFM data for fouled RO membranes

	Roughness Average (*RA*) (nm)	Root Mean Square (*RMS*) (nm)	Average Diameter (*AD*) (nm)
Unused membrane	1.55	2.2	17.27
Run 1	2.69	3.85	18.5
Run 2	3.43	4.71	25.1
Run 3	1.06	1.49	5.67

In addition, the analysis of the AFM images revealed that the roughness of the surface of the membrane varied across the several runs that were carried out. The variation in fouling behavior was a result of the difference in membrane roughness [6]. In runs 1 and 2, for instance, the roughness began to rise as a consequence of the development of inorganic scaling from feedwater contained salts and heavy metals, which formed a layer of crystalline-shaped foulants on the membrane, resulting in an increase in the surface roughness. Nonetheless, in run 3, where oil was added to the feedwater, the roughness of the fouled membrane was dramatically decreased owing to the entirely covered porosity of the membrane caused by various inorganic foulants, which were covered with a layer of oil. Apparently, this happened because the membrane pores were filled with oil droplets, which then accumulated on the membrane surface [30].

4. CONCLUSIONS

Investigations revealed that when pollutants are added to a feed, the tested membrane's flux productivity, rejection rate and recovery ratio are all negatively affected. Overall, the rejection percentages of the performed runs clearly showed that the tested TFC-PA membrane rejected a high proportion of Na^{+}, Mg^{+2}, Cu^{+2}, Cr^{+3}, oil, and a considerable amount of Ca^{+2} ions. In general, the results showed that when different contaminants were added to the feedwater, the shape and roughness of the membrane surface changed from one run to another. For example, SEM images of run 2 demonstrated that a scaling layer had totally covered the membrane's surface when the feedwater contained only salts and heavy metals. This caused permeate flux to drop and membrane to deteriorate.

Nevertheless, when the feed included all impurities, the SEM images showed that the surface was entirely covered with a dense sludge-like fouling layer made up of a combination of contaminants. Moreover, the AFM images demonstrated that the variance in fouling behavior was caused by the variability in membrane roughness across the several runs. It was also discovered that adding oil to the feedwater reduced the surface roughness of the membrane, which in turn lowered the permeate flux.

In addition, heavy metals, represented by Cu^{+2} and Cr^{+3}, and organic pollutants, represented by oil, created an issue for membrane efficiency due to the significant fouling layer development, resulting in a sharp decrease in water recovery of about 33.6% and 36.8%, respectively. As a result, a pretreatment for heavy metals and organics in RO plant feed is recommended to minimize fouling, improve membrane performance, and prolong membrane life expectancy. For a better understanding of fouling, more research on fouling behavior phenomena is needed to provide a firmer foundation for improving fouling mitigation strategies. Fouling may also be solved by researching new membrane materials. Future studies on this topic are expected to provide important results.

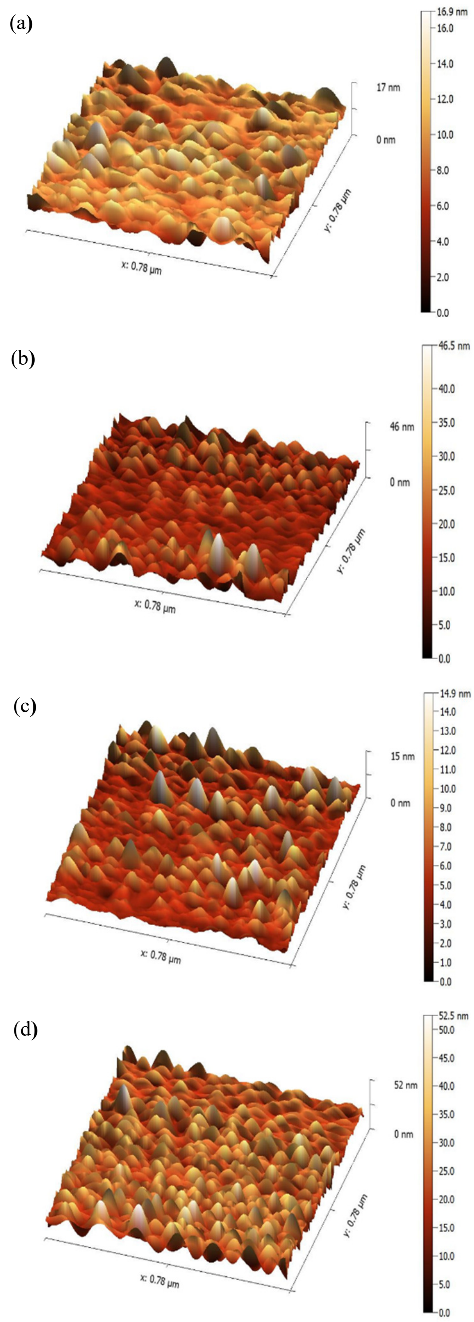

Figure 10. AFM images of the membrane for (a) clean membrane; (b) run 1; (c) run 2; and (d) run 3

NOMENCLATURES

1. Acronyms

RO Reverse Osmosis
TFC-PA Thin Film Composite Polyamide
SEM Scanning Electron Microscope
AFM Atomic Force Microscopy
TDS Total Dissolved Salts
RA Roughness Average
RMS Root Mean Square
AD Average Diameter

2. Symbols / Parameters

J_w : The permeate water flux
V : The permeate water volume
A : The effective area of membrane
t : The accumulation times
C_P : The salts concentration in the permeate
C_f : The salts concentration in the feedwater
V_p : The permeate volume
V_f : The feedwater volume
i : The ionic dissociation numbers
C : The concentration of ion
R : The ideal gas constant
T : The absolute temperature

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