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Investigation of reusability of effluents from an organized industrial zone wastewater treatment plant using a pressure-driven membrane process

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ABSTRACT

The quantity of wastewater being discharged into the environment due to the rise in industrial activities is progressively growing over time. Aside from large environmental risk posed by untreated wastewater discharge, the reuse of treated water prevents wastage of large amount of water. For this reason, in this study, the reuse potential of an organized industrial zone wastewater was investigated by membrane processes. The appropriate membrane type and rejection performance were determined for various pollutant parameters including, conductivity, chemical oxygen demand (COD), total nitrogen (TN), chloride, and sulfate. Laboratory-scale batch membrane filtration experiments were performed by using three different membrane types (BW30, XLE, and X20). The experiments were conducted at 15 and 20 bar pressures and flux data were collected during the operations. The results showed that BW30 and X20 membranes could be operated comfortably with 80% recovery for the wastewater containing low and high sulfate concentrations. For the wastewater with low sulfate concentration, the fluxes of BW30 and X20 at 20 bar were 19.7 and 16.4 L/m^2/h, respectively, at 80% recovery. On the other hand, for the wastewater with higher sulfate concentration, the fluxes of BW30 and X20 at 20 bar were 8.6 and 11.5 L/m^2/h, respectively.

Key words: membrane, membrane fouling, water recovery, water reuse

HIGHLIGHTS

- Two different wastewater samples released in the organized industrial zone were treated in the wastewater treatment plant.
- Reverse osmosis filtration process was applied using three different membrane types (BW30, XLE, and X20).
- X20 membrane was chosen as the most suitable membrane type with 80% water recovery under 20 bar pressure.

1. INTRODUCTION

The availability of water is the most important supporter of the socio-economic development of man and his society. The water demand profile is determined by the population, economic-industrial development, and consumption patterns. In recent years, water demand has increased by 1% per year worldwide, but this rate is rising even faster in developing countries due to industrial advances (Santos *et al.* 2020). In addition, increasing environmental concerns due to the disposal of industrial wastes and the economic dimension of resource consumption are an important issue that constantly draws the attention of the industry (Xu *et al.* 2022). Due to increasing water demand and decreasing water availability, a future restriction on access to water is envisaged, which may hinder community development. Considering these concerns, water recovery is of great importance.

The preferred process for water recovery can be determined by the water quality requirements (Ozbey-Unal *et al.* 2020). The water recovered with an appropriate treatment technology that meets certain usage criteria can be used for purposes such as irrigation, industrial use, and toilet flushing (Gundogdu *et al.* 2019). Methods involving only physicochemical and biological processes do not produce water of the targeted quality. Membrane technologies offer an important solution in wastewater discharge, water reuse and recovery, recycling valuable components from waste streams to achieve environmental standards (Bunani *et al.* 2013). This makes membrane technologies popular, including microfiltration (MF), ultrafiltration

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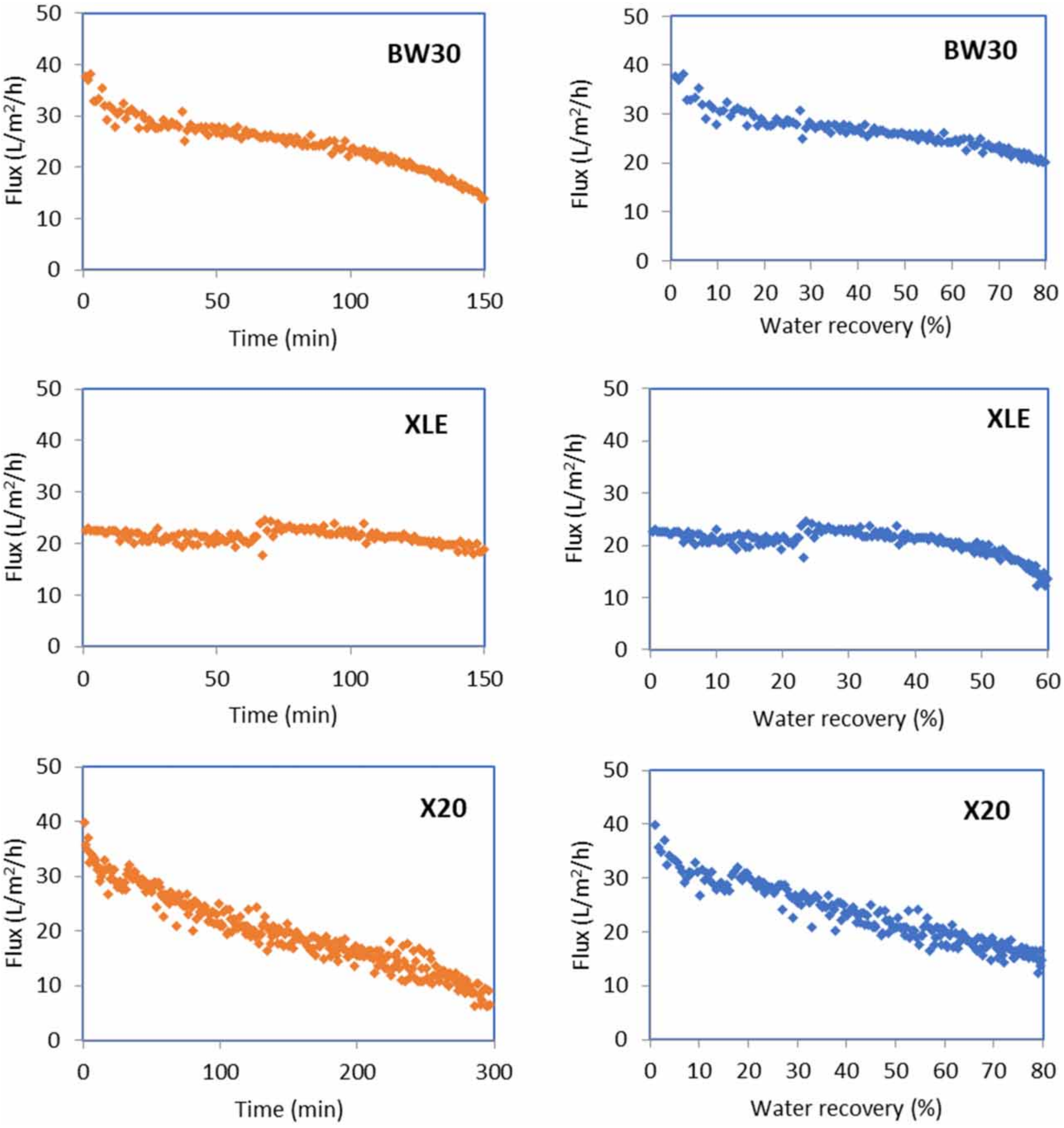

Figure 1 | The graphs of flux-time and flux-percent recovery (TMP = 20 bar).

(UF), nanofiltration (NF), and reverse osmosis (RO). In particular, NF and RO membranes are more effective in removing salt, organic pollutants, and ions that cause hardness in water, resulting in very high quality water (Bunani *et al.* 2013; Xiao *et al.* 2014). MF or UF, mostly come to the forefront with their micro-organism and high pathogen reduction properties during the treatment of wastewater, and can prolong the service life by reducing the contamination of the RO membrane, which provides the basic purification (Ning & Troyer 2007). NF performs particularly well in removing color components from textile wastewater, but is concerned with removing dyes, which are relatively large organic molecules and in most cases have electrical charges (Frank *et al.* 2002). RO technology, on the other hand, is widely preferred by various industries such as chemistry, electrochemistry, textile, paper, leather, food and even petrochemistry. Recent research has shown that it is highly effective in wastewater containing dissolved species aimed at water recovery by RO (Rubio-Clemente *et al.* 2015; Colla *et al.* 2016; Venzke *et al.* 2018).

In a study in which the wastewater coming from the balancing tank of the automotive industry was purified using the electrocoagulation technique with aluminum electrodes. This study was carried out with a treatment time of 50 min and an electric current density of 20 mA/cm^2, 99.9% of metal ions, and oil and grease were reduced below the detection limit. However, since the formation of $Al(OH)_3$ from aluminum electrodes increases the pH value of the wastewater, pH adjustment was required before discharge (Zini *et al.* 2020). In the study of Sarioglu & Gökçek (2016), it was carried out with anaerobic treatment, in the presence of molasses as an auxiliary substrate in batch mode at mesophilic temperature, and COD removal was achieved at the rate of 47%. In a study in which the biological treatability of the dye house wastewater together with the mainstream wastewater of the automotive industry was tested with a sequencing batch reactor, the carbon and nitrogen removal performance was obtained as 89 and 58%, respectively, in the case of treating wastewater alone. However, it was observed that efficient removal could not be achieved by including dye house waste into the wastewater (Guven *et al.* 2017). This situation creates an incentive for the investigation of membrane processes for automotive wastewater.

Membrane fouling control is one of the important problem of membrane processes. Since RO membranes are non-porous, surface fouling is observed rather than pore clogging (Ahmed *et al.* 2023). Increasing the concentration of rejected salt leads to the formation of a cake layer (Yu *et al.* 2021). This condition can be more easily managed than pore clogging. In RO membranes, fouling occurs in the form of biological, organic, colloidal, and calcification (Du *et al.* 2017).

The aim of this study is to determine the appropriate process by applying an advanced treatment process and to investigate the reusability of the effluent of the organized industrial zone wastewater treatment plant. The wastewater is mainly generated from automotive industries. Automotive industries use large volumes of water with organic and inorganic pollutants. Water recovery and reuse of such mixed wastewater is not well documented in the literature. Within the scope of the study, the appropriate membrane type was determined depending on both the flux and rejection properties. By characterizing the recovered water, the treatment performance was determined and compared. Fouling trends and conditions of the membranes were investigated.

2. MATERIALS AND METHODS

2.1. Definition of an organized industrial zone wastewater treatment plant

Wastewater was obtained from Asım Kibar Organized Industrial Zone Wastewater Treatment Plant (AKOSB WWTP), Izmit, Turkey. The industrial wastewater reaching AKOSB WWTP is provided to the infrastructure network and reaches the WWTP without being subjected to any treatment. The wastewater reaching the centralized WWTP is treated to meet the discharge limits of the Water Pollution Control Regulation of Turkey. The primary activities within the AKOSB involve operations within the automotive manufacturing sector. AKOSB WWTP was designed for an inflow rate of 4,000 m^3/day. The WWTP includes pretreatment (coarse and fine screen, oil/sand trap, and balancing), chemical treatment, and biological treatment followed by sand filtration and disinfection processes. Chemical treatment is operated according to the pollution value of the wastewater. If the phosphate and heavy metal concentrations of the wastewater are high, the chemical treatment is operated followed by the biological treatment. A decanter is used to dewater the sludge formed in the facility.

2.2. Properties of the treated wastewater

The treated wastewater, used for water reusability studies, was obtained from the WWTP effluent and named as 'treated wastewater' for the studies. The properties of the treated wastewater used in the experiments are shown in Table 1. During the studies, the treated wastewater samples were taken from the WWTP effluent at two different times. The first sample had the characteristics of the wastewater that usually was discharged from the WWTP, and the second sample was an example with a high sulfate content, which was observed less frequently.

The criteria followed to determine the membrane performance are rejected salt, pressure drop, and permeate flux. A decrease in the membrane performance is observed as a result of the accumulation of rejected salt on the membrane surface

Table 1 | Properties of treated wastewater

	Conductivity (μS/cm)	COD (mg/L)	TN (mg/L)	Cl^- (mg/L)	SO_4^{-2} (mg/L)
Sample 1	1,720	74	7.2	104.8	380
Sample 2	3,550	90	5.3	215.8	1,332
WPCR[a]	–	400	20	–	1,500

[a] Water Pollution Control Regulation Limits (for 2-h composite samples).

(An *et al.* 2023). Sulfate compounds are one of the most common residues in RO processes and cause a strong blockage of the membrane surface (Su *et al.* 2018; Melliti *et al.* 2023). The characteristics of the wastewater and operating conditions are effective in delaying the blockage, but it has not yet been possible to prevent sulfate blockage, even in processes with effective pretreatment (Nghiem & Cath 2011; Lu *et al.* 2023). It allows the performance of membranes using sulfate-containing wastewater to be determined in a short time under challenging conditions.

2.3. Properties of the membranes

Laboratory-scale batch membrane filtration experiments were carried out in order to determine an appropriate membrane based on the rejection performance of the pollutant parameters. Three different membrane types were used in the experiments, chosen based on their commercial availability in the market. The membrane properties of thin-film composite membranes made by interface polymerization are determined by the materials used in the thin layer, and this allows membrane production according to the properties of the water (Liu *et al.* 2011). It is frequently preferred due to its high salt rejection, tolerance of wide pH range, high permeability, and has a wide range of use. However, despite this high flux and selectivity effect, its resistance to chlorine is low. In order to increase the chlorine resistance, amide nitrogen or aromatic rings must be added to the polyamide layer (Kwon *et al.* 2006; Suresh *et al.* 2022). Although the addition of aromatic rings delayed membrane fouling, it did not show high resistance to chlorine (Liu *et al.* 2006). It has been determined that polyamide-urea membranes produced with the addition of m-phenylenediamine (MPD) have high chlorine resistance (Liu *et al.* 2006, 2008; Kamali & Khodaparast 2015). In addition, it provides long-term resistance to highly acidic and highly alkaline conditions (Zhao *et al.* 2023).

The treated wastewater sample taken from the wastewater treatment plant effluent was passed through an UF (UC100) membrane before RO filtration and then NF membrane studies were carried out. The experiments were carried out with three different RO membranes at 15 and 20 bar pressures. The membranes used in the experiments and their properties are presented in Table 2.

2.4. Methods for the analyses

The conductivity was measured by multimeter (340i WTW). The chemical oxygen demand (COD) was measured using the closed reflux method-titrimetric method as described in Standard Method No. 5220C. The concentration of the chloride was analyzed by Argentometric Method (Standard Method No. 4500B). Total nitrogen (TN) was measured by the process which uses a high temperature combustion catalyst reaction (typically 720–950 °C) with a catalyst and oxygen method following Standard Method No. 4500-N. Sulfate (SO_4^{-2}) was measure by the Standard Method No. 4500- SO_4^{-2}-C.

Table 2 | RO membranes used in the filtration experiments

	BW30	**XLE**	**X20**
Membrane type	Polyamide thin film composite	Polyamide thin film composite	Polyamide-urea thin film composite
Salt rejection (%)	99.0	99.0	98.5
Max. pressure (bar)	41	41	41
pH range	2–11	2–11	4–11
Chlorine tolerance	<0.1 ppm	<0.1 ppm	<0.1 ppm

Table 3 | Flux-percent recovery values for sample 1 (TMP = 20 bar)

	Recovery rate (%)							
Flux (L/m²/h)	**10**	**20**	**30**	**40**	**50**	**60**	**70**	**80**
BW30	30.4	27.5	27.1	27.1	25.5	24.2	22.2	19.7
XLE	23.0	22.6	21.8	21.0	19.1	13.6	-	-
X20	30.0	29.0	25.5	23.0	21.0	20.1	17.7	16.4

The removal efficiency was calculated using Equation (1). All experiments were repeated in duplicate and the averages of the results were presented.

$$\text{Removal efficiency } (\%) = \frac{\text{Initial concentration} - \text{Final concentration}}{\text{Initial concentration}} \tag{1}$$

The morphological definition and chemical composition of the membranes were observed by scanning electron microscopy (SEM, Zeiss Supra 55, Germany).

In order to determine the membrane performance, flux versus time and flux vs. percent recovery relationships were obtained. The flux was calculated using Equation (2).

$$\text{Flux } (\text{L/m}^2/\text{h}) = \frac{\text{Hourly treated water that passes through the permeate(L/h)}}{\text{Surface of the membrane } (\text{m}^2)} \tag{2}$$

3. RESULTS AND DISCUSSION

3.1. Experimental results for sample 1

The flux versus percent recovery values obtained for each membrane filtration is summarized in Table 3. According to these results, BW30 membrane supplied better results than XLE and X20 membranes in terms of flux. The results showed that BW30 and X20 membranes could be operated comfortably with a high recovery (80%). Since the flux of the XLE membrane was substantially lower than that of BW30 and X20, higher recoveries were not achieved at reasonable flux values. Therefore, the filtration was stopped at 60% recovery when the XLE membrane was used.

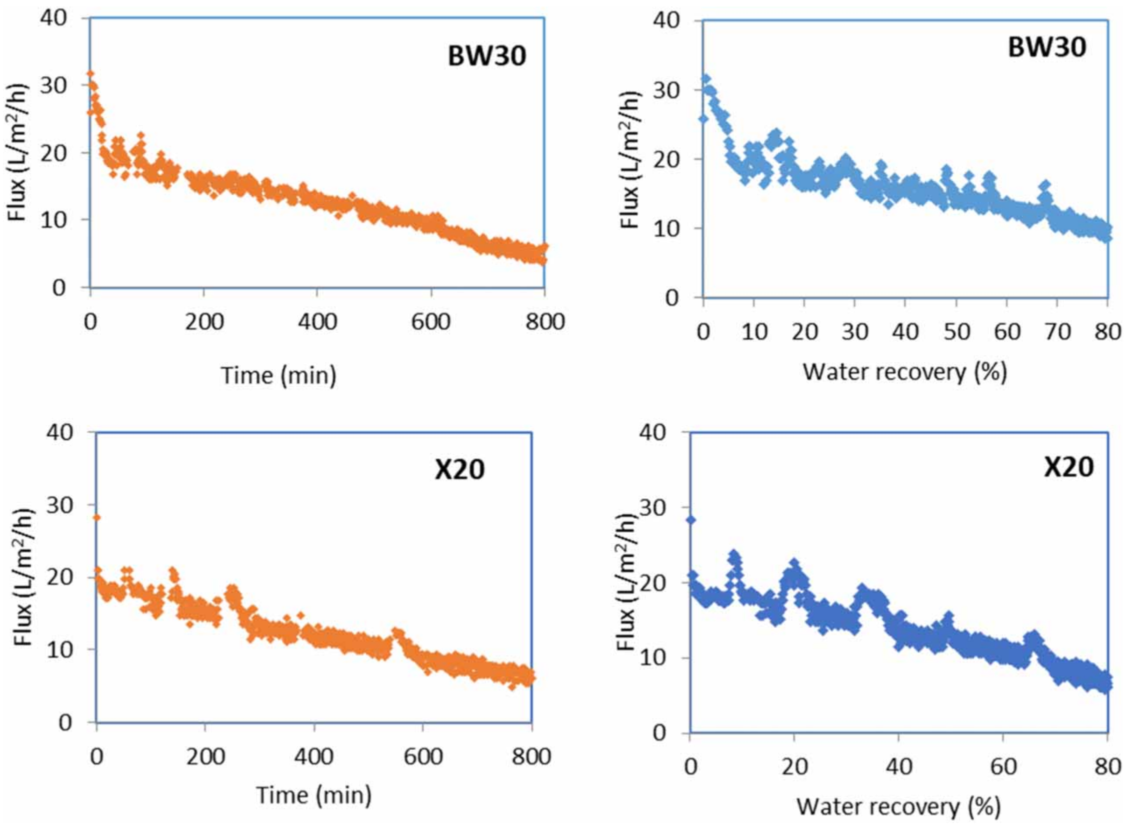

Figure 2 | The graphs of flux-time and flux-percent recovery (TMP = 15 bar).

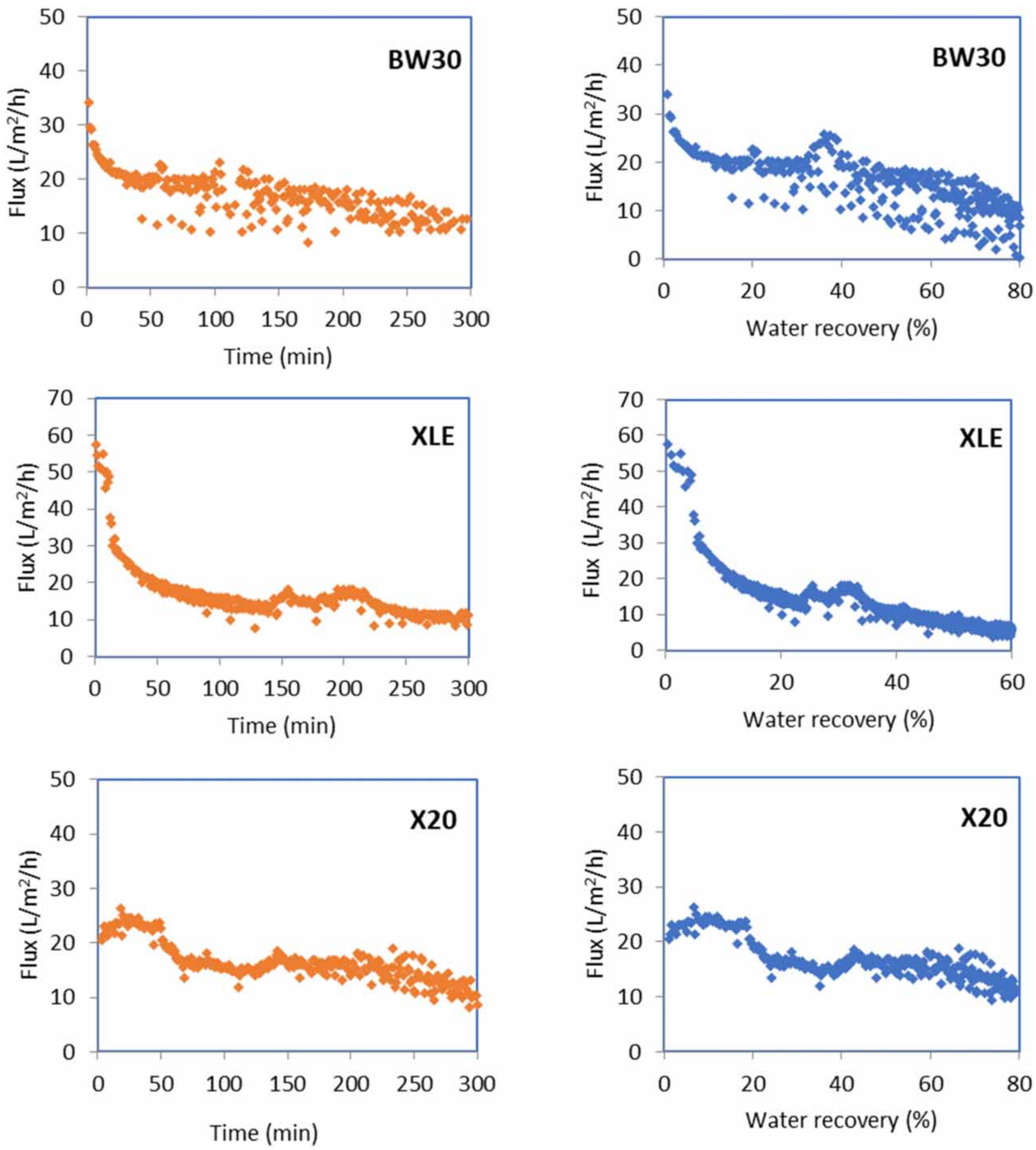

Figure 3 | The graphs of flux-time and flux-percent recovery (TMP = 20 bar).

Table 4 | Flux-percent recovery values for sample 2 (TMP = 15 and 20 bar)

	Recovery rate (%)							
Flux (L/m^2/h)	**10**	**20**	**30**	**40**	**50**	**60**	**70**	**80**
BW30-15 bar	19.7	16.8	16.4	15.6	14.4	12.7	11.9	8.1
X20-15 bar	20.1	18.2	14.9	14.1	12.7	10.8	8.0	7.0
BW30-20 bar	21.0	19.7	18.5	17.5	14.7	13.3	11.4	8.6
XLE-20 bar	21.6	16.0	15.8	9.5	7.4	5.9	–	–
X20-20 bar	24.2	19.0	16.0	15.7	15.6	14.7	11.8	11.5

Table 5 | Effluent water values of UC010 and RO membranes (TMP = 20 bar)

Sample name	Membrane type	Conductivity (μS/cm)	COD (mg/L)	TN (mg/L)	Cl^- (mg/L)	SO_4^{-2} (mg/L)
Sample 1 (wastewater sample with low sulfate)	**UC010 permeate**	1,610	36–41	4.1	297.6	257.6
	X20 permeate	151	<10	–	62.3	27.5
	XLE permeate	481	12–28.4	–	105	39.1
	BW30 permeate	532	20.8	3.2	95.7	70.3
Sample 2 (wastewater sample with high sulfate)	**UC010 permeate**	3,665	81	2.8	188.7	1,338
	X20 permeate	235	<10	1.7	21.5	44.3
	XLE permeate	206	<10	2	19.2	41.9
	BW30 permeate	190	<10	1.8	38.7	71.9

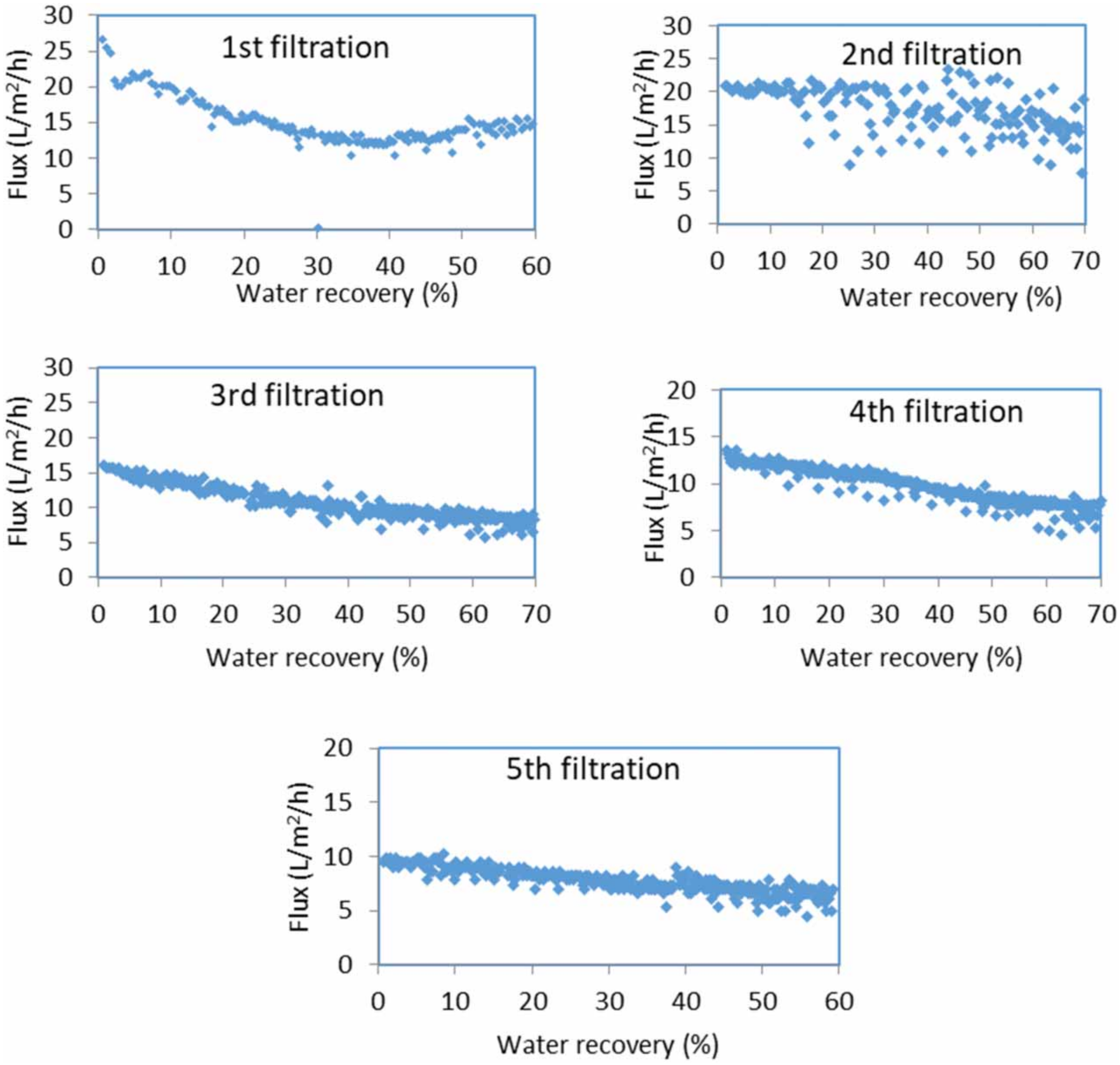

Figure 4 | Flux-percent recovery rates obtained in repeated filtration experiments (BW-30 membrane at TMP = 20 bar).

3.2. Experimental results for sample 2

The filtration experiments performed for sample 2 were carried out at both 15 and 20 bar pressures for X20 and BW30, but only at 20 bar for XLE due to the poor filtration performance with the sample 1. The results are presented in Figures 2 and 3.

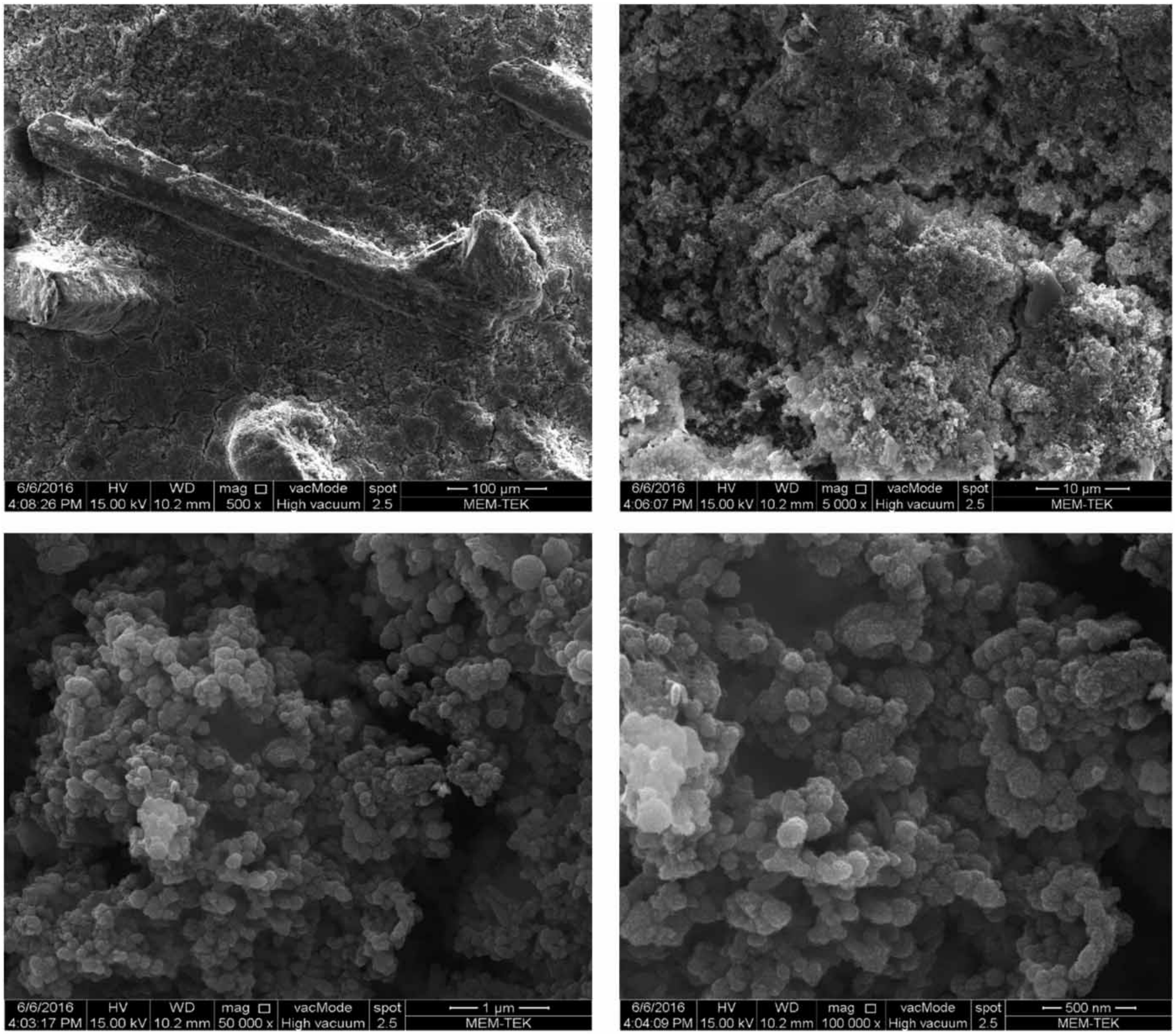

Figure 5 | SEM images of different magnifications obtained from fouled membrane.

The recovery rates obtained under both 15 bar and 20 bar pressure are summarized in Table 4. It was determined that the membranes giving the best flux results in the wastewater sample with the maximum load were BW30, X20, and XLE, respectively. Similar to sample 1, flux performance of the XLE membrane was poor (5.9 L/m^2/h at 60% recovery) in sample 2 and therefore, 80% recovery was not obtained at reasonable time frames.

The permeate composition of UF and RO is presented in Table 5. The UC010 filtrate shows similar characteristics to the feed water (treated wastewater). In general, all parameters are below the pollutant discharge standards as given in Table 1. In the first sample with an average load, the UF permeate sulfate concentration was 258 mg/L, while the chloride concentration was 298 mg/L. The total conductivity was measured as 1,610 μS/cm. The sulfate concentration of the second sample with the maximum load was 1,338 mg/L, while the chloride concentration was 189 mg/L and the conductivity was 3,665 μS/cm. Among the tested membranes, the X20 membrane retained higher chloride and sulfate concentration and was found to be the membrane with the lowest effluent conductivity. Especially in the first sample, the effect on the conductivity value of the X20 membrane was more evident due to the partially higher chloride and lower sulfate content. However, since the sulfate concentration was high in the second sample and the removal efficiencies were close to each other, the conductivity values were similar. Chemical analysis results show that all membrane types could be used in the process, but the X20 membrane performed better in the presence of high chloride concentration. However, in general, no significant difference was found among the membranes in terms of chemical substance retention.

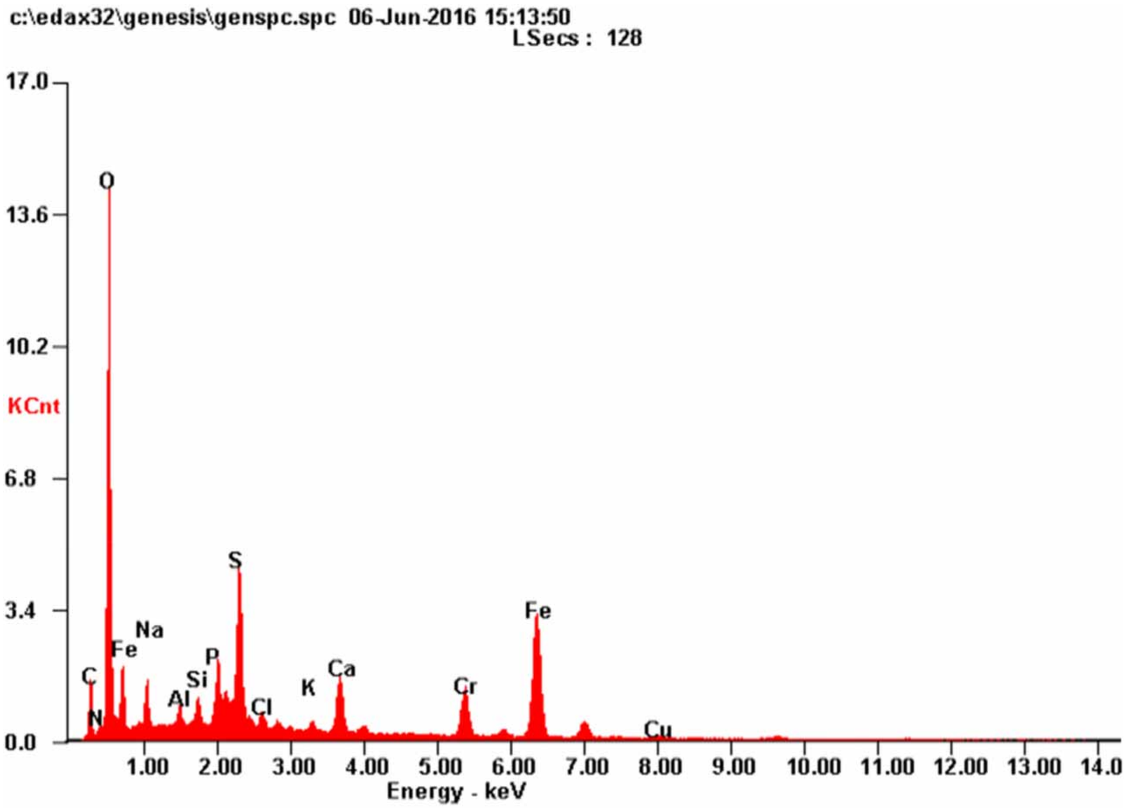

Figure 6 | EDX results for fouled membrane.

Table 6 | Weight and atomic percentages of elements causing membrane fouling

Element	Weight (%)	Atomic (%)
C	11.40	19.97
N	2.12	3.19
O	40.43	53.16
Na	3.84	3.51
Al	0.94	0.73
Si	1.26	0.94
P	2.69	1.83
S	6.44	4.22
Cl	0.78	0.46
K	0.45	0.24
Ca	3.18	1.67
Cr	5.20	2.11
Fe	20.33	7.66
Cu	0.95	0.31

3.3. Membrane fouling studies

In order to investigate membrane fouling, the membrane filtration experiments were performed five times using the same membrane. In the classical filtration process, the flux drop is mainly due to both foulants and increased osmotic pressure as the filtration takes place. For this reason, the change in the initial fluxes of the filtration was investigated using the same membrane, and this change was associated with fouling. In Figure 4, flux and percent recovery graphs are given. As a result of the filtration experiments, the initial flux was 25 L/m^2/h in the first filtration, these values were 21 L/m^2/h in the second filtration, 16 L/m^2/h in the third filtration, 13 L/m^2/h in the fourth filtration and it decreased to 10 L/m^2/h in the final filtration. The results showed that there were significant flux reductions due to membrane fouling.

After the last filtration step, the membrane was removed from the system and SEM-EDX was performed to investigate the cause of foulants. SEM images and EDX results are presented in Figures 5 and 6, respectively. Moreover, the weight percentages of the elements caused by the fouling on the membrane are presented in Table 5. The results showed that foulants with both organic and inorganic content were present. Iron, chromium, and calcium were the most important types of inorganic foulants. Especially, the solubility of $CaSO_4$ is very low at over a large pH range. The solubility of $CaSO_4$ is 0.411 mg/L at pH vales in between 3.5 and 11 (Peng *et al.* 2009). Therefore, the formation of $CaSO_4$ was the major inorganic foulant as suggested by EDX results. In order to prevent organic foulants, activated carbon treatment after UF and then RO membrane filtration can prevent organic fouling. For this purpose, an activated carbon study was carried out. In addition, anti-scalant should be used to prevent inorganic contamination.

4. CONCLUSION

In this study, the reuse potential of an organized industrial zone wastewater was investigated by laboratory-scale membrane processes. Three types of RO membranes (BW30, XLE, and X20) were used in the experiments. Bot flux and chemical rejection performances of each membrane were determined at 15 and 20 bar operational pressures. The experimental results showed that BW30 and X20 membranes could be operated comfortably with 80% recovery for the wastewater containing low and high sulfate concentrations. For the wastewater with low sulfate concentration, the fluxes of BW30 and X20 at 20 bar were 19.7 and 16.4 L/m^2/h, respectively, at 80% recovery. On the other hand, for the wastewater with higher sulfate concentration, the fluxes of BW30 and X20 at 20 bar were 8.6 and 11.5 L/m^2/h, respectively. The X20 membrane showed slightly better performance with wastewater containing high sulfate concentrations. In order to investigate the fouling behavior of the RO membranes, the filtration experiments were conducted in five cycles. Results showed a steady decrease in membrane flux in consecutive use. The initial flux was 25 L/m^2/h in the first filtration; however, it decreased to 21, 16, 13, 10 L/m^2/h in the second, third, fourth, and fifth filtrations, respectively. The initial flux decreased by about 60% after the fifth use. The results showed that foulants with both organic and inorganic content were present. Iron, chromium, and calcium were the most important types of inorganic foulants. In larger scale applications, foulants especially calcium should be controlled since the formation of $CaSO_4$ on the membrane surface is a major fouling which is very difficult to remove due to low and constant solubility at pH between 3.5 and 11. Furthermore, organic fouling reduction by activated carbon need to be investigated.

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AUTHORS' CONTRIBUTIONS

Z.B.O. performed methodology and data curation. A.K. and B.K. performed investigation and wrote the original draft. A.K. and N.D. performed conceptualization, wrote the original draft, did formal analysis, reviewed, and edited the article. All authors read and approved the final manuscript.

DATA AVAILABILITY STATEMENT

Data cannot be made publicly available; readers should contact the corresponding author for details.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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