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# NF-RO Membrane Performance for Treating the Effluent of an Organized Industrial Zone Wastewater Treatment Plant: Effect of Different UF Types

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**Abstract:** Reuse of water is necessary in Organized Industrial Zones (OIZ) due to excessive use of groundwater in semi-arid Turkey. Membrane treatment of the OIZ wastewater treatment plant (WWTP) effluents should be explored for new treatment options. In this study, three different UF membranes with variable molecular weight cutoff (MWCO) values (4, 10, and 250 kDa) were used to treat the effluent of an OIZ WWTP at laboratory scale. Six different nanofiltration (NF) and reverse osmosis (RO) membranes were used for the effluent of UF membranes to evaluate the difference in the membrane filtration performance and the water quality. Effluent electrical conductivity (EC) values of NF membranes were 1.77 ± 0.17, 3.73 ± 0.27, and 4.20 ± 0.23 ms/cm for NF (NF90, NF270, and TM610) membranes, respectively while they were 0.83 ± 0.47, 1.17 ± 0.47, and 1.13 ± 0.57 ms/cm for RO (XLE, AD90, and BW30) membranes, respectively. Scanning electron microscope (SEM), energy dispersive X-ray Spectroscopy (EDS), and confocal laser scanning microscope (CLSM) images showed severe biofouling in UF 4 kDa and UF 10 kDa membranes whereas UF 250 kDa membrane showed larger metal precipitates and little bacterial fouling. The results indicated that OIZ WWTP effluent could be reused as irrigation water according to Turkish regulations with UF 250 kDa and RO-XLE membranes, effectively.

**Keywords:** reuse; membrane; organized industrial zone; wastewater treatment plant

## 1. Introduction

The reuse of water is beneficial in water stressed countries when water resources are used excessively for industrial purposes [1]. Pollution of limited water resources and stringent water discharge standards results in the implementation of a well-known practice, maintaining reusable water quality [2].

In Turkey, industrial establishments are organized in a zone away from the city center to minimize environmental problems. Wastewater originating from various processes in these regions are managed collectively in mixed industrial wastewater treatment plants (WWTPs) [3]. Since the number of organized industrial zones (OIZs) has reached 300 in Turkey as of 2017, their wastewater management strategies should be improved. Water sources of these organized industrial zones are mostly freshwater which includes both the groundwater and the treated municipal water network. Conventional wastewater treatment methods are used in these regions because wastewater usually contains biodegradable municipal wastewater from the daily use of workers. However, state of the art of the treatment options should also be explored for newly established WWTPs.

Membrane bioreactors (MBRs) are one of the best solutions in terms of sustainability for mixed industrial wastewater; however, conducting an MBR in an existing conventional activated sludge (CAS) WWTP is not a good solution if the effluent meets discharge standards since the biological stabilization of the wastewater takes place in CAS treatment [4]. Membrane treatment of the effluent of a WWTP is another option for this type of wastewater. Reuse of mixed industrial wastewater could be achieved by nanofiltration (NF) or reverse osmosis (RO) filtration following pre-treatment by microfiltration (MF) or ultrafiltration (UF). Many studies are conducted about NF/RO treatment for industrial wastewater; however, very few of them are after MBR treatment [5–8]. Conducting NF or RO filtration in a WWTP has also been studied by some researchers. For industrial wastewater, the textile sector was dominant in the number of membrane treatment studies [9–11].

There is a limited number of studies about mixed industrial wastewater treatment with membrane applications. The MBR performance of industrial zone wastewater was evaluated in Iran [12], a municipal WWTP mixed with industrial discharges was determined about some micropollutants in Spain [13], and a feasibility study for MBR plant for decentralized mixed wastewater was evaluated in Vietnam [14]. In an industrial park WWTP, UF, and RO filtrations were applied to both the secondary clarifier of the WWTP and the final clarifier of the chemical coagulation unit [15]. Fouling mechanisms in the UF membrane were discussed and a suitable treatment option was proposed in this industrial park study. Also, it was stated that pre-filtration with a 1 µ filter enhanced filtration performance [15]. Selecting a membrane sequence for municipal wastewater was evaluated in many studies [5,7,16]; however, application of membrane filtration to mixed industrial wastewater treatment plants is limited. Selection of different membranes is also not a new concept for NF and RO membranes. However, selecting different membranes for NF and RO is mostly studied in the context of specific contaminant removal or specific types of wastewater [17–19]. Although there are some studies about the effects of UF membrane on different subjects, researchers did not focus on the effects of different UF types for NF and RO performance [20,21]. Also, this study is the first laboratory scale study on the effluent of an OIZ WWTP which investigated treatment options and fouling behaviors with different membranes.

In this study, an organized industrial wastewater treatment plant effluent was filtered with three different UFs and six different NF or RO membranes at a laboratory scale to evaluate the performances and effluent water qualities for potential reuse in irrigation. For this reason, permeate qualities, filtration performances, and membrane characterizations (by SEM and CLSM visualization) were also investigated.

## 2. Materials and Methods

### *2.1. Kayseri Organized Industrial Zone (KOIZ) Wastewater Treatment Plant (WWTP)*

Kayseri is located in the Central Anatolian region of Turkey, and it has three OIZ sites. KOIZ is the biggest one with about 1000 different businesses. Although most of the plants are related to furniture industry, they contributed only with municipal wastewater to the treatment plant. The WWTP was put into operation in 2013 with 20,000 $m^3$/day flowrate; however, it was reached to 30,000 $m^3$/day in the early 2017. A typical list of contributions from sectors is textile (30%), paper and recycling (30%), municipal (15%), and other (25%) sources including metal, electric-electronic, and food industry sectors with or without pre-treatment according to discharge standards set in their own regulations. The WWTP includes mainly physical pre-treatment, equalization, sedimentation, and conventional biological treatment units. The influent and effluent characterization of the WWTP is given in Table 1 as the average of two-month sampling of weekly samples.

**Table 1.** Influent and effluent water characterization of KOIZ WWTP.

| Parameter | Influent | Effluent |
|---|---|---|
| COD (mg/L) | 1233 ± 67.4 | 81.9 ± 7.4 |
| SS (mg/L) | 1189 ± 90.2 | 28.6 ± 3.1 |
| TN (mg/L) | 27.1 ± 9.3 | 2.9 ± 1.0 |
| TP (mg/L) | 4.4 ± 1.1 | 0.1 ± 0.08 |
| EC (ms/cm) | 6.05 ± 0.9 | 5.44 ± 1.3 |
| Cu (mg/L) | 4.8 ± 0.79 | 0.2 ± 0.03 |
| Zn (mg/L) | 1.3 ± 0.31 | 0.2 ± 0.03 |
| Pb (mg/L) | 0.9 ±0.25 | 0.2 ± 0.06 |
| Fe (mg/L) | 13.9 ± 2.96 | 0.7 ± 0.23 |
| $Cr^{6+}$ (mg/L) | 1.0 ± 0.22 | 0.0 ± 0.00 |
| Total Cr (mg/L) | 1.6 ± 0.22 | 0.1 ± 0.22 |
| Cd (mg/L) | 0.0 ± 0.00 | 0.0 ± 0.00 |

### 2.2. Lab-Scale Filtration System

Effluent water from the final sedimentation tank of KOIZ WWTP was brought into the laboratory and kept in +4 °C until the experimentation was over. HP4750 Stirred Cell (Sterlitech, WA, USA) was used in all filtration experiments. The cylindrical cell had 300 mL capacity, and was 47 mm in diameter with a 14.7 cm$^2$ effective membrane area. Separation was provided with $N_2$ gas at 3 bar pressure for ultrafiltration (UF) campaign, while they were 3-6-9-12 bars for NF and RO membranes. Recovery rates were chosen as 90, 70, and 50% for UF, NF, and RO experiments, respectively. Flux was measured as $L \cdot m^{-2} \cdot h^{-1}$ (LMH) with a balance connected to a computer. Filtration process was terminated when the flux fell below 2 LMH.

The UF membranes were selected with different molecular weight cutoff (MWCO) values. The specifications of the UF and NF/RO membranes are given in Tables 2 and 3, respectively. Data were obtained from the literature of membrane manufacturers and additional information was adapted from the literature [22].

**Table 2.** Properties of UF membranes.

| Membrane Name | Pore Size (μ) | MWCO (kDa) | Material | Brand |
|---|---|---|---|---|
| UF 4 kDa | 0.07 | 4 | PES | Philos |
| UF 10 kDa | 0.10 | 10 | PES | Philos |
| UF 250 kDa | 0.44 | 250 | PVDF | Philos |

**Table 3.** Properties of NF/RO membranes.

| Membranes | Membrane Material | Max. Oper. Temp. (°C) | Max. Oper. Pres. (Bar) | pH Range | MWCO (Da) | Salt Rejection (%) |
|---|---|---|---|---|---|---|
| NF90 | PA | 35 | 41 | 4–11 | 201 ± 25 | 85–95 |
| NF270 | PA | 45 | 35 | 3–10 | 330 ± 48 | 40–60 |
| TM610 | PA | 45 | 60 | 2–11 | 100 | 80–97 |
| BW30 | PA | 45 | 41 | 2–11 | 116 ± 30 | 99.5 |
| XLE | PA | 45 | 41 | - | - | 95 |
| AD90 | PA | 50 | 82 | 2–11 | - | 99.75 |

### 2.3. Experimental Methods

Analytical experiments were conducted for chemical oxygen demand (COD), pH, turbidity, and electrical conductivity (EC) in both the influent and effluent of filtration experiments. Hach (HQ40D) multimeter was used for pH, EC, and temperature. COD was measured according to standard methods 5220-C [23]. Turbidity was measured with a turbidimeter (Hach 2100Q, Düsseldorf, Germany). The metal analysis of the KOIZ was also carried out with metal measuring kits (Hach).

*2.4. Membrane Characterization*

Scanning electron microscope (SEM) were conducted using FEI Quanta FEG 200 (Thermofisher Scientific, Eindhoven, The Netherlands) in Environmental Scanning Electron Microscope (ESEM) mode. Energy dispersive X-ray (EDX) analysis of the membranes were conducted using AMATEK EDAX Apollo X (Amatek GmbH, Wiesbaden, Germany). Confocal Laser Scanning Microscope (CLSM) observations were done using Nikon C2 Confocal microscope (Nikon, Tokyo, Japan). Membranes were stained with LIVE/DEAD BacLight™ Bacterial Viability Kit (Thermofisher Scientific, Eindhoven, The Netherlands). After staining procedure, 1 × 1 cm membranes were kept for 30 min. NIS-Elements AR 4.10.01 program was used with 10× lens. Yellow spots represent living bacteria, while red spots represent dead bacteria.

## 3. Results and Discussion

*3.1. UF Filtration of KOIZ Effluent*

Flux–time profiles of the UF membranes treating KOIZ WWTP effluent are shown in Figure 1. Clearly the fluxes are directly proportional to the pore sizes of the membranes. In the first minute of UF 250 kDa membrane filtration, the flux was 297 LMH (data not shown on the graph). This filtration lasted one and a half hour while it took more than four hours for UF 4 kDa membrane. This was not only due to the pore sizes but also due to the membrane materials since PVDF membranes showed lower cake resistances than PES membranes [24]. Surface hydrophobicity may be another reason for flux decline in PES UF membranes, with MWCOs of 4 kDa and 10 kDa regardless of the pore sizes [25,26]. A severe flux decline of UF 250 was due to the very large pore size of this membrane compared to UF 4. Pore blocking of UF 250 may be enhanced while cake blocking could be the reason for UF 4 as it is discussed in the next section.

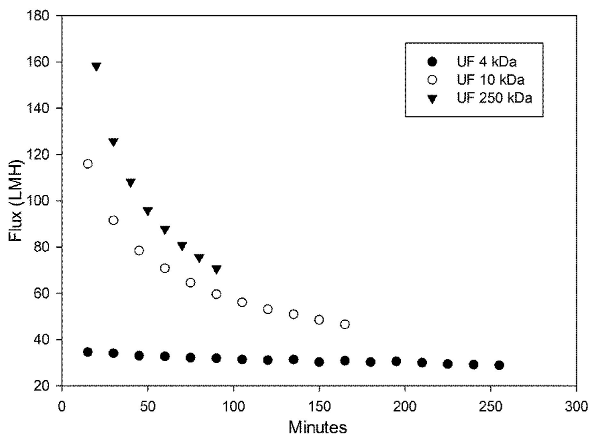


**Figure 1.** Flux-time profile of the UF membranes at constant pressure of 3.0 bars. Data in the figure indicates average fluxes of all runs.

Water qualities of the UF effluents are similar to each other as given in Table 4. Most of the ions were not rejected in UF filtrations. Therefore, EC of the water was not changed significantly after filtration as it was expected.

**Table 4.** Effluent water qualities of UF membranes.

| Membrane | pH | COD (mg/L) | EC (ms/cm) | Turbidity (NTU) | Flux (LMH) |
|---|---|---|---|---|---|
| UF 4 kDa | 8.61 ± 0.36 | 73.5 ± 16.4 | 4.21 ± 0.25 | 0.85 ± 0.12 | 35.1 ± 11.5 |
| UF 10 kDa | 8.75 ± 0.47 | 83.0 ± 12.7 | 4.33 ± 0.21 | 0.88 ± 0.08 | 52.9 ± 4.1 |
| UF 250 kDa | 8.72 ± 0.25 | 46.9 ± 21.3 | 4.02 ± 0.33 | 1.10 ± 0.14 | 74.2 ± 5.1 |

Some UF filters differed from the other ones since further NF/RO filtrations yielded better results with that UF filter than the others. RO membrane filtrations showed that each RO membrane performed better with one UF filter as shown in Figure 2. The UF 4 membrane showed better performance in terms of fluxes when it was further filtrated with NF270. Similarly, UF 250 membrane indicated significantly better pre-filtration performance than the other UF membranes when it was further filtrated with an XLE membrane. The BW30 and AD90 RO membranes showed little flux values, up to 6 LMH at AD90, and up to 2 LMH with BW30 membranes at 12 bars pressure. UF10 membrane showed better performance with BW30 membrane as it had higher flux values than the other UF membranes in all pressures. In Figure 2, however, UF performances were not significant indicators for some membranes since NF90, BW30, and GE membranes reached fluxes not above 5 LMH.

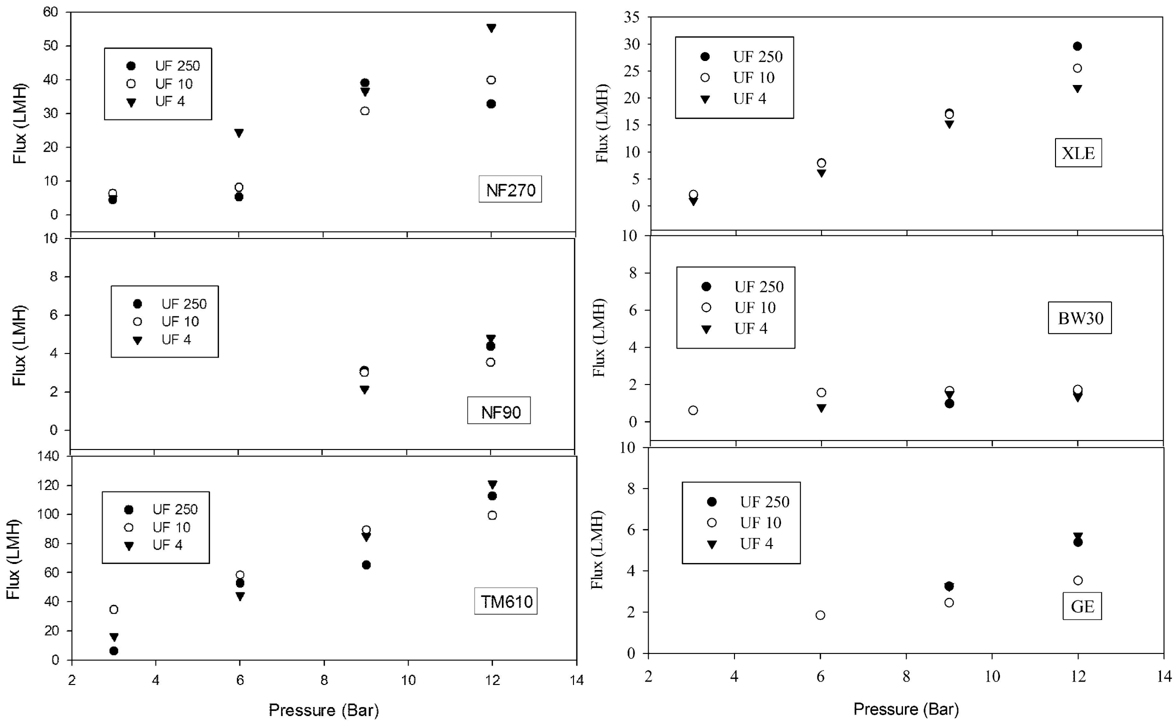


**Figure 2.** Performances of UF filters in different NF-RO membranes at varying pressures.

### 3.2. Characterization of UF Filters

In order to evaluate the fouling behavior of different membranes, SEM images and SEM-EDS analysis were performed to visualize the membranes. Figure 3 shows the SEM images for UF membranes. UF 4 kDa and UF 10 kDa membranes were PES membranes with a hydrophobic nature and low MWCO values. Despite its hydrophobic structure, UF 250 kDa membrane showed a lower fouling tendency than the rest of the membranes as it showed large openings between precipitates and broken cake layers (Figure 3c). Additionally, there were more and larger metal precipitates on the UF 250 kDa PVDF membrane than the other PES membranes. This layer on the membrane surface made the fluxes higher than the other PES membranes, which may make the membrane surface less resistive.

Therefore, UF 250 PVDF membrane exhibited pore blocking instead of cake blocking which was seen in UF 4. These results were in accordance with an earlier study which stated that the PES membranes had high pore resistance ($R_p$) than both the PVDF and mixed cellulose esters (MCE) [24].

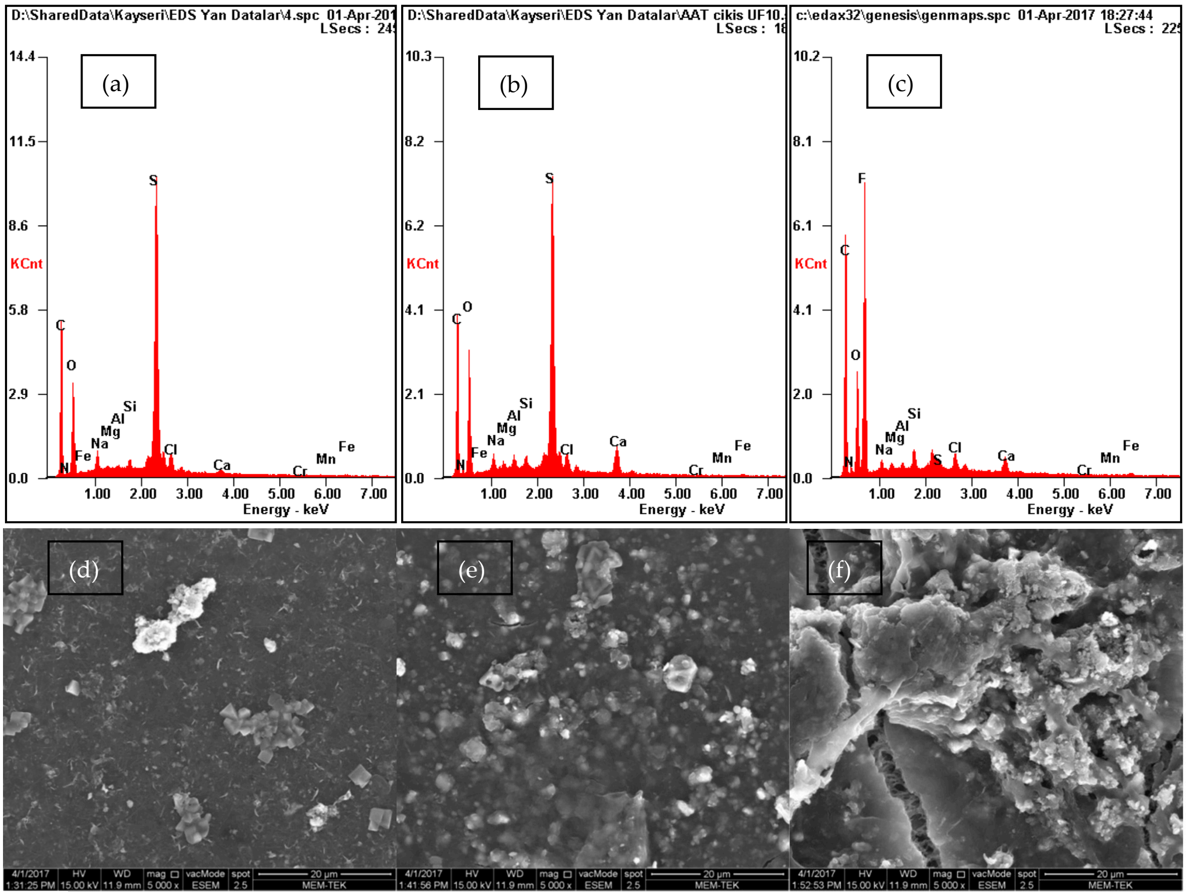


**Figure 3.** SEM-EDS analysis and SEM photographs of UF membranes; (**a**,**d**) UF 4 kDa membrane; (**b**,**e**) UF 10 kDa membrane; (**c**,**f**) UF 250 kDa membrane.

SEM-EDS analysis showed some differences the UF membranes. Excluding C, N, O, and S; F, Na and Cl were the dominant elements for UF 4 kDa membrane; Ca and Cl were the dominant elements for UF 10 kDa membrane; and Ca, Na, Cl, and Si were the dominant elements for UF 250 kDa membrane as shown in Figure 3a–c. These results indicated that UF 250 kDa membrane showed different and large precipitates agglomeration as it was seen in SEM images. Additionally, in Figure 3a,b severe cake blocking could be seen while in Figure 3c, cake blocking could be hindered by precipitates.

Confocal laser scanning microscope (CLSM) surface images of the UF membranes were shown on Figure 4a–c. CLSM visualization of the three UF membranes clearly showed that highest biofouling was observed in UF 4 kDa PES membrane. The result was supported by the low flux of that membrane. The highest dead bacterial cell layer was observed in UF 10 kDa membrane. Bacterial growth was not present on UF 250 kDa membrane's surface. SEM and CLSM images indicated that the precipitates were dominant on the membrane surface, not the bacterial cells.

Cross sectional views of the three membranes (Figure 4d–f) showed the thinnest biofilm layer was observed in the lowest pore sized membrane, UF 4 kDa. This result revealed that the biofilm layer fouled the membrane, blocking the pores. Thicker cake layer was observed in larger pore sized membranes, UF 10 kDa and UF 250 kDa. This finding has not been stated before as the was the first time WWTP effluent has been used in these membranes. However, it was stated that a modified PVDF membrane performed better than the original one as its mean pore size was increased [27]. In this study, the larger pore sized UF 250 kDa membrane enhanced the filtration efficiency, enabling the precipitates stay on the surface as it was a modified membrane.

CLSM images, SEM images, and fluxes of UF 250 kDa membrane showed that precipitates provided a filter layer for membrane without biofouling. This was not only due to the large pore size of the UF 250 kDa membrane but also due to the hydrophobic environment of the PVDF structure [28].

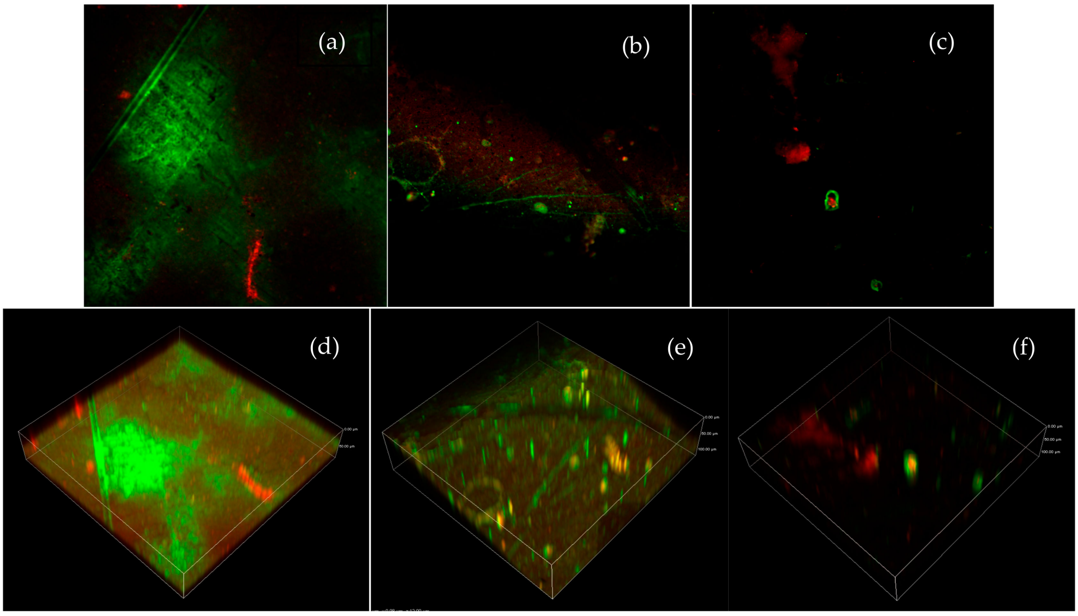


**Figure 4.** Confocal laser scanning microscope (CLSM) surface images of UF membranes; (**a**) UF 4 kDa membrane; (**b**) UF 10 kDa membrane; (**c**) UF 250 kDa membrane. Cross sectional images; (**d**) UF 4 kDa membrane; (**e**) UF 10 kDa membrane; (**f**) UF 250 kDa membrane.

### *3.3. NF/RO Treatment of UF Effluents*

Within three NF and three RO membranes, the TM610 membrane had the highest flux and the lowest water quality. This may be due to the fragile structure of the membrane since most of the experiments were repeated because of the tearing of the membrane surface. Therefore, inconsistent results were observed. The results of this membrane were not shown in the rest of the paper except for Table 5 in which EC values of the membranes are presented. As it is seen on the Table 5, among all membranes, TM610 and NF270 have the worst effluent qualities. Conductivity is the key parameter in Turkish irrigation water standards [29] since salinity related parameters like total dissolved solids (TDS), Na, Cl, and sodium adsorption rate (SAR) are in relation with the EC value. Recent studies have focused on the rejection and effects of salinity in membrane operations especially in membrane bioreactors (MBR) [30,31].

**Table 5.** Average EC values of NF-RO membrane effluents.

| Membranes | EC (ms/cm) |
|---|---|
| NF90 | 1.77 ± 0.17 |
| NF270 | 3.73 ± 0.27 |
| NF-TM610 | 4.20 ± 0.23 |
| RO-XLE | 0.83 ± 0.47 |
| RO-AD90 | 1.17 ± 0.47 |
| RO-BW30 | 1.13 ± 0.57 |

Average flux values of NF and RO membranes after UF filtration are shown in Figure 5. There was no significant difference in each NF/RO membrane, in terms of fluxes at the same pressure of different UF filtrates ($p < 0.05$). The highest average flux values were obtained in NF270 and TM610 (data not shown) membranes, while NF90 and AD90 membranes could not provide sufficient fluxes as low as 5 LMH in most of the pressure values studied. The BW30 membrane was also one of the poorest performing membranes with flux values down to 10 LMH.

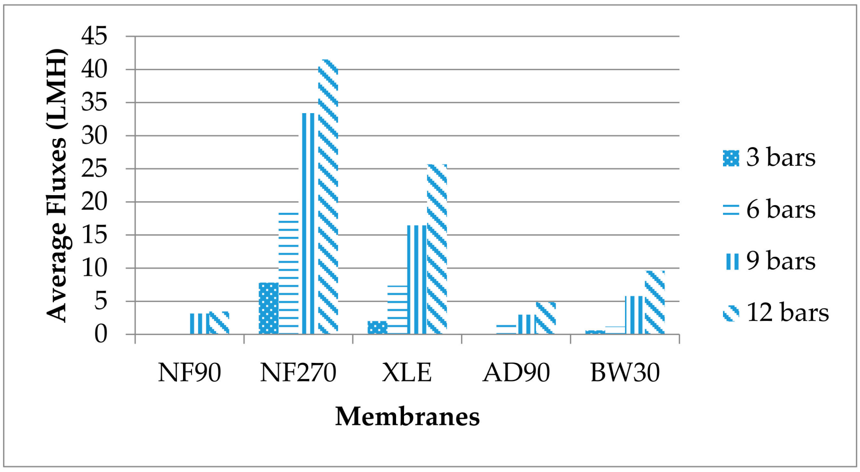


**Figure 5.** Average flux values of different NF-RO membranes at different pressures.

A textile effluent treated with NF (NF90) and RO (BW30) membranes showed that color, biochemical oxygen demand (BOD), and chemical oxygen demand (COD) could be effectively brought to desirable levels by both of the membranes. However, COD was better removed in NF membranes than the RO membranes while it was vice versa in conductivity [32]. Different NF filtration options for water reuse were evaluated in several papers [33] with fluxes varying from 23 to 32 LMH. However, effluent EC values were not reduced in each NF membrane studied. In our study, similar results were observed for only NF270 membrane with little conductivity reduction and high fluxes. It was reported in a review study [10] that salts were effectively removed by applying RO to MBR process.

These results showed that, for water reuse, not only was the flux data important but good water quality was necessary if the effluent was expected to be used as irrigation water with low conductivity according to Turkish irrigation water standards.

## 4. Conclusions

Evaluation of the effluent treatment of an organized industrial zone (OIZ) wastewater treatment plant (WWTP) with UF and NF/RO membranes showed that UF 250 membrane did not perform better than the other UF membranes when it was further filtrated with XLE (RO) membrane. Only XLE membrane showed conductivity less than 1.0 ms/cm in all the filtration experiments. The highest water quality was observed at 12 bars pressure and 26.1 LMH flux value with these membranes. SEM-EDS analysis showed some metal precipitates and high organic fouling in PES membranes. UF filtration could only be used with large pore sizes and hydrophobic membranes, UF 250 kDa PVDF membrane. CLSM images, and fluxes of this membrane showed that there was little biofouling observed over the surface of the membrane. However, bacterial foulants may be adsorbed on the precipitates which were visualized in cross sectional CLSM images and observed in SEM images.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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