Desalination and Water Treatment
www.deswater.com
doi: 10.1080/19443994.2013.855884

53 (2015) 1254–1262
February

Taylor & Francis
Taylor & Francis Group

The filtration performance and efficiency of olive mill wastewater treatment by integrated membrane process

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Received 10 April 2013; Accepted 20 September 2013

ABSTRACT

The effect of operating pressure on olive oil mill wastewater (OMW) purification and filtration efficiency was determined by the evaluation of the flux decline during volume reduction factor (VRF) experiment. One of the other effective factors is the rejection coefficients based on several parameters, which measure the global pollutant content of the OMW, namely chemical oxygen demand, UV absorbance at 254 nm, total phenols, color as well as conductivity. The integrated membrane system (UF-NF) was used in this study and OMW was pre-filtered by three steps tubular microfiltration modules with nominal pore size of 50, 5, and 0.2 micron in series mode and afterward a 35 kDa ultrafiltration membrane. Filtration experiments in concentration mode of the filtration (with recycling of the retentate stream) were performed in laboratory scale, by using three nanofiltration (NF) membranes. The fouling behavior analysis of the NF membranes was also performed by assessment of the flux recovery ratio and degree of the total flux loss (R_t) during VRF experiments. The NF-270 had resulted higher permeate flux than other examined NF membranes, while both NF-90 and self-made NF rejection efficiencies were better than NF-270. On the other hand, the NF-90 had shown better results in comparison with the other NF membranes at high operating pressure. It was concluded that the increasing of the operating pressure enhanced the effects of fouling.

Keywords: Olive oil mill wastewater (OMW) treatment; Integrated membrane system; VRF experiments; COD removal; Total phenols rejection

1. Introduction

Olive oil mill wastewater (OMW), as a byproduct of olive oil production, is becoming a severe environmental hazard, especially in Mediterranean regions, due to its high organic chemical oxygen demand (COD), phytotoxic properties, and resistance to biodegradation. The composition of each type of OMW is different than the others and it depends on a wide range of parameters such as climatic, cultivation parameters, and milling method applied for oil extraction technology [1–3]. Based on literature, COD of OMWs range from 35 to 200 g/L, biochemical oxygen demand from 15 to 135 g/L, suspended solids (SS) from 6 to 69 g/L, total phenols from 2 to 15 g/L, while pH range from 4.5 to 5.8 [4–8]. This is one of the

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highest organic loads in known concentrated wastes, which is 100–200 times greater than domestic wastewater. In case of disposing into the environment, OMW produce can cause serious deteriorations such as coloring of natural waters, serious threats to the aquatic life, pollution in surface, and ground waters, alterations in soil quality, phytotoxicity, and odor nuisance [9].

In previous works, different kinds of wastewater management methods have been used for OMW purification, applied either alone or in combination with other techniques. For instance, OMW disposal to uncultivated and agricultural soils, lagooning or natural evaporation, and thermal concentration, treatment with lime and clay, physical–chemical treatment [10–13], electrocoagulation process [14–16], Fenton and Electro Fenton processes [3,17] have been reported and in several cases practiced. It must be noted that the complication, capital and operating costs, and the processes efficiency might be significant parameters that can affect the process performance in general. Membrane processes including microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) have been previously used for OMW purification [2,18–20]. Membrane processes are developing as the leading contenders for OMW purification and for the recovery and concentrate products from the OMW, or as a pretreatment step before the effluents are discharged.

If OMWs are not mixed with other wastewaters but collected and stored separately, it is possible to recover some valuable solutions such ad phenolic compounds by membrane processes.

Low installation cost and operation of membranes favored the use of membrane processes. MF and UF are used mainly for primary treatment purposes while NF and RO are used for final treatment [18]. NF has tremendous separation abilities to remove both organic and inorganic compounds including dissolved inorganic compounds, bivalent ions (multivalent ions), natural organic matter, and micropollutants. The main advantages of NF process are lower operating pressures than RO, subsequently low investment and maintenance costs, high permeate flux, and sensible efficiency are achieved through this system [21]. However, membrane fouling is one of the common problem associated with OMW purification and also other membrane filtration processes that strongly reduces the permeate flux and subsequently changes both membrane selectivity and efficiency. Thus, a pretreatment step is necessary to decrease membrane fouling and increase the filtration efficiency. On the other hand, operational conditions and the most important of them operating pressure have a tremendous effect on NF membranes performance, efficiency, and fouling.

In this study, different varieties of NF membranes with different properties are used for OMW purification. The membranes performances as functions of transmembrane pressure (TMP) are investigated by measuring the permeate flux, rejection, and antifouling properties during OMW purification. Since the comparison between membranes with different properties (e.g. permeability, molecular weight cut-off (MWCO), surface roughness, surface charge, and hydrophobicity/hydrophilicity) was not yet performed, thus the influence of different parameters on the performance of membranes as function of pressure is investigated herein. The key membrane filtration factors that are affecting the process performance and efficiency are right selection of the membrane type and operating conditions. Depending on the goal that whether a high degree of purification (high degree of COD removal) is needed, or recovery of specific elements is target, the membrane selection and applied operating pressure might vary. Finally, the selection of NF membranes at different TMP and choosing suitable membrane and operating pressure for the purification of OMW in order to achieve higher process performance and efficiency will be carried out.

2. Materials and methods

2.1. OMV sample

The raw OMW used in this work was obtained from olive mill located in Gorgan, Golestan province, Iran. For this study, COD, $UV_{254\,nm}$, color, total phenols, and conductivity were measured. Table 1 shows a list of main characterizations of the raw OMW.

NaOH solution was used for adjusting pH of the raw OMW manually to eight. Afterward, OMWs were pre-filtered by tubular MF modules to remove large particles, SS, oil, and grease. Three tubular MF modules made of polypropylene with nominal pore size of 50, 5, and 0.2 μm were used in series mode,

Table 1
Composition of olive oil mill wastewater

Parameter	Unit	Value
COD	g/L	57 ± 1
Suspended solids	g/L	15 ± 2
Oil and Grease	g/L	10 ± 2
Total phenols	g/L	6.7 ± 0.2
Conductivity	mS/m	12.8
pH	–	4.6 ± 0.1

respectively. The MF pretreatment stage was operated at pressure of 0.2 bar and at temperature of 28˚C. At the end of each run, the system was stopped and the MF membranes were cleaned by pure water at 40˚C.

2.2. Experimental equipment and membranes

The integrated membrane system (UF/NF) was constructed and used during this study. The 35 kDa UF membrane was used for additional pretreatment of microfiltered OMW. Three NF membranes (NF-270, NF-90, and self-made) used and compared in this study. PES UF flat sheet membrane as UF membrane was prepared by phase inversion via immersion precipitation technique [22,23]. The composite polyamide self made NF membrane was prepared by interfacial polymerization of m-PDA in aqueous phase and TMC in organic phase. For the preparation of polyamide composite NF membrane, the UF membrane was used as support layer. The homemade automatic machines used to fabricate the both UF and NF self-made membranes, based on our previous work [24]. Therefore, the membranes with uniform thickness and structure were formed.

All mentioned membranes were as flat sheet and spiraled manually. The main characteristics of the NF membranes are listed in Table 2.

The effect of pressure on performance of the NF membranes was investigated at 28˚C and TMP ranges of 5–20 bar. These experiments were performed by a homemade cross-flow filtration system in concentration mode of filtration, where permeates were collected in a separate vessel and concentrates were circulated back to the feed tank. The membrane containers were used for testing spiral wound membranes. High-pressure pumps were used for passing the feed solution across the membranes modules. Before and after each run, a test using pure water as feed was done to determine permeability and antifouling attributes of the membrane. In the first of all experiments, the system was kept for 20 min at experimental conditions for stabilization.

As the feed solution was reduced continuously in the concentration mode experiments, the feed quality continuously degraded. So, volume reduction factor (VRF) is defined by [25]:

$$\mathrm{VRF} = \frac{V_F}{V_R} \tag{1}$$

where V_F and V_R are the initial volume of the feed and the retention volume ($V_R = V_F - V_P$), respectively. This is an important parameter in concentration operating mode.

The resistance appearing during the filtration can be quantified fouling and cleaning can be specified by the removal of this resistance. The resistance is due to the formation of a cake or gel layer on the membrane surface. In order to evaluate the fouling resistant ability of membranes, flux recovery ratio (FRR) was introduced and calculated as follows:

$$\mathrm{FRR}\ (\%) = \left(\frac{J_{w1}}{J_{w0}}\right) \times 100 \tag{2}$$

where J_{w0} and J_{w1} are the pure water flux of virgin and fouled membrane, respectively. When a concentration test ended, the filtration system was flushed with pure water at 40˚C for 30 min or until the rinsing water came out clear, and J_{w1} (L/m^2 h) was measured to evaluate the degree of irreversible fouling. To analyze the fouling process in details, several equations were used to describe the fouling resistance of the membranes [26]. The total fouling ratio (R_t) was defined and calculated as following:

$$R_t = \left(1 - \frac{J_v}{J_{w0}}\right) \times 100 \tag{3}$$

Here, J_v is flux of OMW in steady state condition and R_t is the degree of total flux loss caused by total fouling. Reversible fouling ratio (R_r) and irreversible fouling ratio (R_{ir}) were also defined and calculated by following equations, respectively.

$$R_r = \left(\frac{J_{w1} - J_v}{J_{w0}}\right) \times 100 \tag{4}$$

Table 2
Properties of the NF membranes used

Membrane	Manufacturer	Permeability (L/m^2 h bar)	NaCl rejection (%)	MWCO (Da)	Contact angle (˚)	Roughness (nm)
NF-270	Dow-Filmtec	14.6	35	200–250	29.1 ± 0.5	9.0 ± 2
NF-90	Dow-Filmtec	8.1	84	150–200	65.6 ± 0.5	112 ± 2
NF-self	Self made	5.7	73	450	51.3 ± 0.5	67 ± 2

$$R_{ir} = \left(\frac{J_{w0} - J_{w1}}{J_{w0}}\right) \times 100 \quad (5)$$

Obviously, R_t was the sum of R_r and R_{ir}.

Separation performance of the membrane is evaluated by the percent rejection (%) of feed components, which is calculated as:

$$R(\%) = \left(1 - \frac{C_p}{C_f}\right) \times 100 \quad (6)$$

where C_p and C_f represent the concentration of total phenols in permeate and feed solutions, respectively.

2.3. Analytical methods

The levels of COD were measured using spectrophotometer model AL250 AQUALYTIC Germany. The natural organic matters and color rejections were measured as the absorbance values at 254 and 395 nm by spectrophotometer, respectively. Total phenols were estimated by using the Folin–Ciocalteu method [27]. Conductivity of samples was analyzed by digital conductivity meter of Hanna (model: HI 8733, Padova, Italy). MWCO of membranes was obtained from the separation data obtained by filtration of polyethylene glycol with various molecular weights and polyethylene oxide with molecular weight of 100 kDa [28].

3. Results and discussion

3.1. Pretreatment results

The MF stage was performed by three different types of MF membranes with nominal pore sizes of 50, 5, and 0.2 micron in series mode. The initial COD of wastewater, SS and oil, and grease were reduced about 67, 99, as well as 94% by MF pretreatment stage, respectively. Afterward, the UF-(self made) was used for further treatment as pretreatment of NF stage. Based on MF stage permeate, the COD removal of UF pretreatment stage was 44.3%.

3.2. Effect of pressure on permeates flux and flux decline

In this stage, the UF permeate was used as feed for NF filtration experiments. The filtration experiments of the OMW were performed with the three selected NF membranes in filtration concentration mode. The permeate flux was measured as a function of TMP and VRF.

Fig. 1 represents the evolution of OMW flux (J_v) with VRF and TMP for the NF membranes, a clear decline of J_v occurs with the increase in VRF for all NF membranes, due to the concentration polarization and fouling effect on the membrane.

A slightly decrease in flux was observed at especially 5 and 10 bar, but at 15 and 20 bar, the flux decline was more rapid. As clearly seen from Fig. 1, the flux decline due to concentration polarization and fouling was increased with increasing pressure.

These curves may be divided into three steps: (1) OMW flux decreased quickly at the first step of filtration, (2) at the second step, it had a slighter decline in which nearby VRF = 1.5, and (3) at the last

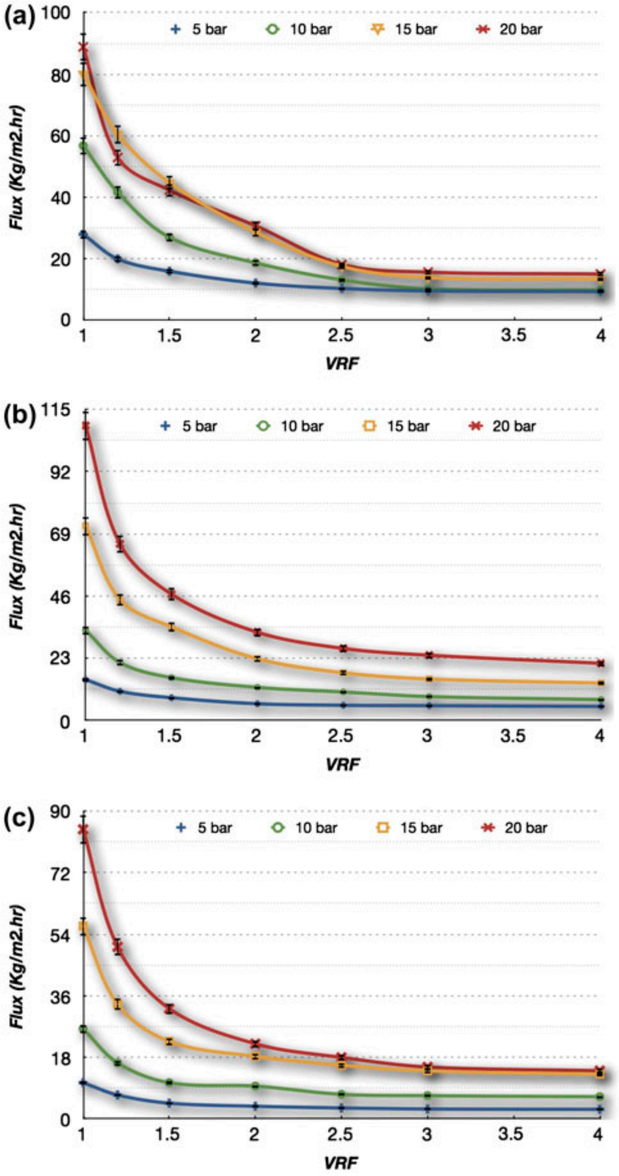

Fig. 1. The evolution of OMW flux (J_v) with VRF and TMP, (a) NF-270, (b) NF-90, and (c) NF-(self made), ($T = 28$°C).

step, a very slight reduction in permeate flux up to steady-state conditions happened after VRF = 2.5. Similar trends have also been seen in earlier studies and reported by other researchers as well [25,29]. It seems that the effect of pressure on the first step is higher than the other steps. It is known that as pressure increases, concentration polarization become bigger and the components on the membrane surface increase [30].

It also can be seen from Fig. 1 that increasing pressure augmented the effects of fouling. In the case of NF-270 (Fig. 1(a)), at 5 bar pressure, permeate flux had 63% reduction to steady state in VRF = 2.5, while it has shown 80% reduction at 20 bar pressure. In the case of NF-90 (Fig. 1(b)), in VRF = 2.5, reduction of permeate flux was 62 and 76% at 5 and 20 bar pressure, respectively. The same trend was observed for the NF-(self-made). Thus it can be concluded that with increasing pressure although initial permeate flux was improved dramatically, the flux decline also increased and there are no considerable difference between permeates flux of different operating pressure in steady state condition of flux (VRF = 2.5 onwards).

It is known that flux decline can be caused by various factors such as concentration polarization, gel layer formation, and pore blocking by OMW components. All these factors produce extra resistances on the feed side to transport through the membrane [31].

At the first step, flux decline was affected by concentration polarization, which results in a rapid flux decline. The cake formation step that happened after concentration polarization resulted in a smaller flux decline than the previous step.

The initial permeate flux and flux decline were different during OMW purification for different membranes and pressure. The NF-270 resulted in a higher initial flux than both other NF membranes in pressures of 5, 10, and 15 bar. However, this difference was reduced with increasing pressure, due to higher concentration polarization and fouling, and ultimately at 20 bar pressure the NF-90 had shown higher permeate flux than NF-270. This difference in initial permeates flux and flux decline might be related to the membrane properties including permeability, MWCO, as well as hydrophilicity.

Membranes with higher permeability and MWCO are expected to show a higher permeate flux at beginning of filtration, and on the other hand, higher flux decline, due to higher concentration polarization, fouling, and pore blocking by OWM components.

Membrane surface hydrophilicity is an important property which affects upon the behavior of permeates flux and flux decline. The large amounts of free water are absorbed by highly hydrophilic groups of membranes surface to form a thin water film, which prevents the deposition of OMW components on the membrane surface or pore walls [32,33].

As it can be seen from Table 2, the NF-270 in terms of hydrophilicity and permeability has considerable difference than both other NF membranes. Higher hydrophilicity and permeability of NF-270 may result in a lower and higher flux decline than the other NF membranes, respectively. However, it was observed that flux decline of NF-270 than NF-90 was slightly higher at all operating pressure tested, and it might be due to the negative effect of higher permeability on fouling. Moreover, the flux decline of NF-(self made) during VRF experiment found to be significantly higher than both other NF membranes. However, it had shown slightly lower flux decline at 20 bar pressure than NF-270. Therefore, it is safe to say that the permeability is an important property of NF membranes that significantly affects the flux decline. Especially at high operating pressure its effects are more visible.

According to the taken results, it was seen from VRF = 2.5 onwards, there were no significant changes in permeate flux of all the membranes and studied pressure. Thus, this VRF value was measured as start of steady-state conditions of flux for this study. In addition, this point is taken that operating pressure had no significant effect during purification in steady-state condition of filtration.

3.3. Pressure effects on membrane rejection efficiency

The efficiency of membrane filtration processes for the removal of OMW pollutants was estimated based on rejection measurements. It was formerly explained, the selected pollution indices in the present study were: COD, absorbance at $UV_{254\,nm}$ (UV absorbance at 254 nm is attributed mainly to the natural organic matter), color, total phenols, and conductivity (conductivity is attributed mainly to the inorganic salts). The rejection coefficients of pollution indices by all the membranes were determined as a function of TMP and the values taken are shown in Fig. 2.

From Fig. 2(a), the NF-270 has presented different removal efficiency for different pollution indices, it can be seen that at all pressure studied conductivity rejection and COD removal of this membrane were lower than the other. On the other hand, both NF-90 and NF-(self made) showed high removal efficiency for all pollution indices studied by more than 90%. From Fig. 2, it is observed that by increasing pressure, the rejection coefficients of pollution indices were improved for all membranes used. These improvements were varying for different

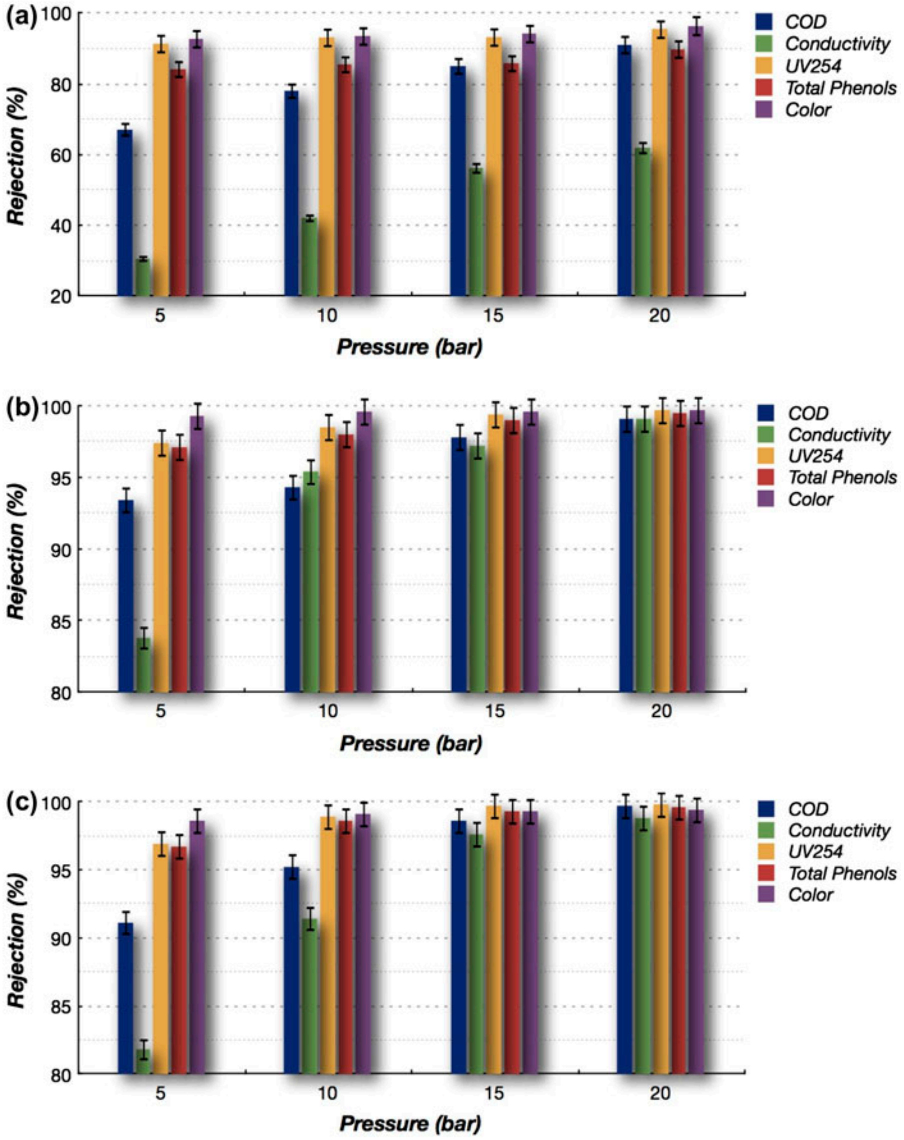

Fig. 2. Determined of pollution indices rejection coefficients as a function of TMP, (a) NF-270, (b) NF-90, and (c) NF-(self made), ($T = 28$°C).

pollution indices and membranes. In general, permeate concentrations of components decrease when the pressure is increased. However, during the formation of a concentration polarization layer on the membrane surface, higher permeate concentrations are obtained. As can be seen, the conductivity rejection and COD removal were improved, due to increasing pressure. On the other hand, pressure effect on the other pollution indices was lower, duo to higher size of these contaminations.

In general, the size exclusion is dominant mechanism for the rejection of OMW large components such as organic compounds. The rejection will generally increase with the molecular size. Therefore, the lower salt rejection can be attributed to this fact that salt compounds that cause conductivity are considerably smaller than organic compounds of OMW.

The NF-90 COD removal at 5 and 20 bar were about 93.4% (COD = 690 ± 10 mg/L) and 99.1% (COD = 100 ± 10 mg/L), respectively. Therefore, it can be concluded that TMP has different effect on different pollutant indices, and its effects are higher for lower size contaminations. Based on the goal for concentration and purification specific components, it might be used different TMP to achieve the purpose.

To the best of our knowledge, a few researchers for OMW purification have used membrane processes up to now. Therefore, there are not lots of results for comparison study. Coskun and coworkers [18] were used different kinds of NF and RO membranes for OWM purification. They had presented final COD of 11,200 mg/L when NF-270 was used after pretreatment by UF filtration. In addition, the best results of permeate COD that they reported was 1,000 mg/L, when RO membrane was applied at 25 bar pressure. Abbassi and coworkers [34] were performed OMW purification by MEUF process and presented 95.7% COD removal (final COD = 5,880 mg/L). Results presented herein demonstrated that our presented membrane process reduces effectively more than 99.8% of initial COD during whole process. These obtained results are higher and more efficient than those previously reported in the purification of OMWs with the other methods.

3.4. Pressure effects on fouling and resistance

As for analysis of the membranes fouling behavior, different parameters namely FRR, degree of the total flux loss (R_t), reversible fouling resistance (R_r), and irreversible fouling resistance (R_{ir}) were presented in Table 3. The R_r is due to concentration polarization and deposition of solids (cake layer) on the membrane surface, and therefore, it can be removed by cleaning with water after the wastewater (OMW) filtration process. On the contrary, the R_{ir} is due to pore blocking and adsorption of materials onto the membrane surface and pores, which cannot be removed by water cleaning [35]. The mentioned resistances were determined from permeate flux data obtained in the filtration experiments using Eqs. (3–5). As can be seen from Table 3. It can be concluded that the UF-(self made) had higher FRR and reversible fouling than UF-CSM, due to the higher surface hydrophilicity and lower MWCO.

The NF-90 had the highest FRR among the NF membranes. In addition, the reversible fouling (R_r) of NF-90 was higher than both other NF membranes, and on the contrary, the irreversible fouling of NF-90 (R_{ir}) was significantly lower. These show that the concentration polarization and cake layer provided a higher contribution of fouling than the adsorbed particles in/on membrane. The R_{ir} of NF-(self made) was significantly higher in comparison with NF-270 and NF-90. Higher R_{ir} and lower FRR of NF-(self made) can be attributed to the pore blocking and adsorption of OMW components onto the membrane surface and pores wall. It might be due to the higher MWCO of NF-(self made) than NF-270 and NF-90.

The reversible fouling (R_r) and FRR of all the NF membranes were decreased by increasing pressure, on the contrary irreversible fouling (R_{ir}) and total flux loss (R_t) were increased. Reduction of FRR and reversible fouling, on the other hand, increment of total flux loss and irreversible fouling means that pore blocking and cake enhancement were augmented, duo to higher TMP.

4. Conclusions

Three types of NF membranes were used in OMW treatment in spiral wound mode. First, membranes permeate flux were measured during VRF experiments at different operating pressure (ranges from 5 to 20 bar). By increasing TMP, initial permeate flux was improved dramatically, but flux decline also increased. In addition, it was observed that the effect of pressure was higher at the initial steps of VRF filtration. Between the NF membranes, NF-270 resulted higher permeate flux in comparison with the others at all operating pressures, except 20 bar. In addition, the contributions of both irreversible and reversible fouling were different in the all membranes and operating pressure studies. The higher flux decline and contribution of irreversible fouling of NF-Self attributed to its higher MWCO when it evaluated with the other studied membranes. The VRF = 2.5 was measured as start of steady-state conditions of flux for this study.

Table 3
Fouling analysis of the NF membranes at different operating pressure

	NF-270				NF-90				NF-(self made)			
Pressure (Bar)	R_r (%)	R_{ir} (%)	R_r (%)	FRR (%)	R_r (%)	R_{ir} (%)	R_t (%)	FRR (%)	R_r (%)	R_{ir} (%)	R_r (%)	FRR (%)
5	66.5	18	84.5	82	76.6	5.9	82.6	94.1	34.1	44.8	78.9	55.2
10	62.2	28	90.2	71.9	76.8	7.2	84	92.8	36.5	49.2	85.7	50.8
15	52.2	39.2	91.5	60.8	72.2	11.6	83.8	88.4	20.5	59.5	80.1	40.5
20	40.3	53.4	93.7	46.6	68.4	13.1	81.4	87	18.4	65.7	84.2	34.3

It is shown that, although the increase in operating pressure can result in improvement of permeate flux, but during VRF experiments its effect was reduced to a point that there were no significant difference between permeate flux of different operating pressure at steady state condition of flux (VRF = 2.5 onwards). In addition, high operating pressure had a negative effect on FRR and irreversible fouling.

The results of rejection coefficient demonstrated that the NF-90 and NF-Self made as tight membranes have shown higher removal efficiency of the pollution indices than NF-270 as a loose membrane. Among NF membranes, the NF-270 and NF-90 were found to be the high flux and the high rejection membrane, respectively, while both mentioned membranes has shown better antifouling property and FRR in comparison with the NF-Self at all operating pressure which was studied.

TMP has indicated different effects on the different pollutant indices, and its effect was important for COD removal efficiency and lower size contaminations.

Depending on the propose that whether it is wanted to achieve a high degree of treatment and recovery of some compounds or the goal is filtration with a lower operating cost, the operating pressure can be adjusted.

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