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A new approach to evaluate membrane performance: COD and EC rejection rates

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ABSTRACT

Olive mill wastewater (OMW), which contains abundant soluble and particulate matter, negatively influences the receiving environment. Membrane processes have the potential to treat OMW. Electrical conductivity (EC), which is very high in OMW, is one of the key factors of membrane processes. The EC value of the OMW sample used in this study was higher than the common values, and this situation resulted in high osmotic pressure. Several samples with different influent EC values were fed to a reverse osmosis (RO) system. The osmotic pressure values for each influent EC were calculated. The relationship between the osmotic pressure and influent EC values was nonlinear. The permeate flux could not be achieved for some applied pressures and influent EC values because of the high osmotic pressure of the feedwater. Moreover, the terms of the chemical oxygen demand (COD) rejection rate and EC rejection rate were developed and observed and predicted values were compared for these terms and permeate fluxes using linear and nonlinear regressions. It was determined that osmotic pressure is a limiting condition in OMW treatment using RO membranes and that COD and EC rejection rates are useful terms to evaluate membrane performance.

Keywords: COD rejection rate; EC rejection rate; Olive mill wastewater (OMW); Osmotic pressure; Reverse osmosis

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1. Introduction

The olive oil industry is one of the most important trades in Mediterranean countries. According to 2014, data from the International Olive Council, 97.2% of olive oil production, and 88.4% of table olive production were achieved by Mediterranean countries, with 5.7–15.5% occurring in Turkey [1]. Olive mill wastewater (OMW), which is harmful for natural water bodies, was produced in high quantities in Turkey. OMW contains abundant solids and organic contents, including phenolic compounds [2–5]. Moreover, olive oil production has to be performed immediately after the harvest of olives, and for this reason, OMW is produced only between October and February [6]. Because OMW contains abundant soluble materials and is a seasonal product, there is no common treatment method for OMW in practice. However, there are several studies on the treatment of OMW and valuable matter recovery from

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it in the literature. In these studies, electrochemical treatment [7–11], biological processes [12–15], oxidation processes [12,16,17], and membrane processes [18–22] were used for the treatment of OMW and valuable matter recovery [23–25].

The reverse osmosis (RO) process is one the most common systems in water and wastewater treatment. The potential of RO to obtain high-quality effluent makes it desirable in water and wastewater treatment, especially as a post-treatment process. Moreover, the recovery of valuable products from wastewater can be achieved with RO. Although RO has many advantages on several counts, there are also some disadvantages. The most important disadvantage of RO is its capital and operation cost. The main operation cost for RO is transmembrane pressure [26]. Transmembrane pressure is required to overcome the osmotic pressure of the feedwater. Therefore, higher osmotic pressure leads to higher operation cost for RO.

Membrane performance is related to removal efficiency, membrane permeate flux, and recovery rate. However, evaluating membrane performance using only one of these parameters is not representative.

Olive oil production facilities are small and medium-sized industries and operate when olives arrive for oil production in Turkey. Because the work is seasonal and the working time is random, membrane processes are appropriate for the treatment of OMW. Moreover, the total operation cost for a whole city can be reduced with a mobile RO facility. However, the critical factor is osmotic pressure because higher osmotic pressure results in higher costs. Another critical factor, therefore, is total dissolved solids because higher total dissolved solids in the feedwater causes higher osmotic pressure.

Electrical conductivity (EC) parameter results from dissolved solid concentration. EC and dissolved solids concentration give similar results in terms of membrane performance [27]. In this study, the effect of dissolved solids concentrations on removal efficiency and membrane flux were investigated in OMW treatment by RO using the EC parameter. The relation between membrane performance and the EC values of the feedwater was examined using linear and nonlinear regression. Moreover, chemical oxygen demand (COD) rejection rate and EC rejection rate parameters were developed to evaluate membrane performance and were tested with the results in this study and previous studies.

2. Material and methods

2.1. Analyses

The OMW used in this study was obtained from a two-phase olive oil production plant in Gemlik, Turkey. The characteristics of the OMW are given in Table 1. pH and EC analysis were made by Hach HQ 40D multiparameter meter and WTW Inolab 7110 pH meter, respectively. Total suspended solids, COD, and oil & grease were measured in accordance with [28].

2.2. Pre-treatment studies

Pre-treatment with centrifugation and ultrafiltration (UF) was applied to the OMW before the RO experiments. The centrifuge process was performed with a Beckman Coulter Allegra X12 for 30 min at 3,750 rpm. A GE Sepa™ CF II membrane cell system was used for the UF and RO processes. The membrane system used in this study is shown in Fig. 1 [29]. The specifications of the UF membrane are shown in Table 2.

2.3. RO experiments

Pre-treated OMW was passed through the RO membrane with difficulty. Therefore, the OMW was gradually diluted because the effect of the osmotic pressure on the performance of the RO process is significant. RO experiments were performed using six different feedwater conductivity values (5, 9.8, 15, 19.3, 23.9, and 29.4 mS cm^{-1}). The applied pressures were 5, 10, 15, 20, 25, and 30 bar for each of the influent conductivity values. The membrane fluxes were measured by Kern-Precision Balance 440. The specifications of the RO membrane are shown in Table 2. The performance of RO was evaluated by COD removal efficiency, decrease of EC, and membrane flux.

2.4. COD rejection rate and EC rejection rate

Membrane performance is generally determined by removal efficiency and membrane flux. Higher removal efficiencies and/or higher membrane fluxes indicate higher membrane performance. However,

Table 1
The characteristics of the OMW in this study

	Raw OMW	Pre-treated OMW
pH	4.2	4.4
Conductivity (mS cm^{-1})	75.0	74.0
TSS (g L^{-1})	17.3	na
COD (g L^{-1})	54.4	19.7
Oil & grease (g L^{-1})	15.6	na

Note: na: not analyzed.

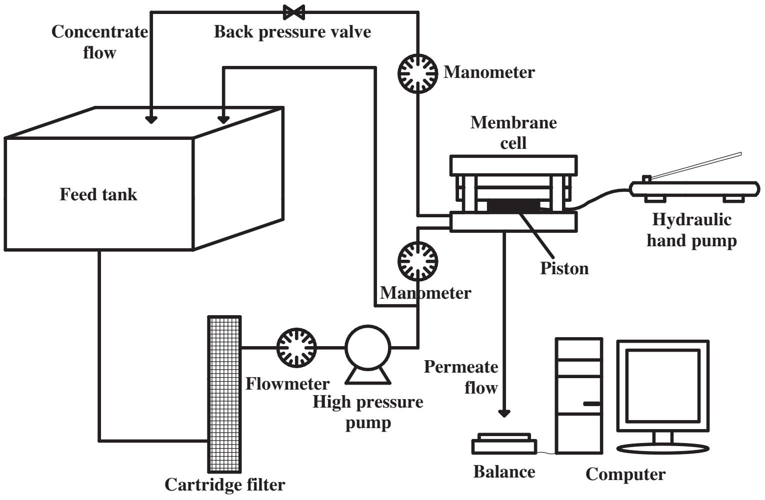

Fig. 1. Schematic diagram of the membrane process [29].

Table 2
The specifications of the UF and RO membranes

Membrane	Type	Manufacturer	Material	MWCO (kDa)	NaCl rejection (%)	pH range	MOP[a] (bar)	MOT (℃)
JW	UF	GE osmonics	PVDF	30	–	1–11	7	50
AG	RO	GE osmonics	Polyamide	–	99.5	1–11	41	50

[a] MOP: maximum operating pressure.

often, when membrane fluxes are high, removal efficiencies are low, and high removal efficiencies with low membrane fluxes can be obtained. Moreover, the feedwater stream separates into two streams: permeate and concentrate. Higher permeate stream/feed stream ratios (called recovery rate) are the desired situation. Consequently, removal efficiency, membrane flux, and recovery rate are important parameters to determine membrane performance. COD and EC rejection rates were developed for these reasons. These values are the product of the membrane flux (F_p) and the ratio of the concentration of "concentrate" to the concentration of "feed" (C_c/C_f). The more the membrane rejects pollutants, the more pollutant concentration in the concentrate stream. Similarly, the higher recovery rates cause the higher pollutant concentration of the concentrate stream and this means the higher membrane performance. Therefore, membrane flux, removal efficiency, and recovery rate were together used to evaluate membrane performance in the calculation of COD and EC rejection rate.

COD removal efficiencies and the decrease of EC were calculated by the following equation:

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

The simple equation for the rejection rate is as follows [30]:

$$Q_f = Q_p + Q_c \tag{2}$$

where Q_f is the feed stream flow (kg h^{-1}), Q_p is the permeate stream flow (kg h^{-1}), and Q_c is the concentrate stream flow (kg h^{-1}).

The mass balance for the pollutant is shown in Eq. (3):

$$Q_f\ C_f = Q_p C_p + Q_c C_c \tag{3}$$

where C_f is the pollutant concentration of the feed stream (kg m^{-3}), C_p is the pollutant concentration of the permeate stream (kg m^{-3}), and C_c is the pollutant concentration of the concentrate stream (kg m^{-3}).

The pollutant concentration of the concentrate stream is calculated by the following equation:

$$C_c = \frac{Q_f\ C_f - Q_p C_p}{Q_c} \tag{4}$$

Membrane flux is one of the most important parameters for membrane performance and expresses the flow rate that passes from the membrane per unit surface area:

$$F_p = \frac{Q_p}{A} \tag{5}$$

where F_p is the membrane flux (kg m^{-2} h^{-1}) and A is the membrane surface area (m^2).

The COD rejection rate expresses the COD quantity rejected by the membrane vs. the influent COD concentration of the sample, and it contains three parameters: recovery rate, removal efficiency, and membrane flux. COD rejection rate is calculated by the following equations:

$$R_{\mathrm{COD}} = \frac{C_c F_p}{C_f} = \frac{Q_f C_f - Q_p C_p}{Q_c} \frac{Q_p}{A} \frac{1}{C_f} \tag{6}$$

where R_{COD} is the COD rejection rate (kg m^{-2} h^{-1}), C_f is the COD concentration of the feed stream (g L^{-1}), C_p is the COD concentration of the permeate stream (g L^{-1}), and C_c is the COD concentration of the concentrate stream (g L^{-1}).

Although conductivity is not a pollution parameter, it expresses the dissolved solids of the liquor. Therefore, a rejection rate for the conductivity parameter can also be calculated similar to the COD parameter:

$$R_{\mathrm{Conductivity}} = \frac{C_c F_p}{C_f} = \frac{Q_f C_f - Q_p C_p}{Q_c} \frac{Q_p}{A} \frac{1}{C_f} \tag{7}$$

where $R_{\mathrm{Conductivity}}$ is the conductivity rejection rate (kg m^{-2} h^{-1}), C_f is the EC of the feed stream (mS cm^{-1}), C_p is the EC of the permeate stream (mS cm^{-1}), and C_c is the EC of the concentrate stream (mS cm^{-1}).

2.5. Statistical analysis

The statistical analysis of the study results was performed with MS Excel and Statgraphic software. Multiple linear regression was performed with MS Excel, and multiple nonlinear regression was performed with Statgraphic. The multiple linear equations were obtained in the following form:

$$y = a + bX_1 + cX_2 \tag{8}$$

where X_1 and X_2 are independent variables, a, b, and c are regression coefficients, and y is the dependent variable. X_1 is the influent conductivity and X_2 is the applied pressure in this equation.

The multiple nonlinear equations were obtained in the following form:

$$y = a + bX_1 + cX_2 + dX_1X_2 \tag{9}$$

where X_1 and X_2 are independent variables, a, b, c, and d are regression coefficients, and y is the dependent variable. X_1 is the influent conductivity and X_2 is the applied pressure in this equation.

3. Results and discussion

The characteristics of OMW are related to the specifications of the olives that are used for olive oil production. The specifications of the olives are also related to several factors, such as soil structure and climate. Moreover, the olive oil production process also changes OMW characteristics. Therefore, the characteristics of the OMW studied in the literature are different. The characteristics of some OMW in the literature are shown in Table 3.

Table 3
OMW characteristics in different studies

Region	Process type	COD (g L^{-1})	Conductivity (mS cm^{-1})	Refs.
Marrakech, Morocco	cold pressed	156.0	23.5	[2]
Milas, Turkey	3-phase	44.5	8.7	[7]
Central Italy	–	60.0	5.8	[31]
Milas, Turkey	3-phase	40.3	5.3	[32]
Ayvalik, Turkey	3-phase	54.8	8.2	[33]
Tadmait, Algeria	–	28.0	32.0	[22]
Chania, Greece	3-phase	47.0	17.0	[34]
Jaen, Spain	3-phase	151.4	7.9	[35]
Gemlik, Turkey	2-phase	54.4	75.0	This study

As observed from Table 3, the characteristics of the OMW are different. These differences are more remarkable with regard to dissolved solids. Moreover, a difference in conductivity is observed in samples from the same region. Whereas the COD concentrations of two OMW samples obtained at different times from Milas, Turkey are similar, the conductivities of these samples are different.

The EC of the pre-treated OMW is 74 mS cm^{-1}. This OMW sample has high osmotic pressure, which is also related to the maximum operating pressure of the membrane system. Therefore, the sample was gradually diluted to determine the effect of different influent conductivity values. Consequently, the COD concentrations of the feed samples were reduced according to the dilution factors. The influent conductivity and COD values of the feed samples are shown in Table 4.

Six different influent samples were fed to the RO unit at 5, 10, 15, 20, 25, and 30 bar. The membrane flux values obtained in the RO unit for each applied pressure and influent EC value are shown in Fig. 2. The permeate stream for applied pressures of 5, 10, and 15 bar was not obtained with influent EC values of 23.9–29.4 mS cm^{-1}. With these influent EC values, the membrane fluxes were obtained only for pressures of 20, 25, and 30 bar. A permeate stream cannot be obtained for applied pressures of 5–10 bar with an influent EC value of 19.3 mS cm^{-1} or for the applied pressure of 5 bar with an influent EC value of 15 mS cm^{-1}. A permeate stream for all applied pressures can be achieved with influent EC values of 5.0–9.8 mS cm^{-1}. The membrane fluxes increased linearly with the applied pressure for each influent EC value, especially with lower applied pressure. Nonlinear dependence appeared as the applied pressure increased, similar to [36].

Table 4
Influent conductivity and COD values for the RO process

Sample no.	EC (mS cm^{-1})	COD (g L^{-1})
1	5.0	1.10
2	9.8	2.45
3	15.0	3.45
4	19.3	4.51
5	23.9	5.39
6	29.4	6.79

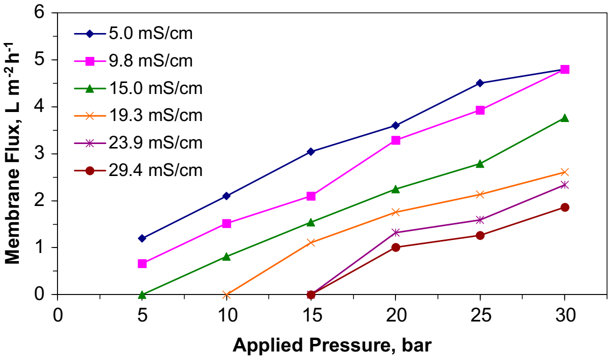

Fig. 2. Membrane fluxes vs. influent EC values and applied pressures.

COD removal efficiencies and decreases in the EC were calculated according to the influent sample to the RO unit and in the permeate stream. However, these parameters cannot be calculated for influent EC values in which a permeate stream cannot be obtained. The COD removal efficiencies and decreases in the EC are shown in Fig. 3. Both the COD removal efficiencies and decreases in EC increased with increased applied pressure. All COD removal efficiencies were higher than 90%, and most of the decreases in EC were higher than 90%. As the influent EC values changed, the removal efficiencies for different influent EC values and the same applied pressure were similar.

After the removal efficiencies and membrane fluxes were determined, linear and nonlinear regression analyses were completed for membrane flux, COD

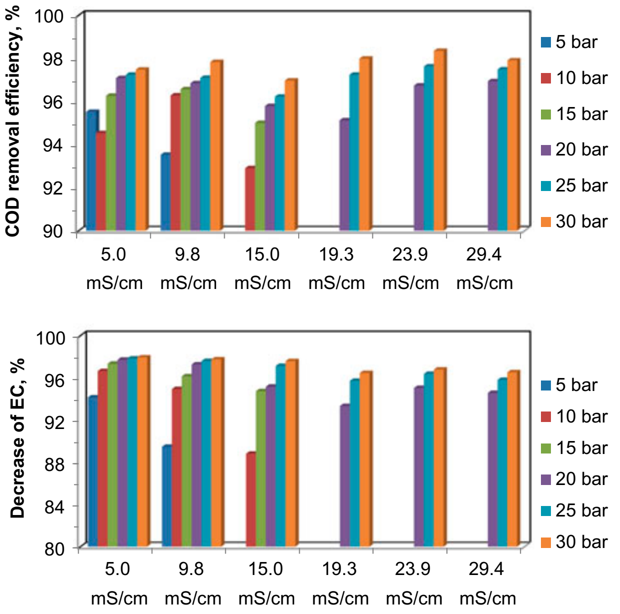

Fig. 3. COD removal efficiencies and decreases in EC.

rejection rate, and EC rejection rate vs. influent EC and applied pressure. Regression coefficients *a*, *b*, *c*, and *d* were calculated with linear and nonlinear regression for each parameter. The values of the coefficients are shown in Table 5. The membrane flux, COD rejection rate, and EC rejection rate values were calculated using these coefficients for linear and nonlinear regression. The graphs for the observed and predicted values were drawn for these three parameters with the *R*-squared values (Fig. 4). In all regressions, because the *P*-value for the effect of the influent EC and the applied pressure was less than 0.05, the effect of these parameters was significantly different from zero at the 95% confidence level. The nonlinear regression obtained better results than the linear regression for each parameter. Reliable and similar regression results were obtained for the three parameters. The *R*-squared values for the three parameters vs. influent EC and applied pressure were approximately 0.95–0.96 for both the linear and nonlinear regressions. This phenomenon demonstrated that the COD rejection rate and the EC rejection rate are useful parameters for evaluating membrane performance.

The COD and EC rejection rates decreased at higher influent EC values. The strength of this decrease diminished gradually with increasing influent EC. This phenomenon demonstrated that the RO process for influent EC values above a critical point was not feasible and that the practicability of the RO process increased with decreasing influent EC. In contrast, the COD and EC rejection rates increased with higher applied pressure. However, the strength of this increase diminished gradually with increasing influent EC. This situation indicates that the practicability of the RO process increases with increasing applied pressure until a critical value and remains stable after this value. The maximum values of both the COD and EC rejection rates were obtained as approximately 9.5 kg m^{-2} h^{-1} with an applied pressure of 30 bar and an influent EC value of 5.04 mS cm^{-1}. The COD and EC rejection rates for an applied pressure of 10 bar and an influent EC of 5.04 mS cm^{-1} were 4.09–4.13 kg m^{-2} h^{-1}, respectively.

In another study, the unit cost of poultry slaughterhouse wastewater treatment was investigated with a nanofiltration (NF) membrane (DK from GE Osmonics) and an RO membrane (AG from GE Osmonics) with and without UF pre-treatment [37]. Lower unit costs were obtained when using UF pre-treatment and the unit cost for the UF + RO process was slightly higher than the UF + NF process. The same results were obtained for the COD and EC rejection rates. These rates increased when using UF pre-treatment. For example, the COD and EC rejection rates increased to approximately 64 kg m^{-2} h^{-1} from 39 kg m^{-2} h^{-1} when UF was used as a pre-treatment for a pressure of 10 bar and an influent EC of 3.6 mS cm^{-1}. COD rejection rate increased to approximately 60 kg m^{-2} h^{-1} from 57 kg m^{-2} h^{-1} and EC rejection rate increased to approximately 46 kg m^{-2} h^{-1} from 41 kg m^{-2} h^{-1} for the NF membrane in the same situation. The difference between the COD and EC rejection rates for the NF membrane resulted from the removal efficiencies of the NF membrane being high for COD and low for EC.

The COD and rejection rates obtained in this study were considerably lower than the values in the literature. For example, the COD and EC rejection rates were 4.09–4.13 kg m^{-2} h^{-1} with an applied pressure of 10 bar and an influent EC of 5.04 mS cm^{-1}. These rates were approximately 64 kg m^{-2} h^{-1} with a pressure of 10 bar and an influent EC of 3.6 mS cm^{-1} [37]. Approximately 15-fold COD and EC rejection rates were obtained with the same membrane (AG) and as opposed to a difference of only 1.4-fold of influent EC. This difference probably resulted from the OMW characteristics. Although different dissolved solids can yield the same influent EC, they might have different effects on membrane flux, due to concentration polarization. Different solutes have different effect in

Table 5
Regression coefficients for the linear and nonlinear regressions

Parameter	Regression type	Regression coefficients			
		a	*b*	*c*	*d*
Membrane flux	Linear	1.457	−0.111	0.126	na
	Nonlinear	0.468	−0.053	0.183	−0.003
COD rejection rate	Linear	2.826	−0.217	0.250	na
	Nonlinear	0.839	−0.101	0.363	−0.007
EC rejection rate	Linear	2.858	−0.219	0.249	na
	Nonlinear	0.994	−0.115	0.350	−0.006

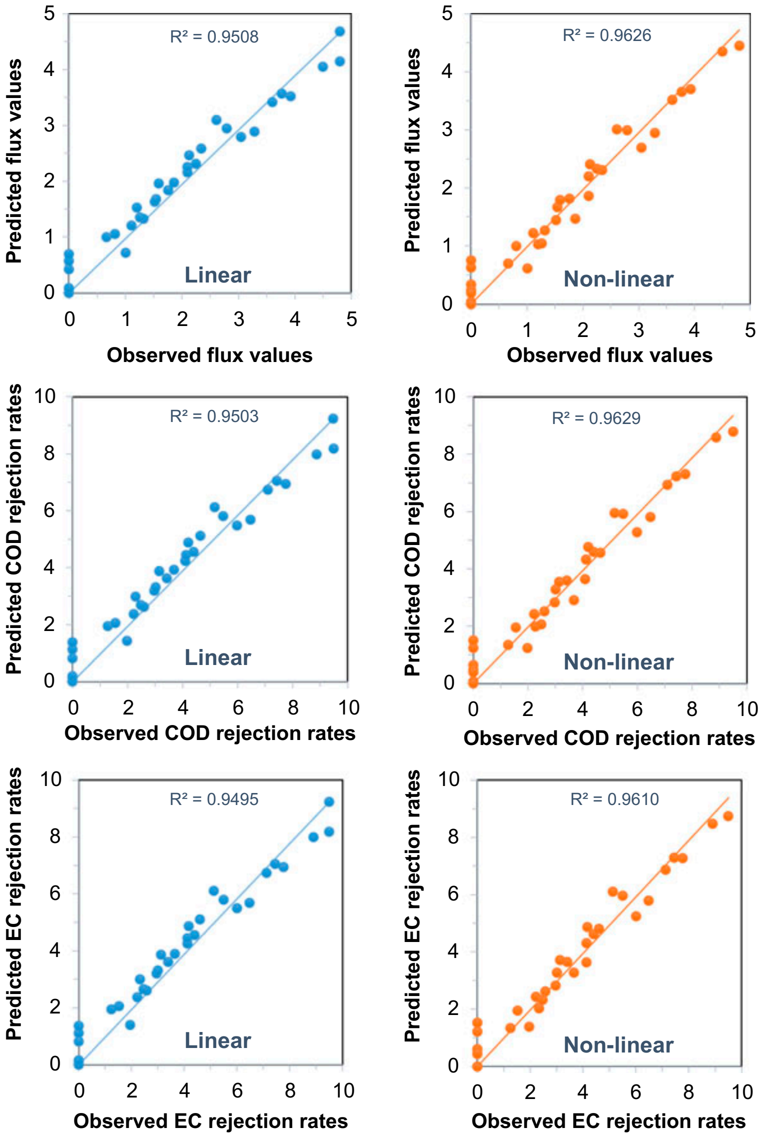

Fig. 4. Predicted/observed values for membrane fluxes, COD rejection rates, and EC rejection rates according to linear and nonlinear regressions (blue circles are linear regression and orange circles are nonlinear regression).

concentration polarization [38]. This situation causes a difference in membrane fluxes. Oil content of olive mill wastewaters is higher than the most wastewater type. With same transmembrane pressure, the higher oil content causes lower membrane flux [39]. Another study which contains only this subject should be made to determine the effect of different solute on osmotic pressure in future studies.

Osmotic pressure was the key factor for all parameters. Therefore, osmotic pressure values were estimated by extending the linear portion of the flux curves to the *x*-axis, for each influent EC using a regression function in Excel similar to [40]. The estimated osmotic pressure for each influent EC value is shown in Fig. 5. The osmotic pressure increased non-linearly as the influent EC increased, similar to [41]. According to this graph, the EC value of 20 mS cm^{-1} caused an osmotic pressure of 7.3 bar, similar to [40]. When Figs. 2 and 5 are examined together, it can be observed that a permeate stream was not obtained with applied pressure lower than the osmotic pressure for a given EC value. Moreover, a permeate stream was not obtained for an applied pressure higher than, but close to, the osmotic pressure.

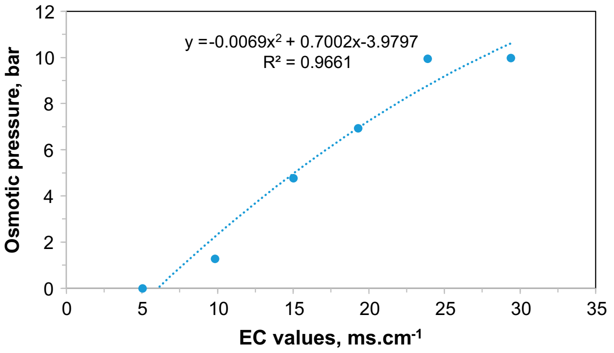

Fig. 5. Calculated osmotic pressures vs. influent EC values.

For example, the osmotic pressure was approximately 7.0 bar for the EC value of 19.3 mS cm^{-1}. At this osmotic pressure, a permeate stream was not obtained at 5–10 bar.

4. Conclusions

This study focused the effect of osmotic pressure on RO performance. COD and EC rejection rates were developed to evaluate membrane performance. The performance of the membrane was evaluated using these parameters, the removal efficiencies, and membrane fluxes. The COD removal efficiencies and EC decrease were approximately 98% in this study. The membrane fluxes increased when the applied pressure increased and the influent EC decreased. COD and rejection rates were calculated with the obtained results, and the linear and nonlinear models could predict these terms. Predicted and observed graphs were drawn for these terms and the membrane flux. All predicted values were well-matched with the observed values, which showed that COD and rejection rates are useful parameters for evaluating membrane performance. All parameters showed that osmotic pressure is very critical factor on membrane processes. RO can be used as a mobile solution for the treatment of OMW, as long as the osmotic pressure is considered. The total dissolved solids content and osmotic pressure of OMWs should be examined zone-by-zone and for all seasons. Moreover, the dissolved solids content of OMW can be diluted with other wastewater, such as municipal wastewater. For this reason, studies on membrane performance and membrane fouling while feeding this combined wastewater should be performed. COD and EC rejection rates should be determined to evaluate membrane performance in future studies.

Abbreviation list

A	—	membrane surface area (m^2).
C_c	—	pollutant concentration of the concentrate stream (kg m^{-3} for COD and mS cm^{-1} for EC)
C_f	—	pollutant concentration of the feed stream (kg m^{-3} for COD and mS cm^{-1} for EC)
C_p	—	pollutant concentration of the permeate stream (kg m^{-3} for COD and mS cm^{-1} for EC)
COD	—	chemical oxygen demand
EC	—	electrical conductivity (mS cm^{-1})
F_p	—	membrane flux (kg m^{-2} h^{-1})
NF	—	nanofiltration
OMW	—	olive mill wastewater
Q_c	—	concentrate stream flow (kg h^{-1})
Q_f	—	feed stream flow (kg h^{-1})
Q_p	—	permeate stream flow (kg h^{-1})
R	—	coefficient of determination, unitless
R_{COD}	—	COD rejection rate (kg m^{-2} h^{-1})
$R_{\mathrm{Conductivity}}$	—	electrical conductivity (EC) rejection rate (kg m^{-2} h^{-1})
RO	—	reverse osmosis
TSS	—	total suspended solids
UF	—	ultrafiltration

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