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Comparison of the Performance of two Reverse Osmosis Membranes for the Final Purification of Olive Mill Wastewater

Javier Miguel Ochando-Pulido*[a], María Dolores Víctor-Ortega[a], Marco Stoller[b], Antonio Martínez-Férez[a]

[a]Chemical Engineering Department, University of Granada, Avda. Fuentnueva s/n, 18071 Granada, Spain
[b]University of Rome "La Sapienza", Department of Chemical Engineering, Via Eudossiana, 18-00184 Rome, Italy
jmochandop@ugr.es

Two quite different reverse osmosis (RO) polymeric membranes were examined for the final purification of olive mill wastewater from two-phase olive mills (OMW2): the first one is a thin-film composite (TFC) membrane consisting of polyamide active layer on polysulfone ultrafiltration support, whereas the other one is a low-pressure membrane made of asymmetric polyamide.
A net operating pressure (P_{TM}) of 25 bar was found as the target for the TFC membrane, whereas for the asymmetric one a P_{TM} of 8 bar was chosen, given that similar flux decay but still significant productivity was observed by increasing the P_{TM} for this membrane. These results are confirmed by the fouling index (b) values calculated for each membrane.
Complete removal of suspended solids, phenolic compounds and iron was achieved by both membranes. Otherwise, the asymmetric membrane ensured slightly higher organic matter (COD) and electroconductivity (EC) reduction, leading to a COD concentration in the permeate stream equal to 3.7 mg L^{-1} and 1.4 mg L^{-1} (TFC vs. asymmetric), whereas the EC values were 97.0 and 31.0 μs cm^{-1}, respectively. This would permit reusing the purified effluent provided by both membranes in the production process and close the loop at industrial scale.
Moreover, the asymmetric membrane provides a steady-state flux value of the same order of that yielded by the TFC membrane upon more than three times less P_{TM} (14.9 L $h^{-1}m^{-2}$ at P_{TM} = 8 bar vs. 15.2 L $h^{-1}m^{-2}$ at P_{TM} = 25 bar), implying a reduction of the specific energy consumption above 50 %, from 0.30 € m^{-3} for the TFC membrane to 0.14€ m^{-3} for the asymmetric one.

1. Introduction

Modern medium-sized olive oil factories by-produce between 10 and 15 m^3 of olive mill wastewater (OMW) daily on average. OMW therefore represents not only an environmental threat, but also implies a huge cost for this industry, since OMW cannot be directly reused for irrigation purposes. OMW presents a high toxicity given by the high concentration of aromatic compounds and a wide range of other organic pollutants which cannot be biologically abated (Ammary, 2005; Paraskeva et al., 2006; Martínez-Nieto et al. 2011).
A plethora of reclamation processes and integral treatments have been developed and reported in the last decade, without yielding complete satisfactory results whether from the point of view of the economic efficiency or the efficacy (Aktas et al., 2001; Ammary, 2005; Al-Malah et al., 2000; Bouranis et al., 1995; Espuglas et al., 2002; Grafias et al., 2010; Inan et al., 2004; Lafi et al., 2009; Papastefanakis et al., 2010; Paraskeva et al., 2006; Rizzo et al., 2008; Tezcan Ün et al., 2006). Among them, Fenton's process seems to be the most economically efficient due its equipment simplicity and operational ease, and the key fact that it can be performed at ambient temperature and pressure conditions (Cañizares et al., 2009; Martínez-Nieto et al. 2011).
Pressure-driven membranes were revealed in the last decade as a very promising technology for separation and purification processes. The improvement of this technology has permitted microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) to be implemented in the last years for

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municipal, agricultural and industrial wastewater reclamation (Lee et al., 2006; López et al., 2006; Pizzichini et al., 2005; Turano et al., 2002; Stoller et al., 2006). Several works have been conducted in the past by means of membrane technology with the target to reduce the organic load of OMW, including ultrafiltration (UF) (Akdemir and Ozer, 2009; Stoller and Chianese, 2006a, b and 2007; Turano et al., 2002), nanofiltration (NF) (Stoller, 2009, 2011; Stoller et al., 2013a, b) and reverse osmosis (Coskun et al., 2010), but only few focusing on OMW by produced by two-phase olive oil factories (OMW2) (Ochando-Pulido et al., 2012a, b).
In many research works membrane fouling has been highlighted to influence deeply the membrane operation, leading to operating and energy costs increase, frequent plant shut-downs for in-situ membrane cleaning and even irretrievable membrane life shortening, too. Hence, control of fouling is a key factor to increase the cost-efficiency and competiveness of this technology.
Some research groups have observed that the non-adoption of specifically tailored pretreatment processes leads to rapid development of fouling on the membranes (Stoller and Chianese, 2006a, b and 2007; Turano et al., 2002; Ochando-Pulido et al., 2012a, b). Moreover, other factors with high influence on the membranes performances are the feedstock composition, the membrane type, roughness and porosity, and especially the hydrodynamic conditions.
The present study focuses on the reclamation of OMW2 in order to achieve the quality to recirculate the final effluent to the manufacture process, that is, to the olives washing machines to finally close the loop, or reuse it for irrigation. Two rather different RO polymeric membranes were selected and tested for the final purification of OMW2 after a secondary treatment comprising Fenton's reaction, flocculation and filtration through olive stones previously set-up by the Authors (Hodaifa et al., 2013a,b; Martínez-Nieto et al., 2011). The SC module is a thin-film composite (TFC) RO membrane consisting of a polyamide active layer on a polysulfone ultrafiltration support, whereas the AK module is a low-pressure RO membrane made of asymmetric polyamide.
The performance of the selected membranes in terms of productivity and permeate quality were confronted and discussed. Moreover, the fouling build-up was also analyzed as a function of the applied operating pressure conditions and modelized by the critical flux theory. Finally, the compliance of the pursued standards was checked.

2. Materials and methods

2.1 Analytical methods

Wastewater samples were collected from various olive oil mills in the Andalusian provinces of Jaén and Granada (Spain), operating with the two-phase centrifugation system (OMW2), and rapidly analyzed in the lab.
After this, the OMW2 samples were conducted to a secondary treatment previously optimized by the Authors (Martínez Nieto et al., 2011; Hodaifa et al., 2013a, 2013b), prior to the entrance to the final membrane purification process.
Chemical oxygen demand (COD), total suspended solids (TSS), total phenolic compounds (TPh), total iron, electroconductivity (EC) and pH were measured in the raw effluents samples and at the end of each depuration step following standard methods (Greenberg et al., 1992). All the chemical reagents used for the analytical proceedings presented analytical grade with purity over 99 %, and the analyses were performed in triplicate.

2.2 Membrane bench-scale plant and RO experiments

The membrane RO filtration tests were carried out in a lab-scale membrane plant (Prozesstechnik GmbH, Basel, Switzerland) schematically showed in Fig. 1. The membrane plant was equipped with a non-stirred double-walled tank (5 L maximum capacity) where the OMW2 samples (2 L) were poured, then pumped through the flat membrane module (3.9 cm width x 33.5 cm length) by means of a diaphragm pump (Hydra-Cell model D-03).
The main process parameters comprising the operating pressure, temperature and tangential velocity were measured and displayed. The operating pressure was accurately adjusted (P_{TM} ± 0.01 bar) with a spring loaded pressure-regulating valve on the concentrate outlet and monitored by a digital pressure gauge, allowing independent control of the operating pressure and the flowrate. The operating temperature (T ± 0.1 °C) was regulated automatically via an electronic temperature controller.
The characteristics of the selected RO membranes (GE Water and Process Technologies, USA) are reported in Table 1. The active area of the membranes was 200 cm^2. Before each RO experiment, the corresponding membrane was equilibrated by filtering MilliQ® water at a fixed pressure and temperature until a constant and stable flux was observed, to allow for membrane compaction.
Once a stable flux was achieved, the hydraulic permeability (K_w) of each selected membrane was determined by measuring the pure water flux within their pressure range, at ambient temperature and turbulent crossflow velocity.
RO experiments were driven in a semibatch mode, at ambient temperature (22 ± 0.1 °C) and turbulent tangential-flow regime over the membrane (tangential velocity equal to 2.55 m s^{-1}, providing $N_{Re} > 4000$). The

P_{TM} was fixed at 3, 5 and 8 bar for the experiments with the low-pressure asymmetric RO membrane (AK), whereas P_{TM} values equal to 15, 25 and 35 bar were set for the runs with the composite (TFC) RO membrane (SC).

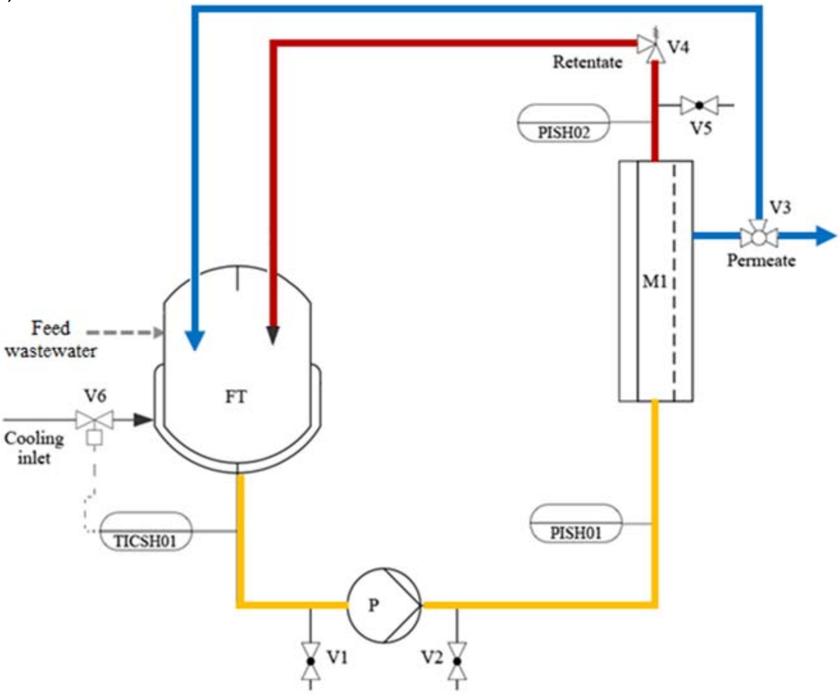

Fig. 1. Flow diagram of the bench-scale membrane filtration unit. M1: flat-sheet membrane module, P: feedstock pump, FT: feedstock tank; V1, V2: emptying valves; V3: permeate flux by-pass valve V4: pressure regulating valve; V5: venting valve; V6: magnetic valve for cooling jacket inlet; PISH01, PISH02: pressure gauges; TICSH01: temperature gauge.

All the experiments were performed for a complete diafiltration cycle (Dc), i.e. 2 L of permeate volume collected, which means a feed recovery rate (F_R) over 90% taking into account a 200 mL dead volume in the system. The concentrate stream was returned continuously to the feed tank whereas the permeate stream was steadily collected, replacing it with the same volume of fresh pretreated OMW2.
Samples of permeate were collected and analyzed to evaluate the membrane separation effectiveness with regard to the COD and EC. The permeate flux was continuously measured during operation time by a precision electronic mass balance (AX -120 Cobos, 0.1 mg accuracy). After each diafiltration run, the membrane was recovered for the following experiment by cleaning it in situ with 0.1-0.15% w/v NaOH and 0.1-0.15% w/v sodium dodecyl sulfate (SDS) solutions (provided by Panreac S.A.).

Table 1: RO membranes' specifications

Membrane type	Model series	Material	Membrane structure	Membrane surface	Pore size, nm	MWCO, Da	Max. P, bar	Max. T, °C
RO	AK	Aromatic PA*	Asym.+	Hydrophilic	< 0.1	-	9	50
RO	SC	PA*/ PS**	TFC++	Hydrophilic	< 0.1	-	40	90

* PA: polyamide; **PS: polysulfone; ++Asym.: asymmetric; ++TFC: thin-film composite.

3. Results and discussion

The physicochemical composition of the OMW2 stream prior to the inlet to the final RO unit is reported in Table 2. The objective of this RO purification stage is the reduction of the high organic matter concentration (COD) remaining in the pretreated OMW2 stream, as well as the removal of the inorganic dissolved solids, mainly sodium and chloride ions, which confer high EC values to this effluent.
The measured pure water permeability values (K_w, L $h^{-1}m^{-2}bar^{-1}$) of the selected RO membranes are given in Table 3. As it can be observed, the asymmetric RO membrane (AK) presented a K_w more than four times higher than that yielded by the TFC membrane (SC), that is, 6.0 L $h^{-1}m^{-2}bar^{-1}$ for the SC membrane module in

contrast with 1.4 L $h^{-1}m^{-2}bar^{-1}$ for the AK membrane. This is mainly due to the higher rugosity of the former membrane if compared with the latter, which enables higher contact surface between the active layer and the bulk solution (OMW2).

Table 2: OMW2 characterization prior to RO inlet

Parameter	Parametric value
pH	7.7
EC, μS cm^{-1}	3430
TSS, mg L^{-1}	13.1
COD, mg L^{-1}	150.8
Total phenols, mg L^{-1}	0.4
Total iron, mg L^{-1}	0.03
Cl^-, mg L^{-1}	990.9
Na^+, mg L^{-1}	718.6

EC: electrical conductivity; TSS: total suspended solids;
COD: chemical oxygen demand.

Table 3: Permeability measurement of RO membranes

Membrane	K_w, L $h^{-1}m^{-2}bar^{-1}$
SC	1.4 ± 0.3
AK	6.0 ± 0.5

K_w: pure water permeability.

Once the permeability value of each membrane was determined, RO runs for the purification of the pretreated OMW2 stream were carried out in diafiltration mode at different net operating pressure values as a function of the admissible operating pressure range of each of the selected membranes. The results of the RO experimental runs are reported in Table 4, where the initial ($J_{p\ 0}$, L $h^{-1}m^{-2}$) and steady state ($J_{p\ ss}$, L $h^{-1}m^{-2}$) permeate flux values yielded by the selected RO membranes are reported for comparison purposes.

It is very important to analyse the steady-state performance of a selected membrane. The reason is that although a chosen membrane may present higher permeability, that is, it may initially provide higher permeate flux values, the development of a fouling resistance during the course of the operation time may shift the productivity and selectivity. This is an error typically committed by engineers when selecting the target permeate flux values and hence setting the operating conditions of the membrane plant (Coskun et al., 2010). In this regard, the analysis of the dynamic fouling build-up during the RO operating time gives very useful information.

As it can be observed in Table 4, the increment of the net operating pressure (P_{TM}) of the system leads on one hand to higher initial permeate flux values for both membranes, but on the other hand it leads to higher permeate flux loss ($-\Delta J_p$) during the operation time. For the TFC RO membrane this is especially marked for a pressure value above 25 bar. Choosing a P_{TM} value of 35 bar does not lead to a stable state of the system and the permeate flux continuously decays. The permeate flux loss for this membrane (SC) ranges from 20.5 - 20.0 % for 15 - 25 bar, whereas it is much more severe for a P_{TM} equal to 35 bar (47.0 %). Otherwise, this increment of the permeate flux decay with the operating P_{TM} set is less marked for the asymmetric membrane (AK), ranging from 48.3 to 50.3 % for P_{TM} between 3 - 8 bar.

These results are confirmed by the values of the fouling index (b), calculated for each membrane upon the different conditions assayed by fitting the experimental flux data with the classical extended fouling - flux equation (Cheryan, 1998): $J_{p\ t} = (J_{p\ 0} - J_{p\ c})\cdot exp(-b\cdot t) + J_{p\ c}$ where b is the fouling index (h^{-1}), $J_{p\ 0}$ is the initial permeate flux (L $h^{-1}m^{-2}$), $J_{p\ t}$ is the permeate flux at any time t (L $h^{-1}m^{-2}$) and $J_{p\ c}$ is the critical flux (L $h^{-1}m^{-2}$) (Stoller and Chianese, 2006a; Stoller, 2011) (Table 4). Therefore, a P_{TM} equal to 25 bar was selected as the target for the TFC membrane, whereas for the asymmetric membrane a P_{TM} of 8 bar was chosen, given the fact that similar permeate flux decay but still significant productivity was observed by increasing the P_{TM} for this membrane.

The analysis of the permeate flux streams of the selected RO membranes are reported in Table 5. As it can be noted, both membranes provide similar rejection behaviour of the desired parameters. Complete removal of the TSS, total phenols and total iron was achieved by both RO membranes. Otherwise, the asymmetric membrane ensured slightly higher organic matter (COD) and EC reduction, leading to a COD concentration in the permeate stream equal to 3.7 mg L^{-1} and 1.4 mg L^{-1} (TFC vs. asymmetric), whereas the EC values were equal to 97.0 and 31.0 μs cm^{-1}, respectively. This would permit reusing the purified effluent provided by both membranes in the proper olive oil production and therefore close the loop of the industrial process.

Table 4: Permeate flux (initial and steady-state) yielded by the selected RO membranes

Membrane	P_{TM}, bar	$J_{p\,0}$, L $h^{-1}m^{-2}$	$J_{p\,ss}$, L $h^{-1}m^{-2}$	$-\Delta J_p$, %	b, h^{-1}
SC	15	15.7	12.4	20.5	1.04
	25	21.8	15.2	20.0	0.66
	35	32.1	17.0	47.0	1.59
AK	3	5.8	3.0	48.3	0.31
	5	15.1	7.6	49.7	0.33
	8	30.0	14.9	50.3	0.36

P_{TM}: net operating pressure; $J_{p\,0}$: initial permeate flux; $J_{p\,ss}$: steady-state permeate flux; $-\Delta J_p$: permeate flux loss; b: fouling index.

Finally, a comparison of both membranes in terms of productivity permits highlighting the economic advantage of the asymmetric RO membrane (AK). As it can be withdrawn from the results of the diafiltration RO experiments for the pretreated OMW2 purification, the asymmetric membrane provides, upon the appropriate operating conditions, a steady-state permeate flux value (i.e. 14.9 L $h^{-1}m^{-2}$ at P_{TM} = 8 bar) of the same order of that yielded by the TFC membrane (i.e. 15.2 L $h^{-1}m^{-2}$ at P_{TM} = 25 bar). However, it is clear the higher cost-efficiency of this membrane (AK) in comparison with the TFC one, providing similar productivity upon more than three times less P_{TM}. This would signify the reduction of the specific energy consumption of more than 50 %, that is, from 0.30 € m^{-3} for the TFC membrane to 0.14€ m^{-3} for the asymmetric one.

Table 5: Characterization of the permeate streams of the selected RO membranes

Parameter	Permeate SC membrane	Permeate AK membrane
pH	7.6	7.2
EC, µS cm^{-1}	97.0	31.0
TSS, mg L^{-1}	0	0
COD, mg L^{-1}	3.7	1.4
Total phenols, mg L^{-1}	0	0
Total iron, mg L^{-1}	0	0

EC: electrical conductivity; TSS: total suspended solids; COD: chemical oxygen demand.

4. Conclusions

Two quite different reverse osmosis (RO) polymeric membranes were examined for the final purification of olive mill wastewater from two-phase olive mills (OMW2), that is, a thin-film composite (TFC) membrane (polyamide active layer on polysulfone ultrafiltration support) and a low-pressure membrane of asymmetric polyamide.

A net operating pressure (P_{TM}) of 25 bar was found as the target for the TFC membrane, whereas a P_{TM} of 8 bar was chosen for the asymmetric one, given that similar flux decay but ensuring significant productivity was observed. These results are confirmed by the fouling index (b) calculated for each membrane.

The asymmetric membrane ensured slightly higher organic matter (COD) and electroconductivity (EC) reduction, leading to a COD concentration in the permeate stream equal to 3.7 mg L^{-1} and 1.4 mg L^{-1} (TFC vs. asymmetric), whereas the EC values were 97.0 and 31.0 µs cm^{-1}, respectively. Complete removal of suspended solids, phenolic compounds and iron was also ensured by both membranes. This would permit reusing the purified effluent in the production process and close the loop at industrial scale.

However, the asymmetric membrane provides a steady-state flux of the same order of the TFC membrane but upon much lower P_{TM} (14.9 L $h^{-1}m^{-2}$ at P_{TM} = 8 bar vs. 15.2 L $h^{-1}m^{-2}$ at P_{TM} = 25 bar), implying a reduction of the specific energy consumption above 50 %, from 0.30 € m^{-3} for the TFC membrane to 0.14€ m^{-3} for the asymmetric one.

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