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Technical set-up of a reverse osmosis membrane unit in continuous operation for final purification of two-phase olive oil mill wastewater

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ABSTRACT

In the present work, the final purification of olive mill wastewater from two-phase extraction mills, previously subjected to a secondary treatment (OMW2ST), was performed by means of a RO operation stage in continuous operation. Within this framework, it was found that the performance of the membrane with regard to the steady-state permeate flux started to decrease over an applied net pressure of 25 bar, such that further increments of the net operating pressure did not lead to increased stable flux values. Results point that above 2.5 m s^{-1} cross flow the flux tends to be asymptotic. Moreover, the flux loss due to membrane fouling was similar disregarding the temperature, which would have important implications in the cost-efficiency of the process, offering the possibility of working efficiently at ambient temperature conditions, thus giving a sensible economic boost to the OMW2ST purification. The ratio between the cleaning time, consisting in 15 minutes acid cleaning plus 15 minutes alkaline/detergent cleaning, and the operating cycle (46.8–48 h) was equal to $\eta_{\text{tc/top}} (\%) = 1.04 \%$, recovering satisfactorily the membrane permeability. Finally, the standards for discharging in public waterways or reusing the final treated effluent in the proper production process were accomplished.

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1. Introduction

The technological change of the ancient batch press method for the continuous centrifugation-based processes used currently in olive oil industries has led to a significant increase in the generated amounts of wastewater streams. While in traditional olive oil mills the flowrates of the by-produced effluents ranged between 0.4 and 0.6 m^3 per ton of processed olives, today a medium-sized olive oil mill can lead to the generation of more than $15\text{--}20 \text{ m}^3$ daily. These effluents are mainly generated during the washing of the fruit and in the centrifugation process, but also in the cleaning of the hopper and sanitation, and are known as olive mill wastewater (OMW).

The treatment of these effluents is becoming a problem affecting not only the Mediterranean countries, which are the main olive oil producers worldwide, but also other countries where the climatic conditions can enhance this crop and it is now an emergent agro-food industry. This is especially the case of China, where the production of olive oil is rapidly growing and a great production potential is expected, as well as other European

countries, the USA, Australia and the Middle East. The management of these effluents is therefore becoming a task of global concern.

In addition, water resources are becoming insufficient in order to satisfy the increasing demand of fresh water worldwide in the past few decades. Water scarcity specially concerns agricultural irrigation, which demands more than 70% of the total water consumption worldwide [1]. Nevertheless, there is a big potential to use regenerated wastewater for irrigation purposes, representing a solution with very positive environmental and economic impacts.

Geographical dispersion of olive oil mills and their small size has typically made the management of OMW quite cost-ineffective. OMW are bio-refractory effluents because of their high concentration in recalcitrant organic contaminants, including a variety of phenolic compounds, tannins, fatty acids and organohalogenated pollutants [2]. Many treatment processes have been suggested for the reclamation of these effluents, but still today the complexity, efficiency and/or costs involved have hindered their scale-up [3–13]. Among them, advanced oxidation processes provide the highest OMW organic pollutants abatement efficiencies [9–13].

OMW are among the heaviest organic-polluted industrial effluents, characterized by a strong odor nuisance, acid pH,

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intensive violet-dark color and high saline toxicity, exhibiting considerable electroconductivity (EC) values [14,15]. Uncontrolled disposal of these effluents represents an environmental hazard because they lead to problems in relation with soil contamination, plants growth inhibition, underground leaks, water body pollution and hindrance of self-purification processes, as well as dramatic impacts to the aquatic fauna and to the ecological status [2,13].

In olive mills operating with two-phase centrifuges for the extraction of the olive oil, the injection of water needed to fluidize the olive paste is one third on average in contrast with the amount required for the three-phase system formerly used, therefore the flowrate of liquid effluent derived is considerably reduced (OMW2 vs. OMW3). As a further consequence, much of the organic compounds remains in the solid waste, which contains more humidity than the pomace from the three-phase system (60–70% in two-phase system vs. 30–45% in three-phase one) and hence this effluent exhibits lower pollutants degree, too [13–15].

Thereby, the two-phase system has been strongly promoted in Spain as it appears to be more ecological, although the three-phase system is still surviving in other countries where scarcity of financial support has not permitted the technological switch yet. Nevertheless, neither problems in relation with the reclamation of OMW2 and OMW3 have been solved yet.

In this context, membrane technology has been more and more implemented in treatment processes for decontamination of wastewater of very diverse sources in the recent years, and in particular reverse osmosis (RO) membranes can ensure the compliance of the most stringent regulations and have been proposed to solve the management of the industrial effluents of a wide variety of sectors, such as stainless steel [16], energy cogeneration [17], nuclear-power [18], textile [19,20] and agro-food industries [21,22], among others.

However, up to the current moment scarce is being published on the application of membrane technology for OMW treatment. Moreover, the existing studies report mainly membrane treatment processes for OMW exiting olive mills operating with the three-phase olive oil production technology, but few membrane studies on OMW generated by olive factories working with the two-phase extraction technology can be found [21,22]. Also, the existing research works are focused principally on the use of ultrafiltration (UF) and nanofiltration (NF) membranes [23–31], yet there is a gap of knowledge on RO purification of OMW [32,33].

Furthermore, it is worth highlighting that many of the available studies on OMW treatment by membrane technology do not correctly address the problem of membrane fouling, which in fact determines the feasibility and cost-effectiveness of the process at industrial scale [30–33]. Engineers design membrane processes with an excessive oversized capacity, up to 319%, too often unnecessarily increasing both the investment and operating costs. In other words, the lack of knowledge and control of membrane fouling represents an additional cost for this industry which must be minimized to permit successful competitiveness. This especially concerns wastewater purification processes [25].

Moreover, due to the fact that most of the proposed membrane processes for OMW purification are commonly batch treatment operations, deleterious fouling is suffered as a consequence of the rapid pollutant concentration increase in the feedstock throughout the batch operation time, leading to large flux and permeate quality losses. Irreversible fouling can also cause significant energy costs increase and irretrievable membrane life shortage.

In the present work, the final purification of OMW by means of a RO operation stage in continuous operation mode is studied. OMW was previously subjected to a secondary treatment (OMW2ST) thoroughly described in previous works by the authors [13,34,35].

Within this framework, the operating parameters were studied for the continuous operation of the selected RO membrane.

Furthermore, the fouling build-up was examined and modeled to ensure the steady-state operation of the plant. Finally, the standards to reuse the purified effluent whether for irrigation or for recycle in the production process were checked, with the aim of reducing the environmental impact of the olive oil production process and closing the loop.

2. Experimental

2.1. Physicochemical analyses

Analytical grade reagents and chemicals with purity over 99% were used for the analytical procedures, which were applied in triplicate. Chemical oxygen demand (COD), total suspended solids (TSS), total phenols (TPh), total iron, electroconductivity (EC) and pH measurements were carried out in the raw wastewater and at the end of each depuration step according to standard methods [36].

EC and pH measurements were performed with a Crison GLP31 conductivity-meter and a Crison GLP21 pH-meter, with auto-correction of temperature, whereas a Helios Gamma UV–vis spectrophotometer (Thermo Fisher Scientific) served for the COD, TPh and total iron measurements.

For the analysis of the total iron concentration, all iron ions were reduced to iron ions(II) in a thioglycolate medium with a derivative of triazine, forming a reddish-purple complex that was determined photometrically at 565 nm wavelength (Standard German methods ISO 8466-1 and German DIN 38402 A51) [36].

For the determination of the phenolic compounds, reaction with 4-aminoantipyrine at pH equal to 7.9 ± 0.1 was carried out in the presence of potassium ferrocyanide, forming a reddish antipyrine compound measurable spectrophotometrically at 465 nm (UNE 77-053-83, EPA 420.1, US Standard methods 5530 and ISO 6439).

Ionic concentrations were analyzed in the effluent exiting the secondary treatment (OMW2ST) as well as in the permeate stream of the final membrane stage with a Dionex DX-120 ion chromatograph as described in previous works by the authors [21,22]. Effluent samples were diluted when necessary with MilliQ® water for their analysis, whereas the samples of RO permeate were analyzed directly without dilution.

2.2. Effluent samples

For this research work, samples of OMW2 were collected from several olive oil mills in the Andalusian provinces of Jaén and Granada (Spain) during winter months and rapidly analyzed in the lab and refrigerated for further research when necessary. After this, OMW2 was conducted to a secondary treatment on a pilot scale described in detail in former works by the authors [13,34,35]. The OMW2 effluent after the secondary treatment, hereafter referred

Table 1
Physico-chemical analysis of OMW2ST.

Parameters	Parametric value
pH	7.5–7.9
EC, mS cm ⁻¹	3.4–3.8
TSS, mg L ⁻¹	13.1–32.3
COD, mg L ⁻¹	150.8–226.6
Total phenolic compounds, mg L ⁻¹	0.4–1.0
Total iron, µg L ⁻¹	28–100
Cl ⁻ , mg L ⁻¹	990.9–1045.1
Na ⁺ , mg L ⁻¹	691.8–718.6
HCO ₃ ⁻ , mg L ⁻¹	129.3–132.9
Total pesticides, mg L ⁻¹	–

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