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The Use of Membranes in Olive Mill Wastewater Treatment: How to Control Dynamic Fouling?

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Abstract

Membrane fouling leads to increased expenses in the processes in which they are implemented, and this is especially problematic in wastewater treatments. Therefore, fouling minimization and control represents the key to make those processes feasible. In the present paper, a review on the results of fouling control by the critical and threshold flux theories, focusing on olive mill wastewater coming from Spain (OMW-S) and Italy (OMW-I), is briefly covered. In first place, adequate fouling inhibition methods should be designed upstream the membrane operation, in order to make the downstream membrane processes for wastewater treatment technically and economically feasible. The followed strategy allows the operation of the membranes system in a controlled framework that permits the stable operation of the plant. Moreover, the calculated membrane area upon the adoption of feed control (FC) resulted in a requirement for the OMW-S stream of 74.2 and 50.3 m^2 for the UF and NF membranes, respectively, whilst 113.1 and 49.7 m^2 would be required for the treatment of the OMW-I stream. This also minimizes the membrane plant design, avoiding the common over-dimension wrongly estimated by engineers in order to guarantee sufficient operating autonomy to conduct the process that makes it unfeasible.

Keywords: Olive mill wastewater; Control; Fouling; Ultrafiltration; Nanofiltration; Wastewater reclamation

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Introduction

A huge effort has been carried out by the scientific community to reduce the problem of membrane fouling, but it remains still in these days as one of the main challenges of this technology [1]. In particular, this is a big handicap in the use of membranes for wastewater applications, a market where membranes have gained significant use in the last decade. As an example of the implementation of membrane technology for a variety of wastewater streams already we can point stainless steel [2], energy cogeneration [3], nuclear-power [4], textile [5,6], paper [7] and agro-food industries [8-10], among others.

Membrane fouling leads to increased expenses which comprise mainly the need to substitute prematurely the membrane modules, the increment of operating costs owed to the need to intensify the operating conditions to maintain the target productivity as fouling is built-up on the membranes, as well as cleaning procedures for the fouled membranes, membrane ageing, and overdesign of the membrane process to ensure the long-run performance. Also if fouling is of irreversible nature, it can reduce the membrane service lifetime rapidly and drastically. In addition to this, fouling alters the selectivity of the membranes and reduces the output, often making the integration of the membrane unit economically non-viable in wastewater treatment plants. It is evident that in the case of wastewater purification by membranes these facts pose a negative technical and economic handicap for the feasibility of the engineered process, given the limited economic value of the product, that is, purified water. Fouling is a complex phenomenon which involves different mechanisms: pore blocking, plugging or constriction, cake, gel, biofilm formation, adsorption, cake-enhanced concentration polarization, ageing [11-14]. The main factors that determine the extent of fouling build-up on membranes are the membrane chemical nature, physical structure, layer roughness and porosity, the hydrodynamic operating conditions inside the module and the characteristics of the effluent in contact with the membrane, i.e. composition and concentration.

In the last years, as a result of the advances in new membrane materials and module configurations capable of offering enhanced technical and economical performances, the use of membrane technology has been pointed as a feasible solution for the purification of problematic effluents, in conjunction with other processes, usually in the form of integrated pretreatments upstream the membrane operation. In particular for wastewater applications, membranes are currently being used as tertiary advanced treatment for removal of dissolved species such as non-biodegradable organic pollutants, phosphorus, nitrogen compounds, colloidal and suspended solids, and human pathogens, including bacteria, protozoan cysts and viruses [12-15].

The critical and threshold flux theory sustains that the proper operating conditions of membrane processes are key, indicating that there is an operating region or frame that separates the operation of a given membrane upon nil or low fouling from a high and exponential fouling build-up. This theory has been highlighted to be very useful as a tool for the design and control of membrane plants [16]. This has been demonstrated for the treatment of olive mill wastewater by Stoller and Ochando with microfiltration (UF), ultrafiltration (UF), nanofiltration (NF) and RO membranes [17-19]. In the present paper, a review on the results on fouling control by the critical and threshold flux theories, focusing on olive mill wastewater coming from Spain (OMW-S) and Italy (OMW-I), will be briefly reviewed. Inhibition or minimization of the membrane fouling build-up common in membrane facilities is the answer to make the process feasible and imperative to achieve the proper steady-state control of the process to ensure stable performance of the membrane operation.

Olive Mill Wastewaters

In **Table 1**, the main characteristics - including total suspended solids (TSS), chemical oxygen demand (COD), total phenols (TPh) and pH - of the used OMW streams from Spain and Italy (OMW-S and OMW-I) are reported. OMW is a highly polluted effluent by-produced in olive oil industries. Although historically circumscribed to the Mediterranean Basin, these industries are now expanding to other countries in Europe, the USA, Australia and China, thus the problem related to these effluents is becoming a task of global concern. The difficulty in treating OMW resides in its high content in recalcitrant organic compounds, most of which are resistant to conventional and biological processes [20]. Thereby, many treatments have been investigated in the last decades [21-28], but their complexity or lack of cost-efficiency have hindered their implementation at real industrial scale, thus the problem with regard to OMW is still far from being resolved.

Table 1. Physicochemical composition of the OMW streams from Spain (OMW-S) and Italy (OMW-I).

	OMW-S	OMW-I
pH	4.5-5.1	5.0-5.2
TSS, g L^{-1}	3.4-5.8	32.6-33.0
COD, g L^{-1}	16.0-16.5	32.1-32.5
TPh, g L^{-1}	0.2-0.3	0.9-1.0

Straight discharge of OMW has been reported by several authors to cause strong odor nuisance, soil contamination, plants growth inhibition, underground leaks, water body pollution and hindrance of self-purification processes, as well as severe impacts to the aquatic fauna and to the ecological status, due to the presence of bio-refractory contaminants, including a wide variety of phenolic compounds, tannins, fatty acids and organohalogenated pollutants. Due to the presence of high levels of refractory organic compounds, direct disposal of OMW to the municipal sewage treatment plants is also prohibited. OMW contain high concentrations of a wide range of solutes in the shape of suspended solids and colloidal particles which are all very likely to cause membrane fouling, such as organic pollutants, as well as inorganic matter that may also lead to deleterious scaling problems.

Critical and Threshold Flux Theory Applied to Dynamic Fouling Control

Appropriate fouling inhibition methods should be designed upstream the membrane unit, in order to make the membrane processes for wastewater treatment technically and economically feasible. Here we must distinguish between concentration polarization and fouling. Whereas the first is caused by the increasing concentration of solutes within the membrane boundary region, giving rise to an additional resistance and thus raising the operating costs as well as adversely affecting the quality of the permeate stream, the latter is complex and may involve membrane pore blocking, plugging and clogging, chemical degradation and/or cake formation on the membrane surface caused by microorganisms as well as organic and inorganic material, resulting in loss of the permeate flux and alteration of the membrane selectivity [11-17]. Furthermore, membrane fouling can be distinguished between reversible and irreversible fouling depending on the attachment strength of the foulants to the membrane surface: whereas reversible fouling is caused by loosely attached foulants easily removable by a strong shear force or washing, irreversible fouling is caused by foulants strongly attached in the form of pore blocking and plugging, cake, gel and biofilm, difficultly removable by such physicochemical control methods [11-17].

The first theoretical model giving explanation to transport phenomena of colloidal particles in membranes was proposed by the research group of Bacchin et al. [29]. The existence of the critical flux was theoretically proven and physically explained by the authors, who gave a first definition of the critical flux, stating that it is the point at which the repulsive barrier is overcome, and below which no fouling occurs. Afterwards, Field et al. [16] gave an empirical approach of the concept of the critical flux for MF membranes, defining it as the permeate flux which can be attained without incurring in fouling formation during the operation time. Later on, this concept was also extended to UF and NF membranes [30-34].

Concerning the critical flux Jc, the following equations apply [16]:

dm/dt=0; ∀Jp(t) ≤ Jc (1)

dm/dt=B (Jp(t) - Jc);∀ Jp(t) > Jc (2)

Where m is the permeability of the membrane, B is a fitting parameter and Jp(t) the permeate flux at time t.

After some years, some authors noted that fouling cannot be completely inhibited during the operation of some liquid-liquid membrane systems, such as in the treatment of wastewater. These researchers noticed that fouling was unavoidable to a certain extent at every operating condition. Le Clech et al. [35] reported certain fouling in the treatment of wastewater by membrane bioreactors (MBR) even when operating below the critical flux conditions. This behavior was posteriorly confirmed in the treatment of dairy wastewater with NF membranes by Luo et al. [36] and recently in the treatment of olive mill wastewater (OMW) with UF and NF membranes by Stoller and Ochando-Pulido [37]. To overcome this limitation in the definition of critical flux, Bacchin et al. introduced for the first time the concept of threshold flux in a paper in the year 2006 [38]. Summarizing the concept briefly, the threshold flux represents the flux that divides a low fouling region, characterized by a nearly constant rate of fouling, from a high fouling region, where flux-dependent high fouling rates are observed.

Concerning the threshold flux Jth, the proposed equations are as follows [16,38]:

dm/dt=a; ∀ Jp(t) ≤ Jth (3)

dm/dt=a + b (Jp(t) - Jth); ∀ Jp(t) > Jth (4)

Where a, b are both fitting parameters related to fouling.

In order to determine the critical or threshold flux the main difficulty relies in the impossibility of theoretical prediction, thus experimental estimation is necessary [29-37]. Furthermore, several factors affect these values, such as the membrane type, the membrane surface roughness and mean porosity, the hydrodynamic conditions and the effluent composition and concentration. In regard to the latter, direct treatment by membranes of raw effluents has been reported to lead to rapid emergence of membrane fouling.

The author suggests the use of the pressure stepping method as it is graphically explained in **Figure 1**. This method is extended from the one used by Espinasse et al. in previous work [39,40], and it was found to be reliable and relatively quick. Basically, the method consists of cycling the applied pressure up and down, by a constant pressure (TMP) variation equal to ΔTMP, and to check for the reproducibility of the permeate flux at same pressure values before and after the pressure changes. The lowest pressure value at which the difference between the former and latter flux value at a given TMP becomes positive is the threshold flux value (Jth), and the corresponding threshold pressure (TMPth).

Tailored OMW Pretreatments

An optimized pretreatment process, specifically tailored to the application, is a first essential step for the design of an appropriate fouling inhibition strategy, in particular when polluted streams such as wastewater streams are subjected to a membrane operation [17,19]. Stoller and Ochando-Pulido et al. [41] proposed two different pretreatments for OMW in order to reduce fouling upstream membranes-in-series process studied at lab and pilot scales. Once lab-scale optimization was accomplished, the two pretreatments were scaled up: a rather simple flocculation process based on pH and temperature optimization, and a photocatalytic process enhanced by self lab-made ferromagnetic nanoparticles of TiO_2 [41]. The details of both pretreatments can be found elsewhere [41]. The results are summarized in **Table 2**.

Membranes-in-series Process at Pilot-scale

The UF and NF membranes filtration pilot plant used for the study (100 L feed tank) is shown schematically in **Figure 2**. The used membrane modules were all polymeric ones in spiral-wound configuration (2.5 m^2 surface) provided by GE Osmonics, with the characteristics reported in **Table 3**.

Validation of Critical and Threshold Flux Theories

In first instance, threshold flux measurements were performed with the method for critical flux estimation proposed by Espinasse et al. [39]. Upon operation at pressure values equal to the threshold ones (P_{th}), very low fouling rates were observed for all membrane separation steps. After the first minutes of operation, where polarization establishes, a plateau was reached for both UF and NF membranes up to the end of operation, that is the recovery of minimum 80% of the feedstock. The results are summarized in **Table 4**.

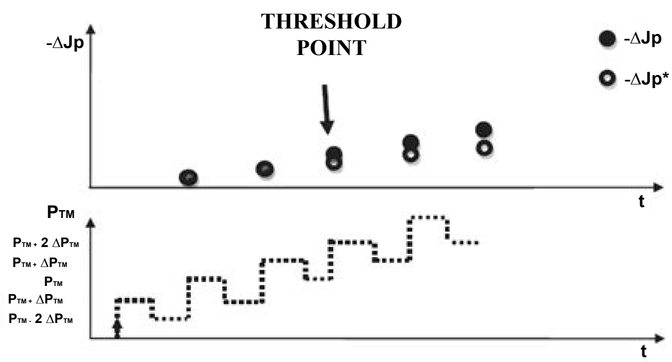

Figure 1 Threshold flux (J_{th}) determination by means of the pressure-stepping method.

Table 2. OMW-S and OMW-I physicochemical composition after whole pretreatment procedure.

Parameters	OMW-S	OMW-I
pH	2.5 ± 0.2	3.0 ± 0.2
TSS, g L^{-1}	1.0 ± 0.1	5.0 ± 0.1
COD, g L^{-1}	11.0 ± 0.1	15.0 ± 0.2
TPh, g L^{-1}	0.1 ± 0.01	-

In particular, further COD reduction for both feedstocks (**Figure 3**) enabled fouling minimization on the downstream membranes, as evaluated by the measured threshold flux values (**Figure 3**).

The obtained results are satisfactory, since many times the author reported that without any pretreatment, that is for values higher than 44000 mg L^{-1} in OMW streams, almost instantaneous zero flux conditions are met. Moreover, the process can hold on approximately 96 h before washing is required. Furthermore, in 2015 Stoller and Ochando merged both critical and threshold flux concepts into the boundary flux (J_b) concept, also relevant for the appropriate design of membrane processes control systems [42]. In case of adopting a feed control (FC) strategy, the required membrane area A_f can be evaluated with the following relationship:

$$A_f = F_{sp}\,(1 - \delta_F)^{-1}\, J_b(KP'_c)^{-1}\,(1 + (C - 1)\,\tau\,\Delta w\%) \quad (5)$$

where KP'_c is the final value of the key parameter KP (in this case the COD value), estimated from a starting value of KP(0) of the feedstock in worst-case conditions, that is, in total rejection conditions of KP, as a function of the feed recovery Y^*; C is the number of desired separation cycles lasting τ hours, considering the loss of permeability Δw% after every membrane cleaning procedure; and δ_F represents a safety margin (5-10%). If $J_b(KP'c) < 0$ the designed process is not technically feasible. Otherwise, F_{sp} is the set point of the feed flow rate, which should be set to:

$$F_{sp} = F_p^* = V\,Y^*\,\theta^{*-1} \quad (6)$$

Where V is the feedstock volume and θ^* is the set batch operation time, generally equal to τ.

Following these guidelines, the calculated required membrane area upon the adoption of FC control strategy resulted in a requirement for the OMW-S stream of 74.2 and 50.3 m^2 for the UF and NF membranes, respectively, whereas 113.1 and 49.7 m^2 would be required for the treatment of the OMW-I stream.

To sum up, this strategy avoids on one hand the operation of the membranes system above the operating conditions that maintain fouling in a controlled framework, and thus permits the stable operation of the plant. On the other hand, it minimizes the membrane plant design, avoiding the common overdesign wrongly estimated by engineers in order to guarantee sufficient operating autonomy to conduct the process for a certain period of time. In most cases the over-design is performed a forfeit or by past experience of the designer, starting only from the knowledge of the permeate project value, without considering in detail the entity and nature of fouling. In other cases, even worse, engineers under design the membrane plant, relying on higher operating conditions, unsustainable to run the plant for long period of time. In both cases the designer partially failed in engineering the process, which becomes at the end too costly or unreliable.

Conclusions

A review on the results of fouling control by the critical and threshold flux theories focusing on olive mill wastewater coming

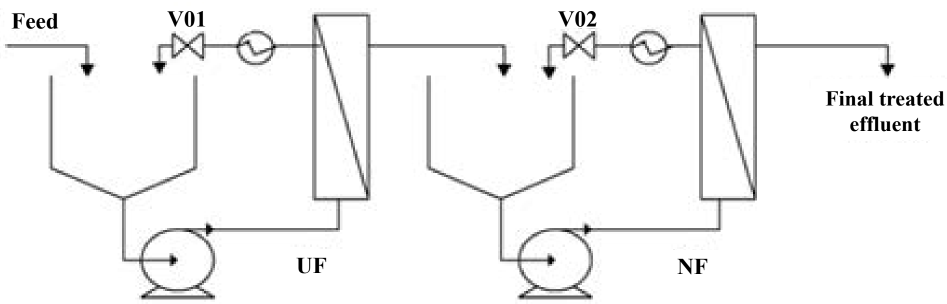

Figure 2 Membranes-in-series process for the treatment of OMW.

Membrane type	Model series	K_w, $L\,h^{-1}m^{-2}\,bar^{-1}$	Pore size, nm	MWCO, Da	Surface, m^2	Max. P, bar	Max. T °C
UF	GM	5.2	2	8,000	2.5	16	50
NF	DK	2.5	0.5	300	2.5	32	50

Kw: pure water permeability; MWCO: molecular weight cut-off.

Table 3. Main characteristics of the selected membranes.

Feed stream	Membrane	P_{th}, bar	J_{th}, $L\,h^{-1}m^{-2}$
OMW-S	UF	9	9.4
	NF	8	12.5
OMW-I	UF	4	0.8
	NF	5	6.9

P_{th}: threshold pressure; J_{th}: estimated threshold flux.

Table 4. Threshold flux and pressure measured on OMW-S and OMW-I streams.

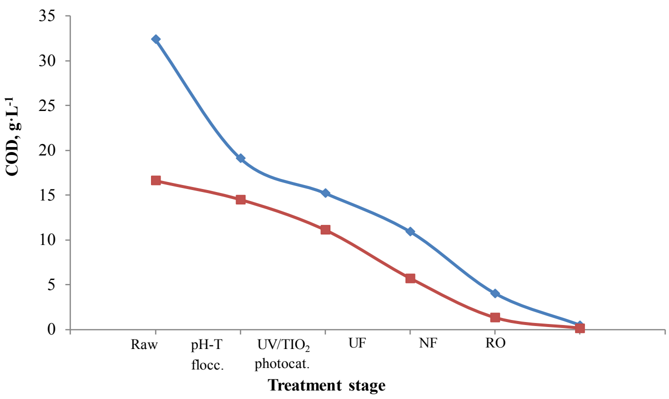

Figure 3 COD removal in OMW (blue: OMW-I; red: OMW-S) during the treatment steps.

from Spain (OMW-S) and Italy (OMW-I) are briefly reviewed in this paper. Inhibition or minimization of the fouling build-up common in every membrane facility is the key to make the process feasible and imperative to achieve the proper steady-state control of the unit to ensure stable performance of the membrane operation. By running the system at pressure values equal to the threshold ones, very low fouling rates were observed on the used UF and NF membranes. After the first minutes where polarization is established, a plateau was observed until the end of operation that is the recovery of minimum 80% of the feedstock. Following these guidelines, the calculated membrane area upon the adoption of feed control resulted in a requirement of 74.2 and 50.3 m^2 for the UF and NF membranes to treat the OMW-S stream, whereas 113.1 and 49.7 m^2 would be required respectively for the treatment of the OMW-I stream. Moreover, this strategy permits the operation of the membranes process in a controlled framework that ensures the stable operation of the plant. In addition, it minimizes the membrane plant dimensioning, avoiding the common overdesign wrongly estimated by engineers in order to guarantee sufficient operating autonomy to conduct the process that makes it not feasible.

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