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### **DRINKING WATER PRODUCTION FROM LOW-QUALITY INFLUENTS BY ULTRAFILTRATION WITH OZONATION PRE-TREATMENT**

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Ultrafiltration is not fully applied to drinking-water production because of the two main drawbacks of the membranes: low retention of natural organic matter and fouling. Nevertheless, the application of pre-treatments can help to improve the membrane performance; thus, a study was made with ozonation prior to ultrafiltration for a fixed influent water quality. The experiments were conducted with real-scale spiral-wound membranes in a pilot plant located in Granada (Spain), for 1.1 m<sup>3</sup>/h flow rate and high-humic-content influents (7.5 mg DOC/L). The aim of the study was to compare the effluent quality achieved with and without pre-ozonation. Doses between 5 and 25 g O<sub>3</sub>/Nm<sup>3</sup> were tested to determine whether the ozone concentration affected the resulting effluent quality and membrane fouling. The results showed that the ozone application slightly improved DOC removal efficiency, from 13 to 30%, and helped control fouling, reducing the average fouling rate by almost 3-fold compared to single ultrafiltration. However, raising the ozone rate from 5 to 25 g/Nm<sup>3</sup>, (equivalent to 0.84 and 4 mg/L) had no appreciable influence. These findings question the suitability of ozonation as pre-treatment for ultrafiltration applied to drinking-water production and suggest that other processes should be applied instead.

**Keywords:** *Ozonation; ultrafiltration; fouling; natural organic matter; low-quality influents; Dissolved Organic Carbon (DOC)*

### **PRODUCCIÓN DE AGUA POTABLE MEDIANTE ULTRAFILTRACIÓN A PARTIR DE INFLUENTES DE BAJA CALIDAD CON OZONIZACIÓN COMO PRETRATAMIENTO**

La obtención de agua potable mediante membranas de ultrafiltración no está totalmente extendida debido a la baja retención de materia orgánica natural de las membranas y su ensuciamiento. Sin embargo, la aplicación de pretratamientos puede paliar estos inconvenientes. En consecuencia, se llevó a cabo un estudio en una planta piloto localizada en Granada (España), aplicando ozonización previa a la ultrafiltración de influentes con alto contenido húmico (7.5 mg/L COD) y caudales de 1.1m<sup>3</sup>/h. El objetivo principal era comparar la calidad de efluente obtenida con y sin pre-ozonización, probando dosis entre 5 y 25 g O<sub>3</sub>/Nm<sup>3</sup>, para determinar si la concentración de ozono afectaba a la calidad del efluente y al ensuciamiento de la membrana. Los resultados indicaron que la aplicación de ozono mejora ligeramente la eliminación de COD, aumentando el rendimiento de 13 a 30%, y contribuye a controlar el ensuciamiento, con velocidades casi tres veces menores que con ultrafiltración en solitario pero incrementar la dosis de ozono de 5 a 25 g/Nm<sup>3</sup>, equivalentes a entre 0.84 y 4 mg/L, no tuvo ningún efecto. Estos resultados cuestionan la idoneidad de la ozonización como pretratamiento para producir agua potable mediante ultrafiltración, sugiriendo en su lugar la aplicación de otros procesos.

**Palabras clave:** *Ozonización; ultrafiltración; fouling; materia orgánica natural; influentes baja calidad; Carbono Orgánico Disuelto (COD)*

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## 1. Introduction

Membrane technology emerged at the beginning of the 20<sup>th</sup> century for several applications (Baker, 2004). Membranes are porous materials that can retain certain particles inside an aqueous matrix; therefore the use of membranes for water treatment is widespread for drinking-water production, waste-water treatment, and desalination.

From largest to smallest pore size, membranes can be classified as being for microfiltration, ultrafiltration, nanofiltration or reverse osmosis. In particular, ultrafiltration membranes are used worldwide for potabilizing surface water, as this physical treatment provides an alternative to conventional drinking-water production, preventing the subsequent generation of disinfection by-products (DBPs) associated with the former, which is based on chlorination.

Today, the great availability of module materials (ceramic, polymeric, metallic, etc.), configurations (hollow-fibre, flat-sheet, tubular, spiral-wound, etc.) and operation modes (dead-end vs. cross-flow, constant-flux vs. constant pressure, vacuum vs. pressure-driven, etc.), has remarkably increased the versatility of the systems. Furthermore, the equipment is simple to operate and provides water free from biological contamination. In fact, mobile ultrafiltration plants for drinking-water production are used in emergency situations such as natural catastrophes to provide safe drinking water. However, these membranes have two limitations: low retention of natural organic matter (NOM) and fouling (Laîné, Vial, & Moulart, 2000).

According to Laîné *et al.*, (2000) ultrafiltration (UF) alone is not enough to guarantee the required water quality when surface waters with high organic content are processed. The low rejection of dissolved organic carbon (DOC) is a great handicap since the presence of DOC alters the organoleptic properties of water destined for human consumption. On the other hand, fouling, which is the loss of permeability caused by the accumulation of aquatic substances on or inside the membrane matrices (Huang, Schwab, & Jacangelo, 2009), has a great effect on the process costs, since it augments energy expenses and reduces the membrane service life (Regula *et al.*, 2014). Consequently, different strategies need to be followed to reduce or eliminate these drawbacks.

The application of backwashing, chemical cleaning or aeration is useful to mitigate the effects of fouling but insufficient to control it. The application of other processes called pre-treatments before ultrafiltration is very common for improving both DOC rejection and membrane performance, particularly coagulation-flocculation (Gao *et al.*, 2011) or adsorption (Huang *et al.*, 2009). Pre-oxidation with chlorine is also common but it is not with ozone. However, despite that ozone oxidizing potential is higher than that of chlorine, the use of ozonation has been limited because this gas needs to be generated *in-situ*, involving relatively high energy consumption. In addition, some polymeric membranes are incompatible with ozone and the examples found have been restricted to ceramic membranes (Kim, Davies, Baumann, Tarabara, & Masten, 2008; Schlichter, Mavrov, & Chmiel, 2004), with a few exceptions (Byun, Taurozzi, Alpatova, Wang, & Tarabara, 2011). As a result, a pilot study for drinking-water production with a spiral-wound polymeric membrane was conducted in order to advance current knowledge of this matter.

The objective of the present study was double: to determine the degree of contaminant removal achieved with increasing ozone doses and to assess the effect of the pre-treatment on membrane fouling.

## 2. Materials and Methods

### 2.1 Influent water

The water source was Canales reservoir (Granada, Spain). The average values for the water physico-chemical parameters were the following: pH 8.00; turbidity 5.2 NTU;  $UV_{254}$   $0.4 \text{ m}^{-1}$ ; and DOC  $0.5 \text{ mg/L}$ . A mixture of humic/fulvic acids (HFA, Carbotecnia Húmico 10-10 solution, 10% fulvic extract, 10% humic extract, Carbotecnia S.L., Spain) was dissolved in the matrix water to reach a concentration in the influent of  $7.5 \pm 1 \text{ mg DOC/L}$  on average, to simulate a low quality influent. The characterization of the influent water is shown in Table 1.

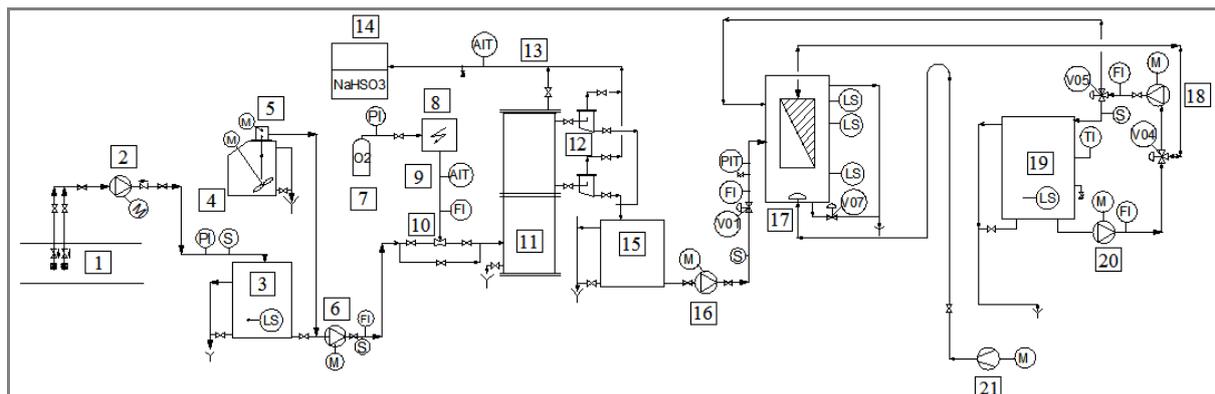
**Table 1. Influent water characterization (Average values)**

Influent	Value
Turbidity, NTU	$5.67 \pm 0.52$
DOC, mg/L	$7.45 \pm 0.46$
$UVA_{254}$ , $\text{m}^{-1}$	$18.67 \pm 1.07$
SUVA, $\text{mg/m} \cdot \text{L}$	$2.51 \pm 0.12$
Colour $_{436}$ , $\text{m}^{-1}$	$3.38 \pm 0.18$
pH	$8.23 \pm 0.09$

### 2.2 Description of the pilot-scale plant

The ultrafiltration facility (Fig. 1) was located in Parque de las Ciencias, Granada (Spain). The membrane used was polyvinylidene fluoride (PVDF) spiral-wound SpiraSep 960, (TriSep Corporation, USA); with an effective pore size of  $0.03 \mu\text{m}$  and  $20.9 \text{ m}^2$  filtration area.

**Figure 1: Flow diagram of the experimental set-up**



1, inlet channel; 2, inlet pump 3, intermediate tank; 4, humic/fulvic acid tank; 5, humic/fulvic acid dosing pump; 6, feed pump; 7, oxygen cylinder; 8, ozone generator; 9, ozone in-line analyser; 10, venturi; 11, ozone contact tower; 12, ozone overflow; 13, off-gas analyser; 14, ozone-destruction solution; 15, ozonated water tank; 16, ozonated water pump; 17, membrane tank; 18, permeate pump; 19, permeate tank; 20, backwashing pump; 21, air blower.

The matrix water was drawn from the inlet channel and the HFA concentrated solution was injected upstream of the plant feed pump (Pentax, PV 10E, Italy) for proper mixing inside the pump body. The flow rate was adjusted manually at  $1.1 \text{ m}^3/\text{h}$  by a variable-speed drive

(Altivar 11 Schneider Electrics, France). Additionally, the feed-pump discharge was connected to a venturi for ozone injection.

Ozone was produced from oxygen (Air Liquide S. A.), with a corona discharge generator (C-L010DTI, AirTree Ozone Technology Co., Ltd., Taiwan) operating at 1.2 bar. The oxygen flow rate applied was 0.2 Nm<sup>3</sup>/h. An in-line ozone analyser (Mini-HiCon, In USA Corp., USA) was used to measure the ozone concentration in the gas line. Doses from 5 to 25 g O<sub>3</sub>/Nm<sup>3</sup> were tested.

Downstream of the venturi, the water-ozone mixture entered a contact column with an 8-min or 12-min retention time to favour ozone transfer. Then, the mixture left the column through an overflow, separating the gas from the water. The ozonated water was conducted to a separate tank, from which it was pumped to the ultrafiltration circuit. The non-reacting gas was released in the upper part of the overflow, ending at a destruction cell, where the ozone reacted with saturated solution of NaHSO<sub>3</sub> producing O<sub>2</sub>. Nonetheless, prior to ozone destruction, the off-gas ozone concentration was measured by a second in-line ozone analyser (Mini-HiCon built in SC010, In USA Corp., USA). Thus, the transferred ozone dose, TOD, was determined with Eq. 1 (Xu, Janex, Savoye, Cockx, & Lazarova, 2002).

$$TOD = \frac{Q_{gas}}{Q_{liq}} \cdot ([O_3]_0 - [O_3]_{res}) \quad (1)$$

where TOD is the transferred ozone dose (g O<sub>3</sub>/L), Q<sub>gas</sub> and Q<sub>liq</sub> are the flow rates for the inlet gas (Nm<sup>3</sup>/h) and the feed water (L/h), respectively, and [O<sub>3</sub>]<sub>0</sub> and [O<sub>3</sub>]<sub>res</sub> are the ozone concentrations (g O<sub>3</sub>/Nm<sup>3</sup>) in the inlet gas and the off-gas streams, respectively.

The water reaching the ultrafiltration circuit was filtrated inside the membrane tank from outside to inside by a permeate pump (Koral KS 75, Kripsol, Spain), creating a vacuum. In addition, the membrane was continuously aerated with an air blower, (SCL 15 DH, FPZ, Italy) and periodically backwashed (CHI2-20, Grundfos, Germany).

The membrane worked with constant flux (1 m<sup>3</sup>/h flow-rate equivalent to 48 LMH) with periodical backwashing phases. The backwashing flow rate (2 m<sup>3</sup>/h), frequency (10 min), and duration (30 s) were fixed according to the results of previous studies (Rojas-Serrano et al., 2015). In addition, continuous aeration (18 Nm<sup>3</sup>/h) was maintained both for permeate production and for backwashing operations. Additionally, transmembrane pressure, TMP, permeate temperature and O<sub>3</sub> concentrations in the inlet and off-gas lines were recorded by a data register (RSG30, Endress Hauser, Switzerland).

## 2.4 Experimental procedure

The study lasted 10 weeks with the operation consisting in 6 working days /1 day of rest with 240-min-long assays. Every ozone dose was tested for two weeks. The influent and effluent were sampled daily and transmembrane pressure (TMP) was registered every 20 s for fouling evaluation. Ozone concentrations in the inlet and off-gas lines were also registered with the same frequency.

After the operation, the membrane remained soaked in clean water. Also, during weekly cleaning in place (CIP), the membrane tank was emptied and filled with permeate while NaClO (50 mg/L) was dosed, followed by 20 min of recirculation.

## 2.4 Analytical methods

Physicochemical analyses were performed daily for the influent and effluent. Some ozonated water samples were also taken at random. All the samples were integrated, 100 mL being collected every 30 min. Then, all the volumes taken during the whole operation time were mixed and analysed.

Turbidity, DOC, UVA<sub>254</sub>, colour<sub>436 nm</sub> and pH were determined. Turbidity was determined as a measurement of the scattered light (DINKO D-112, Spain) while UV-visible spectrophotometer Helios  $\gamma$  (Spectronic Unicam, USA) was used for the analysis of colour<sub>436 nm</sub> and UVA<sub>254</sub>, with 1 cm quartz cell. Glass-fibre filters (Millipore AP4004705) were used to take filtrated samples for DOC measurement, made by combustion TOC Analyser (Formacs HT, SKALAR, The Netherlands).

## 2.5 Fouling assessment

Linear regression was applied to TMP values prior to backwashing vs. operation time for each assay, with the resulting slopes representing the fouling rates. The comparison of fouling rates for the different operating conditions tested (ozone dose and retention time) made it possible to determine whether those operating conditions had any effect on fouling control.

## 2.6 Statistical analyses

Regression models were built for fouling-velocity calculation. In addition, analysis of the variance tests (ANOVA) were made in order to compare the quality of the samples and to determine whether there were statistically significant differences among them at a significance level of 5%,  $p < 0.05$ .

# 3. Results and discussion

## 3.1 Effluent quality

The first overflow, equivalent to an 8-min retention time was used throughout the first week with the lowest ozone doses tested, 5 g O<sub>3</sub>/Nm<sup>3</sup>. However, there was always residual ozone in the off-gas line, meaning the contact time was insufficient. Thus, from then on, all the assays were carried out for 12-min retention times, i.e. using the highest overflow. Even so, residual ozone was found in the off-gas line for all doses (See Fig. 2) but the retention time could not be further increased.

**Figure 2: Ozonation curve for 12-min retention time**

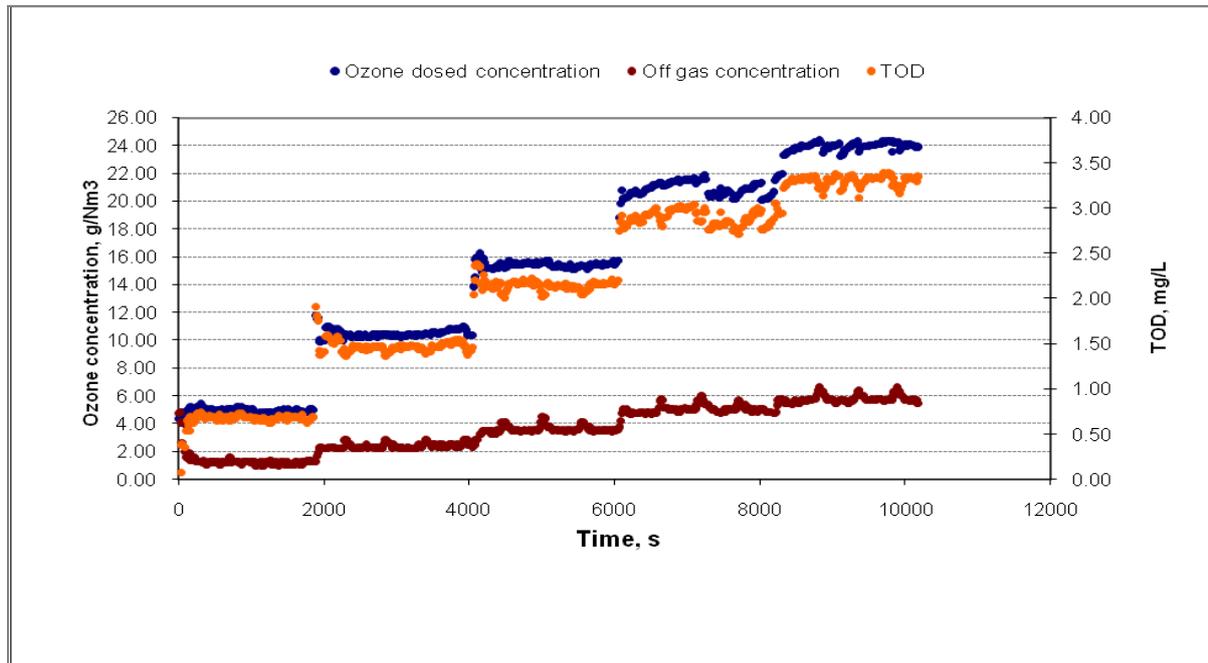


Table 2 shows the effluent quality achieved for single ultrafiltration (results from previous studies) and for the different ozone doses applied.

**Table 2: Effluent quality**

	Single UF		Pre-ozonation+ ultrafiltration			
	0 g O <sub>3</sub> /Nm <sup>3</sup>	5 g O <sub>3</sub> /Nm <sup>3</sup>	10 g O <sub>3</sub> /Nm <sup>3</sup>	15 g O <sub>3</sub> /Nm <sup>3</sup>	20 g O <sub>3</sub> /Nm <sup>3</sup>	25 g O <sub>3</sub> /Nm <sup>3</sup>
Turbidity, NTU	2.0±0.0 (65%)	1.0±0.0 (83%)	1.0±0.0 (83%)	1.0±0.0 (83%)	1.0±0.0 (83%)	1.0±0.0 (83%)
DOC, mg/L	6.5±0.0 (13%)	5.1±0.1 (30%)	5.6±0.0 (29%)	4.9±0.0 (28%)	5.5±0.1 (30%)	5.5±0.3 (30%)
UVA <sub>254</sub> , m <sup>-1</sup>	12.6±0.2 (39%)	11.7±0.4 (41%)	11.2±0.2 (42%)	11.3±0.5 (43%)	11.4±0.1 (42%)	10.4±0.3 (41%)
SUVA, mg/m·L	1.9±0.1	2.3±0.2	1.9±0.1	2.3±0.2	2.1±0.1	1.9±0.1
Colour <sub>436</sub> , m <sup>-1</sup>	1.3±0.2 (55%)	1.2±0.1 (67%)	1.1±0.0 (66%)	1.1±0.2 (68%)	1.2±0.2 (67%)	1.1±0.0 (67%)
pH	8.23±0.1	8.21±0.0	8.26±0.0	8.20±0.1	8.23±0.1	8.22±0.0

The average TOD for 5, 10, 15, 20, and 25 g O<sub>3</sub>/Nm<sup>3</sup> were 0.84 mg/L, 1.99 mg/L, 2.83 mg/L, 3.76 mg/L and 4.00 mg/L, respectively. Nonetheless, increasing the ozone dose did not appear to affect the effluent quality. No significant statistical differences were found for the effluents tested with different doses.

In general, the resulting water quality improved with the ozone application, especially for turbidity. However, the upgrade was not very significant, since the average values achieved for effluent DOC or UVA<sub>254</sub> were only slightly better than for single ultrafiltration. In particular, UVA<sub>254</sub> removal was very low, 41% on average, nearly the same as for the membrane alone. This indicated that almost no aromatic organic matter had been removed with the pre-

treatment. Concerning DOC removal (30%), the figures were better than  $UVA_{254}$  but again insufficient. Effluents with such organic matter contents would require chlorine addition to avoid bacterial growth in the distribution networks and this in turn could generate DBPs, potentially toxic for human consumption (Rojas et al., 2010). Also, the average colour removal was only 67%, a mere 12% higher than without pre-ozonation, very far from the high yields expected for colour degradation.

From the null effect of increasing the initial ozone dose from 5 to 25 g  $O_3/Nm^3$ , it could be deduced that for such organic matter influent concentrations much greater ozone doses would be necessary. Additionally, the figures indicate that the ozone transfer would have been insufficient as well. According to Eq. (1), for a given gas-to-liquid ratio, TOD can be increased by minimizing the residual ozone concentration and/or raising the one at the inlet. On the one hand, higher retention times should have been tested to increase the ozone-water contact and consequently ozone dilution, which would have helped minimize the off-gas concentration, but this was not possible since no further operational height was available in the facility. On the other hand, lower operating temperatures (Langlais, Reckhow, & Brink, 1991) would have allowed greater ozone solubility in water, but this option was disregarded from the beginning as it involved excessive energy consumption. Another option would have been to increase the initial ozone dose to over 25 g/ $Nm^3$ , the maximum allowed by the ozone generator. However, this dose was already equivalent to 0.53 (mg/mg)  $O_3:DOC$  ratio, which should be sufficient according to other researchers (Mozia, Tomaszewska, & Morawski, 2006), who had reported good results with 0.6 (mg/mg)  $O_3:TOC$  ratios. By contrast, Byun et al. (2011) only reached 26% DOC rejection with 0.9 (mg/mg)  $O_3:DOC$  ratios. The explanation for this discrepancy is the different DOC content in the influents used by the different authors: Mozia et al. worked with 4.8 mg DOC/L influents while Byun et al. worked with 11.7 mg/DOC/L. Therefore, our results are consistent with both, since the average DOC in the influent was 7.5 mg/L and 29% membrane rejection was reached, values in between those reported by the aforementioned authors. In addition, Siddiqui et al. (Siddiqui, Amy, & Murphy, 1997) reported that increasing ozone doses beyond an  $O_3:DOC$  ratio of 1:1 does not result in significant reductions in DOC, in agreement with other authors and our own results.

According to these results, further research with a longer contact time and greater initial doses should be performed in order to reach potentially better effluent qualities. However, although the economic feasibility of the process lies beyond the scope of this study, pre-ozonation is not *a priori* a recommendable option because of the high investment required with a potentially modest effluent-quality upgrade.

### 3.2 Membrane fouling

The influence of pre-ozonation on membrane fouling was more significant than on the resulting water quality. Single ultrafiltration fouling velocity was  $-5.0 \cdot 10^{-6}$  bar/s on average with coefficients of determination,  $R^2$ , close to 0.99. In addition, average TMP increments were on the order of -0.07 bar. By contrast, the average fouling velocity achieved during pre-ozonation was  $-1.8 \cdot 10^{-6}$  bar/s. The coefficients of determination were strong but slightly weaker than for single ultrafiltration, between 0.85 and 0.9. Moreover, the TMP increments were around -0.02 bar, substantially lower than without pre-treatment.

Other researchers also found that the application of ozone prior to ultrafiltration enhanced membrane performance (Kim et al., 2008; Lee, Lee, Wan, & Choi, 2005). However, the most remarkable finding was that the ozone dose did not affect membrane fouling, either, as opposed to other reports (Szymanska, Zouboulis, & Zamboulis, 2014). There were statistically significant differences among the values found, but these differences could not be attributed to the ozone doses applied, as the oscillations followed no pattern (Table 3).

**Table 3. Fouling rates for the different ozone doses tested**

Ozone dose (g O <sub>3</sub> /Nm <sup>3</sup> )	Fouling rate (bar/s)
0	-5.00·10 <sup>-6</sup>
5	-1.69·10 <sup>-6</sup>
10	-1.95·10 <sup>-6</sup>
15	-2.00·10 <sup>-6</sup>
20	-1.77·10 <sup>-6</sup>
25	-1.84·10 <sup>-6</sup>

The differences in the fouling rates were due to the fact the TMP data were registered every 20s and backwashing duration was 30s, and therefore the data differed depending on how close to the backwashing the first data was registered. Further experiments should be performed with smaller register frequency, for example 5s, so that the information is more accurate. In any case, these differences were minimum and did not alter the average fouling rate, pre-ozonation helping to delay membrane fouling and to control its effects.

#### 4. Conclusions

The application of ozonation as a pre-treatment for drinking-water production by ultrafiltration membranes slightly improves the resulting effluent quality. However, the upgrade achieved is not sufficient when treating low-quality influents. UVA<sub>254</sub> removal is nearly the same as for the membrane alone, indicating that almost no aromatic organic matter can be removed with the pre-treatment. In addition, average DOC removal is only 29% regardless of whether the ozone dose is increased from 5 to 25 g O<sub>3</sub>/Nm<sup>3</sup>, equivalent to TOD between 0.8 and 4.0 mg O<sub>3</sub>/L. These findings call into question the suitability of ozonation as pre-treatment for ultrafiltration applied to drinking water production and suggest that other processes should be applied instead. Nonetheless, pre-ozonation helps delay membrane fouling, reducing the average fouling rate by almost 3-fold respect to single ultrafiltration, as the lower daily TMP increments demonstrated as well.

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