



## Effect of pH and MWCO on textile effluents ultrafiltration by tubular ceramic membranes

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### ABSTRACT

Textile industries are considered as one of the most polluting among all the industrial sectors. Therefore, the disposal of textile effluents without the appropriate treatment entails high environmental risks. Moreover, and due to water shortage situations, industries are becoming aware of the need for investing in innovative treatment technologies for water reclamation, such as membrane filtration. This work studies the performance of three commercial ceramic ultrafiltration membranes treating raw effluents from a textile mill. The effect of both pH and molecular weight cut-off (MWCO) on membrane performance was determined while working on concentration mode. Results showed a noticeable influence of both pH and MWCO on process performance. The best results were obtained for the lowest pH tested (8). At higher pH values, higher fouling rates were achieved. On the other hand, higher fluxes were obtained as MWCO was increased but simultaneously, higher rates of membrane fouling were also observed. Permeate flux rate decreased as the feed solution was concentrated. However, this drop was more noticeable for the lower VRF values. The best overall results were obtained for the 50 kDa membrane operating at pH 8. TOC and COD removals up to 67% and 80%, respectively, were reached at these conditions. In the same way, nearly complete color and turbidity removals were achieved for all the membranes and operating conditions studied. Regarding these results, the combined process of MF/UF has been proven to be a feasible pre-treatment in order to reduce wastewater volume and produce a permeate of enough quality to be used as influent in the NF/RO stage.

*Keywords:* Ceramic membranes; Textile wastewater; Ultrafiltration; Water reclamation

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### 1. Introduction

The constant increase in the freshwater demand for a great variety of applications (agricultural, industrial and human) has combined with the growing shortage of this valuable resource in many particular regions worldwide [1]. Both governments and environmental agencies have dealt with this issue by developing

more restrictive regulations which have involved increasing freshwater and wastewater disposal costs. For some industrial sectors, as great water consumers, new approaches in water and wastewater management are required in order to reduce costs. In this way, water reclamation becomes almost essential rather than an interesting alternative [2]. In particular, textile industries and their processes involve a huge consumption of freshwater, being even considered as one of the largest water consumers [3]. Depending on the process, an

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estimated volume between 0.2 to 0.4 m<sup>3</sup>/kg fabric, with peaks above 0.5 m<sup>3</sup>, is required [2,4]. In addition, dyeing and finishing processes consist of a great number of operations which involve the use of different types of dyestuffs and reactive agents such as surfactants, grease and oils, heavy metals, pH adjusting chemicals, inorganic salts and fibers [3]. The unfixed dyes, together with part of the reagents used, are finally disposed within the waste streams [5]. Moreover, these effluents regularly show caustic nature and high temperatures (50–70°C) [1]. Consequently, textile industry generates large volumes of wastewater with an elevated variability and complexity due to its components. The disposal of these effluents without the appropriate treatment entails a high environmental risk since they are strongly colored and show high chemical oxygen demand, salts concentration and pH [6]. Furthermore, some dye groups, mainly azo dyes and their by-products, may be carcinogenic and/or mutagenic [7]. As a result, textile industry might be considered as one of the most polluting industrial sectors [8].

The most widespread methods for treating textile industry effluents include physicochemical and biological treatments. Although these conventional methods allow meeting legislative requirements regarding wastewater disposal, they do not allow wastewater reuse in any step of the productive process. Owing to the pressing need of water reclamation, innovative technologies such as membrane filtration, ozonation, electrochemical oxidation and adsorption, have been lately introduced in the wastewater treatment systems. Among all of them, membrane technologies have emerged as a feasible alternative whether enough water quality to allow the effluent reuse wants to be achieved (Table 1) [4,9]. Due to the continuous research and development, as well as the decrease in the costs of these technologies, their utilization has increased noticeably, becoming one of the recommended treatment methods for textile wastewater reclamation in the Best Available Techniques reference document (BAT) [4]. Membrane technologies are capable of achieving high depuration rates by using

either Nanofiltration (NF) or Reverse Osmosis (RO) processes. Nevertheless, these processes may show a rapid productivity decline as membranes get fouled, resulting in a major drawback inherent to all the membrane processes. In order to prevent membrane fouling, effluents must be subjected to an appropriate pre-treatment. In this way, processes such as Microfiltration (MF) and/or Ultrafiltration (UF) have also been proven as a feasible pre-treatment for later NF/RO stages [2]. At the same time, the most suitable operating conditions have to be found to conduct the process while obtaining the highest productivity. Operating conditions such as crossflow velocity [10], pH [11], transmembrane pressure [12] and characteristics of the membrane such as nominal pore size [13] may exert a significant influence on the final process performance.

In this work, the UF with ceramic membranes of textile effluents after mechanical pre-treatment is evaluated. The suggested complete process would also include a further NF/RO step in order to reduce or eliminate the conductivity and allow the final wastewater reuse. Ceramic membranes show high chemical, mechanical and thermal resistance, which have made them gain some popularity over polymeric membranes [14]. These intrinsic characteristics of ceramic membranes perfectly fit the needs for textile wastewater treatment. Many works have proven the effectiveness of ceramic UF membranes to treat industrial wastewater effluents. However, there is a lack of studies that apply ceramic membranes working on concentration mode in the treatment of industrial wastewater effluents and specifically, in the raw textile effluents treatment. In this way, the aim of this work is to study the influence of pH on the process performance of commercial tubular ceramic UF membranes with different nominal molecular weight cut-off (MWCO) treating and concentrating raw effluents from a textile mill.

## 2. Materials and methods

### 2.1. Wastewater sample

Textile effluents sample was collected directly from a textile mill where mainly dyeing, printing and finishing activities were carried out. On normal operating conditions, these activities require a considerable amount and variety of reactive agents, including dyestuffs, salts, caustic soda and surfactants. At the present time, the wastewater volume generated in the entire mill is estimated to be around 2000 m<sup>3</sup> daily. The treatment of these effluents is carried out in a conventional treatment plant located in the factory to meet the legislative requirements that allow their final disposal into the sewage system. The treatment consists of an

Table 1  
Reference values for water reuse in textile industry

Parameter	Criteria
COD (mg/L)	60–80
Conductivity (µS/cm)	1000
pH	6–8
Turbidity (NTU)	1
Color (Pt-Co)	None
Suspended solids (mg/L)	5
Dissolved solids (mg/L)	500
Total hardness (mg/L as CaCO <sub>3</sub> )	25–50

initial physicochemical stage with a dynamic rotating screen for bigger particles removal. Since the pH is highly variable, it is adjusted just after the first stage and the effluents are then gathered in an equalization tank. The second stage of the treatment involves conducting the effluents to the biological reactor and to the dissolved air flotation tank (DAF). In this tank, treated effluents can be separated from the activated sludge. The sample point of the wastewater used in this study was located preceding the pH adjustment tank, where the most basic conditions may be found (pH close to 12).

The characterization of the wastewater was made according to the most frequently measured parameters for water quality control. In this way, total organic carbon (TOC), chemical oxygen demand (COD), conductivity, pH, turbidity and spectral absorption coefficient (SAC) were initially measured and subsequently monitored.

## 2.2. Analytical methods

Both the TOC and COD measurements were determined photometrically by means of a Spectroquant NOVA 60 photometer (MERCK). The determination of the samples conductivity was conducted using a CM 35 conductivity meter (CRISON) and pH values were determined by means of a GLP 22 pH-meter (CRISON). Both conductivity and pH sensors allowed automatic and continuous correction of the measurements by taking into account the temperature of the samples. Turbidity values were determined with a DINKO D-112 turbidimeter according to the ISO 7027:1999. The SAC was determined by means of absorbance measurements by the spectrophotometric method at three wavelengths

( $\lambda = 436, 525$  and  $620$  nm). The SAC values were obtained by using a HP 8453 spectrophotometer (1 cm cell width) once the samples had been filtered with a  $0.45 \mu\text{m}$  filter according to the ISO 7887:1994. Determination of the suspended solids was carried out gravimetrically according to the ISO 11923:1997.

## 2.3. UF membranes

Fig. 1 shows a scheme of the UF pilot plant, designed in a previous work [15], used to carry out the UF experiments. The pilot plant was equipped with a minifilter module (Tami industries, Nyons, France). The membranes selected for this study were three commercial multichannel tubular ceramic membranes Inside Céram™ (Tami industries, Nyons, France). These membranes are composite elements with a support layer of  $\text{TiO}_2$  and an active layer of  $\text{ZrO}_2\text{-TiO}_2$ . All the membranes tested had a length of 580 mm, an external diameter of 25 mm and were divided into eight channels. The equivalent hydraulic diameter of each channel was 6 mm. According to the manufacturer, the total effective membrane area was calculated as  $0.1 \text{ m}^2$ . Three different MWCO were selected for the execution of the experiments (30, 50 and 150 kDa). Table 2 details the membrane characteristics reported by the manufacturer.

Table 2 also includes the pure water permeabilities of the membranes determined experimentally. In order to obtain these values, the permeate flux rate with deionized water ( $J_{\text{pw}}$ ) at different transmembrane pressures (0.5, 1, 2, 3 and 4 bar, respectively) was obtained. Permeate flux was calculated according to Eq. (1):

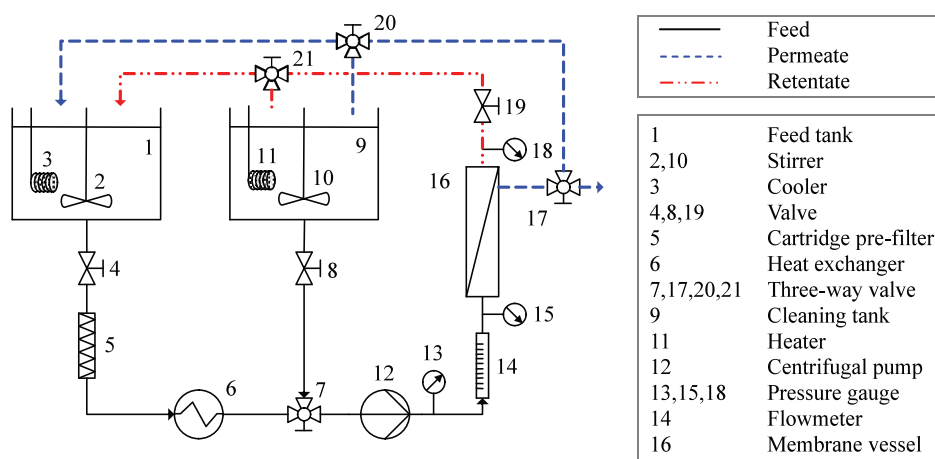


Fig. 1. Scheme of the UF pilot plant.

Table 2  
Ceramic membrane characteristics

Membrane	Inside-Céram™
Manufacturer	Tami Industries
Material	TiO <sub>2</sub> -ZrO <sub>2</sub>
Maximum running pressure (bar)	10
Breaking pressure (bar)	>90
Process temperature (°C)	<300
pH range	0–14
Pure water permeability (L/(m <sup>2</sup> ·h·bar))	30 kDa 50 kDa 150 kDa
Experimental (35°C)	67 155 180

$$J_p = \frac{V_p}{A \cdot t} \quad (1)$$

where  $J_p$  is the permeate flux (L/(m<sup>2</sup>·h)),  $V_p$  is the volume of the sampled permeate (L),  $A$  is the effective membrane area (m<sup>2</sup>) and  $t$  is the collecting time (h). By plotting the permeate flux ( $J_p$ ) versus the transmembrane pressure (TMP) it is possible to calculate the membrane permeability which corresponds to the slope of the obtained straight line.

#### 2.4. Experimental procedure

The wastewater sample coming from the textile mill was initially subjected to a pre-treatment which consisted of two cartridge microfilters in series. This treatment was applied for removing bigger particles and avoiding the rapid membrane clogging. The first filter was a polypropylene pleated cartridge filter (30 μm) whereas the second one was a polypropylene string wound cartridge filter (5 μm). The filtered effluent was then headed to the feed tank, which has a volume of 60 L, located in the UF pilot plant.

According to the conditions of the waste streams at the mill treatment plant, samples were previously heated to 35°C in the feed tank, prior to each experiment, by means of a heating system. A cooler and a heat exchanger were used to keep the temperature constant during the tests. All the experiments were carried out at a crossflow velocity (CFV) of 4 m/s. In this way, the feed flow rate was set according to the manufacturer data (0.9 m<sup>3</sup>/h are needed for a CFV of 1 m/s). The transmembrane pressure was also set at 1 bar taking into account the pressure drop along the membrane at that CFV (0.4 bar). In order to evaluate the effect of the pH on membrane performance, experiments were conducted at three different pH values (8, 10 and 12, respectively), due to the caustic nature of this kind of effluents.

Previously to each experiment, the pH of the feed water was adjusted by adding either HCl or NaOH solutions. Each membrane (30, 50 and 150 kDa) was tested at each particular pH in order to study the influence of operating conditions on a particular MWCO. The filtration experiments were conducted on batch concentration mode, where the retentate was recirculated into the feed tank whereas the permeate stream was stored in a separate tank. The influence of feed concentration may be observed by monitoring the process performance while the volume in the feed tank was concentrated to a final volume of 30 L. To that end, volume reduction factor (VRF) was defined as follows:

$$\text{VRF} = \frac{V_F}{V_C} \quad (2)$$

where  $V_F$  is the initial feed volume and  $V_C$  is the concentrated volume. Each experiment was finished once the VRF reached a value of 2. In order to evaluate the effect of operating conditions (pH, MWCO and VRF) on the final process performance, both the permeate flux rate and permeate quality were monitored. To that purpose, process performance was calculated in terms of the rejection coefficient ( $R$ ). This coefficient was obtained according to Eq. (3):

$$R (\%) = \left( 1 - \frac{C_p}{C_f} \right) \cdot 100 \quad (3)$$

where  $C_f$  and  $C_p$  are the values of the measured parameter in the initial feed wastewater and in the overall permeate stream, respectively.

Subsequently to each experiment, membranes were rinsed with deionized water and then soaked in a 4000 ppm free chlorine solution. Finally, membranes were rinsed again with deionized water until neutrality was reached. Then, permeate flux was measured in order to check the effectiveness of the cleaning protocol. The initial membrane permeability was restored after the chemical cleaning with average flux recoveries higher than 95%.

### 3. Results and discussion

#### 3.1. Effluent characterization

Table 3 shows the obtained results for the raw wastewater effluents characterization according to the aforementioned analytical methods. Even though the experiments were carried out with an only sample, it is clear from the results that by solely modifying the

Table 3  
Textile wastewater characterization

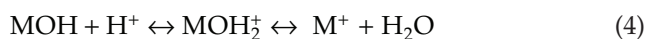
Parameter	pH 8	pH 10	pH 12
COD (mg/L)	3380	2940	3370
TOC (mg/L)	924	1010	1110
Conductivity (mS/cm)	4.1	4.1	6.8
SS (mg/L)	715	594	506
Turbidity (NTU)	243.2	178.5	164.4
SAC (m <sup>-1</sup> )			
436 nm	19.1	17.0	35.3
525 nm	7.3	7.8	16.7
620 nm	4.3	5.5	11.4

pH of the effluent other parameters are also affected. This fact may be attributed to the aforementioned complexity of this kind of effluents together with the appearance of different interactions between the macromolecules in the wastewater. Due to these differences, the results regarding membrane rejection are presented as percentage removal rather than final values.

### 3.2. Effect of pH on membrane flux

In order to determine the fouling rate, the ratio between the wastewater permeate flux ( $J_{ww}$ ) and the pure water permeate flux ( $J_{pw}$ ) was calculated. Fig. 2 shows the relation between the ratio ( $J_{ww}/J_{pw}$ ) versus time for the three membranes tested at each particular pH. The duration of the test was different for each case depending on the time needed to reach a VRF of 2.

To acquire a better understanding of the results, it is important to point out that ceramic membranes can be made of a mixture of diverse mineral oxides (ZrO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> among others). When the membrane surface is put in contact with an aqueous medium, it can be ionized and develop an electrical charge due to the amphoteric behavior of the hydroxyl groups [16]. These groups can be dissociated depending on the pH as:



The effective membrane charge then depends both on the pH of the solution and the isoelectric point (IEP) of the membrane [17]. Consequently, the filtration characteristics of the ceramic membranes are affected by the electrochemical interactions of the surface of the membranes as well as by the steric effects and hydrophobic interactions [16]. In this way, according to the bibliography, the membranes used in

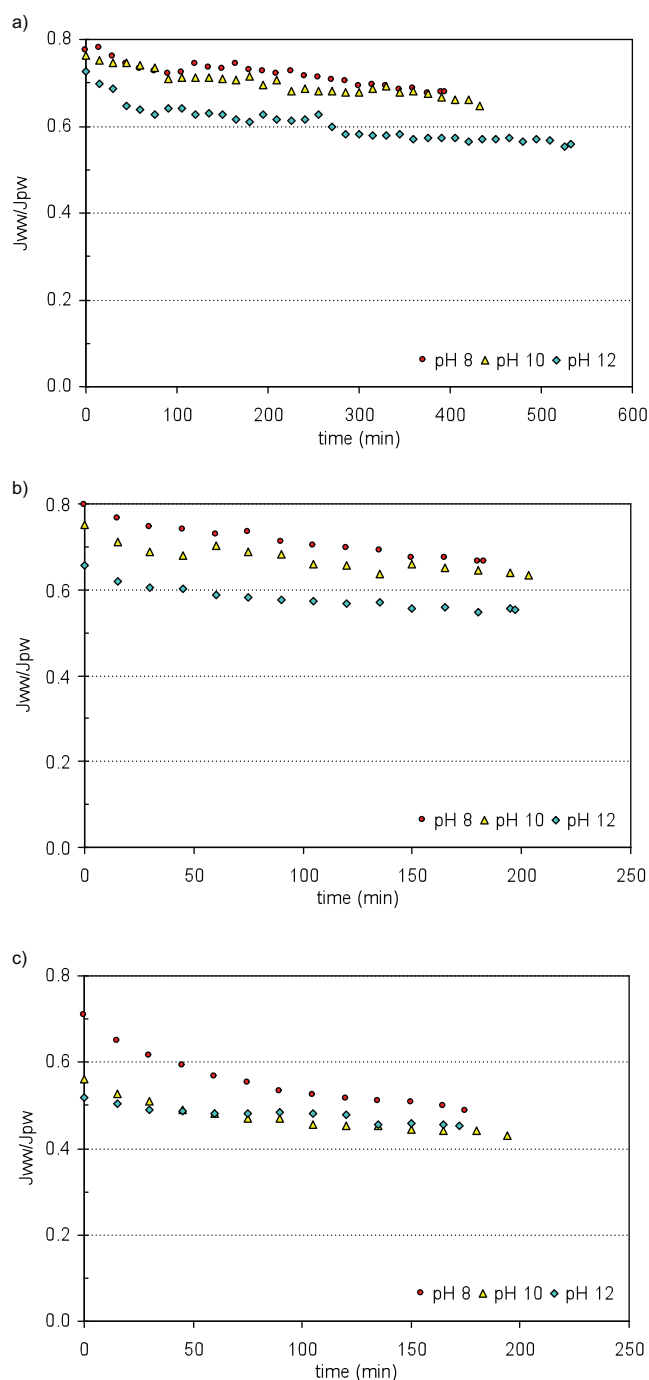


Fig. 2. Ratio  $J_{ww}/J_{pw}$  as a function of time for the three MWCO tested: a) 30, b) 50 and c) 150 kDa.

this study are negatively charged for the whole range of pH studied since the IEP of these membranes is reached at pH values lower than 8 [18]. However, at pH 8 the surface charge becomes less negative since it is closer to the IEP.



According to the results, membrane performance in terms of flux and fouling rate was noticeably influenced by the pH of the effluent. For the three membranes tested, the best overall results were obtained at pH 8 since the flux ratios were 0.68, 0.66 and 0.49 for the 30, 50 and 150 kDa membranes, respectively. As it can be observed in Fig 2, the relation between  $J_{ww}$  and  $J_{pw}$  decreases as pH increases for the three membranes, which implies higher fouling rates at higher pH values. In this way,  $J_{ww}/J_{pw}$  drops from 0.68 at pH 8 to 0.56 at pH 12 for the 30 kDa membrane. This behavior was also observed by other authors and can be attributed to different effects. Kim et al. [19] suggested that a rapid particles precipitation onto the membrane surface may occur for the higher caustic conditions when filtering liquid organic sludge with ceramic membranes. On the other hand, Zhao et al. [20] obtained a decrease of the steady flux from pH 8 to pH 10 filtering  $TiO_2$  suspensions, which was attributed to electroviscous effects.

Due to the nature of the surface active agents (surfactants), which are commonly present in the treated textile wastewater (particularly anionic and non-ionic), it is also needed to consider the effect of these compounds on the filtration performance. These compounds have a hydrophilic and a hydrophobic group and tend to be adsorbed in the interfaces (liquid–solid or liquid–liquid) [21]. Either concentration polarization or adsorption of the surfactant may occur and affect seriously to the process performance [22]. Faibish et al. [23] observed the irreversible adsorption of the surfactant (sodium octanoate) onto the zirconia surface which was attributed to the association between hydroxyl groups present on the zirconia surface at pH higher than the IEP (where both the zirconia surface and the surfactant molecules were negatively charged). Zabkova et al. [24] reported a higher flux decline at higher pH values of the filtered solution that could be mostly attributed to the hydrophobicity of the membrane surface and solute in the ceramic UF membrane used for the recovery of vanillin treating Kraft black liquors. Xu et al. [25] studied the MF of a micro-sized mineral suspension using ceramic membranes. In that work, it is suggested that particles with high surface charges (which can be found at pH values away from the IEP) were well dispersed, due to mutual repulsive electrostatic forces, and formed cakes or fouling layers offering high resistance to filtration, which resulted in low filtrate flux.

### 3.3. Effect of MWCO on membrane flux

Fig. 2 also shows the influence exerted by the membrane MWCO, which in some extent is simultaneously affected by the pH effect. From the obtained results, it

is clear that increasing MWCO leads to higher fluxes, since the time required to reach a VRF of 2 is lower (from an averaged 450 minutes up to 150 minutes for the 30 and 150 kDa membranes, respectively). However, it can be observed that the ratio  $J_{ww}/J_{pw}$  decreases as MWCO is increased, which involves higher rates of membrane fouling. In this way, an increase of the MWCO from 30 to 150 kDa results in a reduction of the ratio  $J_{ww}/J_{pw}$  of a 28% at pH 8 whereas the same increase in MWCO implies a 20% ratio reduction at pH 12. When the nominal pore size increases, initial permeate flux also increases. The higher permeate flux may generate a greater charge displacement [16]. Thus, the electrostatic forces may affect the particle–particle or the particle–surface interactions. The higher initial flux showed by the looser membranes may increase the effect of the concentration polarization layer since concentration polarization is a function of flux [26]. Simultaneously, the higher flux obtained by increasing MWCO may allow that particles accumulate in the proximities of the membrane [27]. These particles might go throughout both the concentration polarization and the cake layer and pass through the pores where adsorption may take place reducing the effective pore radius or even clogging them [28]. Kim et al. [19] reported higher colloidal adsorption for the higher MWCO tested when filtering liquid organic sludge. On the other hand, Gadelle et al. [29] observed a decline in permeate flux along time but reported a higher decrease for the membrane that had the largest pore size, which was attributed to the rapid fouling of the membrane. All these results are in agreement with those obtained in this work.

### 3.4. Effect of concentration on membrane flux

Fig. 3 shows the relation between the ratio  $J_{ww}/J_{pw}$  and the VRF for each membrane at the three pH values tested. As it can be observed from the figure, permeate flux rate decreases as VRF is increased. However, the permeate flux drop seems to be more noticeable for the lower VRF values, which correspond to the initial minutes of the UF process. The results obtained at pH 8 show a lower drop in the ratio for the lowest MWCO (12%) while this drop increases as MWCO is higher (31% for the 150 kDa membrane). On the other hand, the opposite behavior is observed at pH 12. In this case, the highest drop is reported for the 30 kDa membrane (23%), while for the 150 kDa membrane, the ratio  $J_{ww}/J_{pw}$  is reduced in a 13% at a VRF of 2. Beyond a certain VRF value, the process performance tends to the steady-state. In this way, the ratio  $J_{ww}/J_{pw}$  keeps a similar trend for the obtained results beyond a VRF of 1.6 for the three membranes tested at each particular pH.

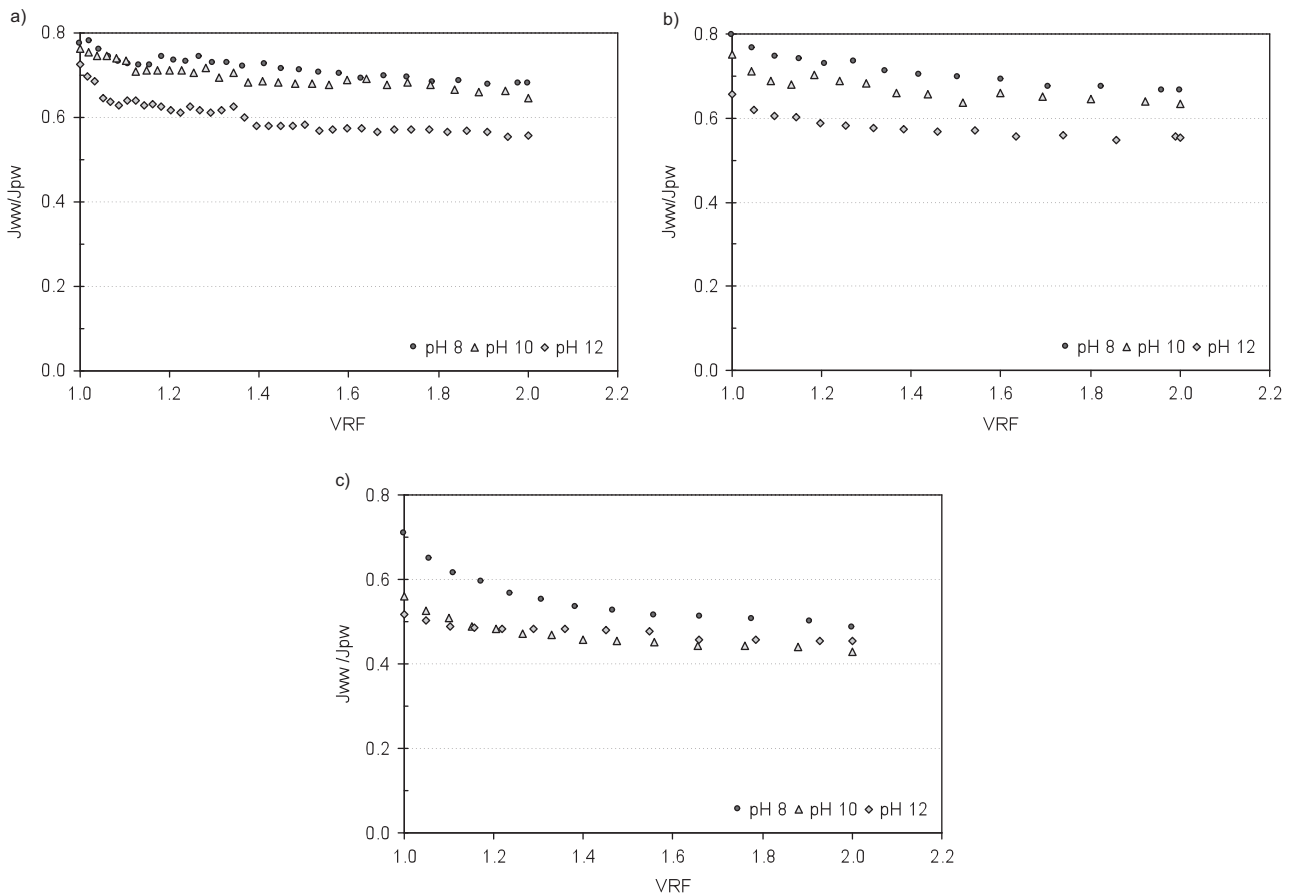


Fig. 3. Evolution of the ratio  $J_{ww}/J_{pw}$  as a function of VRF for the three MWCO tested: a) 30, b) 50 and c) 150 kDa.

### 3.5. Effect of pH and MWCO on membrane rejection

Table 4 shows the total percentage removals obtained once a VRF of 2 was reached. This parameter expresses the rejection results considering the initial feed and the final permeate characterization. As it is clear from the results, the obtained removals are similar regardless the operating conditions of the filtration process

(both for the pH and the MWCO). Nevertheless, it can be observed slightly better overall performances at pH 8. When the pH value is closer to the IEP, the repulsion forces are lower, what allows some of the pollutants to form aggregates or flocs that are better rejected by the membrane.

Table 4  
Total percentage removal values at VRF 2 (%)

Parameter	pH 8			pH 10			pH 12		
	30 kDa	50 kDa	150 kDa	30 kDa	50 kDa	150 kDa	30 kDa	50 kDa	150 kDa
COD	80	78	71	71	73	66	84	72	66
TOC	67	63	63	66	61	61	61	59	55
Turbidity	>99	>99	>99	>99	>99	>99	>99	>99	>99
SAC									
436 nm	98	98	100	97	94	97	99	98	98
525 nm	100	100	100	99	96	100	100	100	97
620 nm	100	100	100	100	97	100	100	100	98

Regarding the MWCO effect on membrane rejection, it is worth pointing out that the best results were obtained for the tightest membrane, as it could be expected. Both the steric effects produced by a smaller pore size as well as the electrochemical interactions may be responsible of this behavior.

According to the obtained results, the use of the tightest membrane would produce the lowest fouling rate. However, the process productivity in terms of permeate flux rate would be significantly lower. In this way, the highest flux for the 30 kDa membrane is 56 (L/(m<sup>2</sup>·h)) whereas the permeate flux rate obtained for the 150 kDa membrane is up to 130 (L/(m<sup>2</sup>·h)). On the other hand, the overall pollutants removal does not show significant improvement by decreasing the MWCO. Taking into account the aforementioned results, the use of the 50 kDa membrane at pH 8 might be the more appropriate choice in terms of permeate flux (120 (L/(m<sup>2</sup>·h))), fouling ( $J_{ww}/J_{pw} = 0.66$ ) and overall pollutants removal.

Regarding the removals obtained for the diverse parameters monitored, the combined process of MF/UF has been proven to be a feasible pre-treatment in order to reduce wastewater volume by concentrating it to some extent and producing a permeate of enough quality to be used as NF/RO stage influent.

#### 4. Conclusions

In this work, UF process with ceramic membranes is applied as a pre-treatment of a raw textile effluent. In order to study the feasibility of the process, the influence of effluent pH, MWCO and volume reduction factor were determined. The major conclusions obtained from this work are presented below.

The pH of the solution shows a significant influence on the fouling rate of the membrane filtration process. The highest ratio  $J_{ww}/J_{pw}$  is obtained at pH 8, and it decreases as the solution pH moves away from the IEP of the membrane. The MWCO also exerts a noticeable influence on the process performance. In this way, an increase in the MWCO from 30 to 50 kDa may double the permeate flux rate with no significant increase in the fouling rate ( $J_{ww}/J_{pw} = 0.66$ ). On the other hand, an increase in the MWCO up to 150 kDa produces a slightly increase in the permeate flux rate at the expense of a considerable increase in the fouling rate ( $J_{ww}/J_{pw} = 0.45$ ).

Nearly complete color (>97%) and turbidity (>99%) removals were achieved after reaching a VRF value of 2 for the three MWCOs at the three pH tested. The highest removal is obtained for the tightest membrane at each particular pH. Moreover, at pH 8 the pollutant rejection is the best overall.

For the raw textile effluents treated, the best overall results were obtained for the 50 kDa membrane operating at pH 8. Lower MWCO does not significantly improve permeate quality and involve half permeate flux rate. Furthermore, increasing the pH value produces higher fouling rate.

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