Influence of operating conditions on ceramic ultrafiltration membrane performance when treating textile effluents

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ABSTRACT

This work studies the performance of three commercial ceramic ultrafiltration membranes (ZrO_2–TiO_2) treating raw effluent from a textile industry. The effect of crossflow velocity at 3, 4 and 5 m s^{-1} as well as membrane characteristics, such as molecular weight cut-off (30, 50 and 150 kDa), on process performance were studied. Experiments were carried out in concentration mode in order to observe the effect of volume reduction factor simultaneously. Results showed a combined influence of both crossflow velocity and molecular weight cut-off on flux performance. TOC and COD removals up to 70% and 84% respectively were reached. On the other hand, almost complete color (>97%) and turbidity (>99%) removals were achieved for all the membranes and operating conditions.

Key words | ceramic membranes, crossflow velocity, textile wastewater, ultrafiltration

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NOMENCLATURE

μ	dynamic viscosity (Pa s)
ΔP	transmembrane pressure (Pa)
C_F	feed concentration
CFV	crossflow velocity (m s^{-1})
COD	chemical oxygen demand (ppm)
C_P	permeate concentration
J_W	permeate water flux (m^3 m^{-2} s^{-1})
MWCO	molecular weight cut-off (kDa)
R	rejection coefficient
R_{cl}	cake layer resistance (m^{-1})
R_{cp}	concentration polarization resistance (m^{-1})
R_{if}	internal fouling resistance (m^{-1})
R_m	hydraulic resistance (m^{-1})
R_{of}	overall fouling resistance (m^{-1})
R_T	total resistance (m^{-1})
SAC	spectral absorption coefficient (m^{-1})
SS	suspended solids (ppm)
TOC	total organic carbon (ppm)
V_C	concentrate volume (m^3)
V_F	initial feed volume (m^3)
VRF	volume reduction factor

INTRODUCTION

Textile industries consume significant amounts of freshwater during dyeing, sizing and finishing processes, as an estimated volume of 20 up to 500 m^3 ton^{-1} of finished product may be needed (Capar *et al.* 2008; Amar *et al.* 2009). Additionally, these processes involve the use of numerous and different types of chemicals. Therefore, textile industry generates large volumes of wastewater with a great chemical complexity, which typically consists of unfixed dyes, detergents, grease and oils, surfactants, heavy metals, pH adjusting chemicals, inorganic salts and fibers (Wijetunga *et al.* 2010). Moreover, these effluents often show a caustic nature and high temperatures (50–70 °C) (Allègre *et al.* 2006). The disposal of these effluents without the appropriate treatment may cause huge environmental damage, not

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only because of their color, but also due to the fact that some dye groups might be carcinogenic and/or mutagenic (Capar et al. 2006). Therefore, the textile industry can be considered as one of the most polluting among all industrial sectors (Koyuncu & Topacik 2003).

Conventional treatment methods for textile effluents usually combine physicochemical and biological processes (Amar et al. 2009). Although it is possible to meet discharge standards by means of these treatments, they are not enough to enable wastewater reuse. Consequently, innovative technologies such as membrane filtration (Koyuncu et al. 2001; Myung et al. 2005; Dogan et al. 2010), ozonation (Brik et al. 2004), electrochemical oxidation (Dogan et al. 2010) and adsorption (Harrelkas et al. 2009) could be used in order to meet the reference water quality values to allow its reuse (Capar et al. 2008). Membrane technologies have been proven as a viable alternative to the conventional treatment processes as well as for water and reagent reclamation purposes in the textile industry (Allègre et al. 2006; Simonic & Lobnik 2011). In this way, a large number of studies have been carried out related to textile effluent treatment by means of either membrane processes (Koyuncu et al. 2001) or hybrid processes, combining them with additional treatments (Rozzi et al. 2000; Lubello et al. 2007; Dogan et al. 2010). Regardless of the effluent quality, process performance and productivity decrease during filtration due to membrane fouling.

In order to limit fouling to some extent, effluents must be subjected to a suitable pre-treatment. Membrane technologies also provide successful alternatives for these pre-treatments as loose membrane processes such as ultrafiltration (UF) may replace conventional clarification methods (Lee et al. 2009). Regarding membrane treatments, limited literature treating mixed raw effluents can be found (Harrelkas et al. 2009; Lee et al. 2009; Yigit et al. 2009; Simonic & Lobnik 2011), as most of the studies deal with either segregated effluents (Orhon et al. 2001), pre-treated (Lu et al. 2010; Qi et al. 2011) or simulated/synthetic waste streams (Sójka-Ledakowicz et al. 2010; Srisukphun et al. 2010). What is more, ceramic membranes have gained popularity due to their better mechanical, thermal and chemical stability over polymeric membranes (Le et al. 2009). Numerous works have proven the effectiveness of ceramic ultrafiltration membranes treating industrial wastewaters (Fan et al. 2000; Majewska-Nowak 2010). However, there is a lack of studies that consider its application in the treatment and concentration of industrial wastewater effluents (Trägardh & Johansson 1998; Lastra et al. 2004), and specifically in the mixed raw textile effluents treatment. In addition, it is well known that crossflow velocity (CFV) is an important factor in the membrane processes (Ahmad et al. 2005; Lobo et al. 2006). In this way, the aim of this work is to study the effect of CFV on the performance of three commercial ceramic ultrafiltration membranes treating raw textile effluents in batch concentration mode.

METHODS

Water samples

The wastewater effluents were supplied from a textile factory which performs dyeing and finishing activities. The normal operation of the different processes generates a considerable volume of effluents (1,750–2,000 m^3 day^{-1}) with extreme variable characteristics. These effluents are treated in a treatment plant inside the factory described elsewhere (Barredo-Damas et al. 2010) prior to being discharged into a sewage system in order to meet legislative requirements.

Some of the most frequently used parameters in wastewater characterization have been selected to monitor the process performance. These parameters are total organic carbon (TOC), chemical oxygen demand (COD), conductivity, pH, turbidity and color.

Analytical methods

Both TOC and COD values determination was conducted using a Spectroquant NOVA 60 photometer (MERCK). Color intensity in terms of SAC was determined by means of absorbance measurements at three wavelengths (436, 525 and 620 nm) by UV–visible absorption with a HP 8453 spectrophotometer (1 cm cell width) after the samples were filtered with a 0.45 μm filter, in accordance with ISO 7887:1994. Turbidity was measured with a DINKO D-112 turbidimeter according to ISO 7027:1999. The conductivity analyses were made using a CM 35 CRISON conductivity-meter and pH values were determined by means of a GLP 22 CRISON pH-meter. Determination of suspended solids was carried out gravimetrically according to ISO 11923:1997.

Ultrafiltration

For the ultrafiltration tests' execution, three commercial multichannel tubular ceramic membranes Inside Céram™ (TAMI Industries, France) were selected. The experiments were carried out in a pilot plant described in previous work (Barredo-Damas et al. 2010). The membranes tested were composite elements with a support layer (TiO_2) and an active layer (ZrO_2–TiO_2). The membrane dimensions were 580 mm long, with an external diameter of 25 mm and eight channels. Each channel had a hydraulic diameter of 6 mm, which meant a total effective filtration area of 0.1 m^2. The molecular weight cut-off (MWCO) values were 30, 50 and 150 kDa, respectively. Pure water permeability was determined experimentally at 35 °C, which is the temperature of the tests. The experimental values were 1.86×10^{-10}, 4.32×10^{-10} and 4.98×10^{-10} $m^3\,m^{-2}\,s^{-1}\,Pa^{-1}$, for the 30, 50 and 150 kDa membranes, respectively.

Experimental procedure

Initially, samples were subjected to a pre-treatment consisting of two cartridge microfilters in series (30 and 5 μm). The filtered effluent was then headed to the feed tank where it was used as influent of the UF experiments.

Samples were previously heated to 35 °C, simulating the conditions of the waste streams at the factory, and kept constant afterwards. Experiments were conducted at three different CFV (3, 4 and 5 $m\,s^{-1}$, respectively). The feed flow rate was set according to the desired CFV on the membrane surface and to the manufacturer data (0.9 $m^3\,h^{-1}$ needed for 1 $m\,s^{-1}$). Simultaneously, the influence of the molecular weight cut-off was also studied and experiments were performed with three different MWCO (30, 50 and 150 kDa). The transmembrane pressure was set to 0.1 MPa taking into account the pressure drop along the membrane vessel, which increases with CFV (from 0.03 MPa at 3 $m\,s^{-1}$ to 0.06 MPa at 5 $m\,s^{-1}$). The filtration experiments were carried out in concentration mode, where the retentate was recirculated into the feed tank and the permeate stream was stored in a separate tank. Thus, the volume reduction factor (VRF) was defined according to Equation (1):

$$\mathrm{VRF} = \frac{V_F}{V_C} \quad (1)$$

where V_F is the initial feed volume and V_C is the concentrate volume. In order to evaluate the effect of the operating conditions on the process as well as membrane fouling, both the total membrane resistance and permeate quality were monitored. The overall total membrane resistance (R_T) may be calculated according to the series resistance equation Equation (2):

$$J_W = \frac{\Delta P}{\mu R_T} = \frac{\Delta P}{\mu(R_m + R_{of})} = \frac{\Delta P}{\mu(R_m + R_{cp} + R_{cl} + R_{if})} \quad (2)$$

where J_W is the permeate flux rate, μ is the dynamic viscosity of the permeate, ΔP is the transmembrane pressure, and R_T, R_m, R_{of}, R_{cp}, R_{cl} and R_{if} are the overall total resistance, the hydraulic resistance of the clean membrane, the overall (total) fouling resistance which includes the concentration polarization resistance, the cake layer resistance and the internal fouling resistance. In this way, R_T was obtained from flux profiles. On the other hand, R_m can be easily measured by filtering deionized water through an unused membrane subsequent to the preparation procedure reported by the manufacturer, as no other resistance is present in the process (7.491×10^{12}, 3.220×10^{12} and 2.789×10^{12} m^{-1} for 30, 50 and 150 kDa, respectively). R_{of} may be numerically calculated as the difference between the total resistance and the hydraulic resistance.

Process performance was calculated in terms of percentage rejection and rejection coefficient (R) according to Equation (3):

$$R\,(\%) = \left(1 - \frac{C_P}{C_F}\right)100 \quad (3)$$

where C_F and C_P are the values of the measured parameters in the initial feed (wastewater) and in the overall permeate stream, respectively.

After each experiment, membranes were rinsed with deionized water and soaked in a 4,000 ppm free chlorine solution. Then they were rinsed again with deionized water until neutrality was reached. The initial membrane permeability was restored after the cleaning protocol with average flux recoveries higher than 95%.

RESULTS AND DISCUSSION

Effluent characterization

According to the described analytical methods, wastewater samples used in this study were initially characterized (Table 1). Due to the variability of the effluents, the obtained results showed a wide range of values for the monitored parameters.

Effect of CFV on membrane performance

According to the results, membrane performance in terms of membrane fouling was significantly influenced by the effect of CFV. Figure 1 shows the relationship between R_T versus time for the three MWCO tested at each CFV. Moreover,

Table 1 | Textile wastewater characterization

Parameter	Range
COD (ppm)	3,100–4,625
TOC (ppm)	978–1193
pH	11.9–12.3
Conductivity (mS cm^{-1})	6.38–9.04
SS (ppm)	506–558
Turbidity (NTU)	159.9–266.8
SAC (m^{-1})	
436 nm	26.3–44.2
525 nm	11.8–21.7
620 nm	7.8–15.0

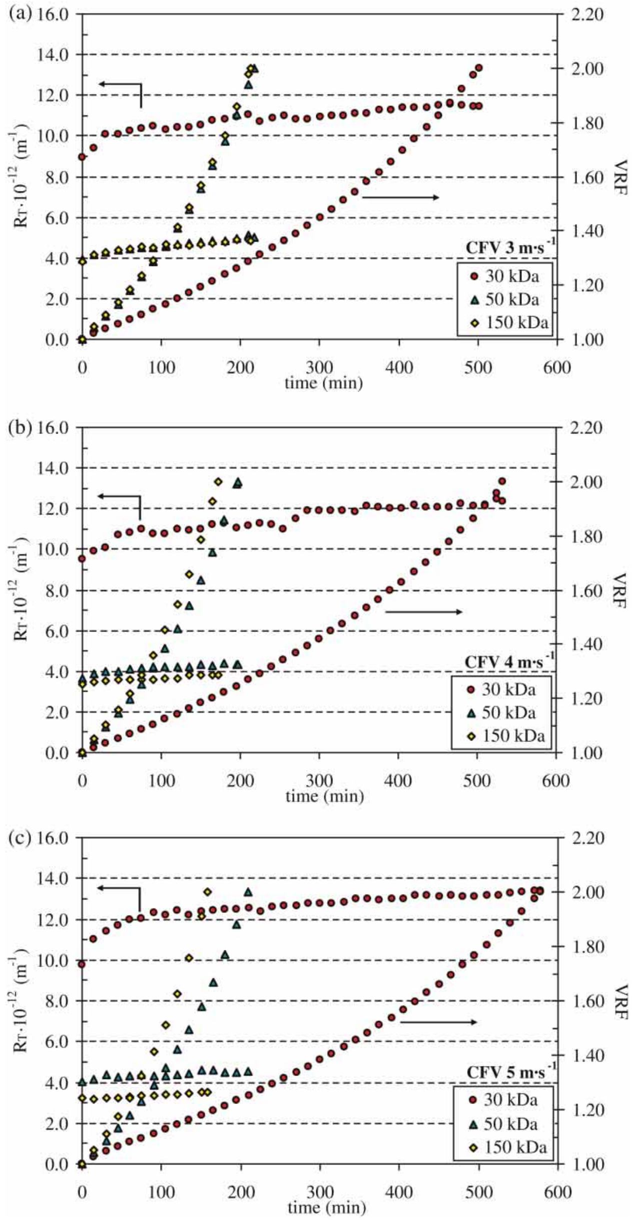

Figure 1 | Total resistance and VRF as a function of time and MWCO for the three CFVs tested: (a) 3 m s^{-1}, (b) 4 m s^{-1} and (c) 5 m s^{-1}.

Figure 1 also shows the evolution of VRF with time. However, a clear effect of the CFV cannot be described as the results seem to be dependent or a combination of the MWCO and CFV. As can be observed, for the tightest membrane (30 kDa), R_T increases 17%, from 1.146×10^{13} ($J_W = 1.213 \times 10^{-5}$ m^3 m^{-2} s^{-1}) to 1.337×10^{13} m^{-1} ($J_W = 1.040 \times 10^{-5}$ m^3 m^{-2} s^{-1}), as CFV is increased from 3 to 5 m s^{-1}. This behavior might be attributed to the presence of two simultaneous competing factors. On the one hand, at higher CFV, the cake layer formation is limited due to higher shear forces which carry deposited particles back to the bulk solution. Furthermore, these forces might also cause changes in the foulant/membrane interactions and remove larger particles at a higher rate. In this way, a selective deposition could take place, where only smaller macromolecules and colloid particles accumulate in the surface vicinity, forming a more compact cake layer (Li *et al.* 2007; Xu *et al.* 2010). By reducing the cake layer thickness, as well as particle size, a higher number of these finer particles may accumulate near the membrane surface and pass through the membrane. Therefore, the plugging/blockage of a higher number of pores and internal fouling may occur (Zhao *et al.* 2003). In this way, this increase in R_T by increasing CFV may be attributed to the increase of the internal and cake layer resistance for the tightest membrane. These authors also observed that beyond a CFV (6.5 m s^{-1}) the R_T increased. In their research, asymmetric α-Al_2O_3 membranes with a mean pore size of 0.8 μm were used to remove hydrate TiO_2 in waste acid.

Should the MWCO be increased to 50 kDa, a completely different result is observed. In this case, the lowest R_T was observed at 4 m s^{-1}, with a value of 4.335×10^{12} m^{-1} ($J_W = 3.207 \times 10^{-5}$ m^3 m^{-2} s^{-1}). At lower CFV, the cake layer becomes thicker, which means higher R_T, whereas higher CFV may lead to a predominant role of the internal fouling and cake layer compression due to finer particles deposition over shear forces influence. These results are in agreement with previous studies (Laitinen *et al.* 2001), which observed an optimum CFV using ceramic ultrafiltration membranes above which the process performance decreased. Xu *et al.* (2010) noticed similar behavior using a tubular ceramic membrane with a pore size of 0.05 μm in pretreatment of seawater desalination. Their research denoted an optimum CFV value at 3.7 m s^{-1} with a steady-state flux of 1.231×10^{-4} m^3

$m^{-2}\,s^{-1}$ at 0.14 MPa and 28 °C. Beyond that CFV a small drop in the permeate flux was observed.

Finally, for the loosest membrane (150 kDa), the CFV showed the opposite effect. In that way, increasing CFV from 3 to 5 $m\,s^{-1}$ reduced R_T from 4.845×10^{12} ($J_W = 2.869 \times 10^{-5}\,m^3\,m^{-2}\,s^{-1}$) to $3.540 \times 10^{12}\,m^{-1}$ ($J_W = 3.927 \times 10^{-5}\,m^3\,m^{-2}\,s^{-1}$). The higher shear forces induced by increasing CFV are enough to avoid or reduce the cake layer thickness; thus, reducing R_T. Moreover, due to the looser pores, the smaller particles forming the cake layer may pass through the membrane without plugging or blocking its pores. These results are in agreement with other works. In particular, Lobo *et al.* (2006) observed higher flux rates by increasing CFV treating oil-in-water emulsions with inorganic membranes. This effect was attributed mainly to the increase in turbulence near the membrane surface. In their study, the highest CFV tested (CFV = 4.2 $m\,s^{-1}$) showed flux rates below 1.111×10^{-5} and $2.778 \times 10^{-5}\,m^3\,m^{-2}\,s^{-1}$ for the 50 and 300 kDa membranes, respectively, at 0.1 MPa and 20 °C. Likewise, Ahmad *et al.* (2005) obtained an increase in the steady-state flux when increasing CFV. In their research, a palm oil mill effluent was treated with a 10 kDa ceramic membrane, with flux rates close to $1.111 \times 10^{-5}\,m^3\,m^{-2}\,s^{-1}$ at 0.1 MPa and 30 °C.

Effect of MWCO on membrane performance

From Figure 1 can also be observed the influence exerted by the membrane mean pore size, although it was also dependent on CFV. It is clear that increasing MWCO led to lesser overall total resistances. Nevertheless, the differences between membranes tended to be reduced as CFV decreased. It is highlighted that for the lowest CFV tested, both 50 and 150 kDa membranes showed the same behavior, as they presented nearly the same R_T (4.845×10^{12} and $5.037 \times 10^{12}\,m^{-1}$, respectively). This result is associated with the higher fouling of the 150 kDa membrane which evens the results with those obtained for the 50 kDa membrane. By increasing the pore size, the capillary pressure would be reduced and consequently, high MWCO membranes are more prone to pore blocking (Lobo *et al.* 2006). Performance of the 150 kDa membrane was enhanced as CFV increased; thus differing from the 50 kDa membrane (from 3.540×10^{12} to 4.536×10^{12} at 5 $m\,s^{-1}$, respectively).

Effect of concentration on membrane performance

Figure 2 shows the relationship between R_T and VRF for each particular membrane at the three CFVs tested. The influence of this factor is noticeable, but it is also determined by both the CFV and MWCO. As can be observed, R_T increases with VRF. Nevertheless, these resistance values seem to reach a steady value after a certain VRF value. For a certain mean pore size, this VRF value increases as CFV diminishes. In this way, it can be lower than 1.2 for the three membranes at the highest CFV, whereas it is not below 1.6 for the lowest CFV tested. For a given CFV, the influence of VRF is higher as MWCO decreases. At 5 $m\,s^{-1}$, the R_T increased about 10 and 13% for the 150 and 50 kDa membranes, respectively,

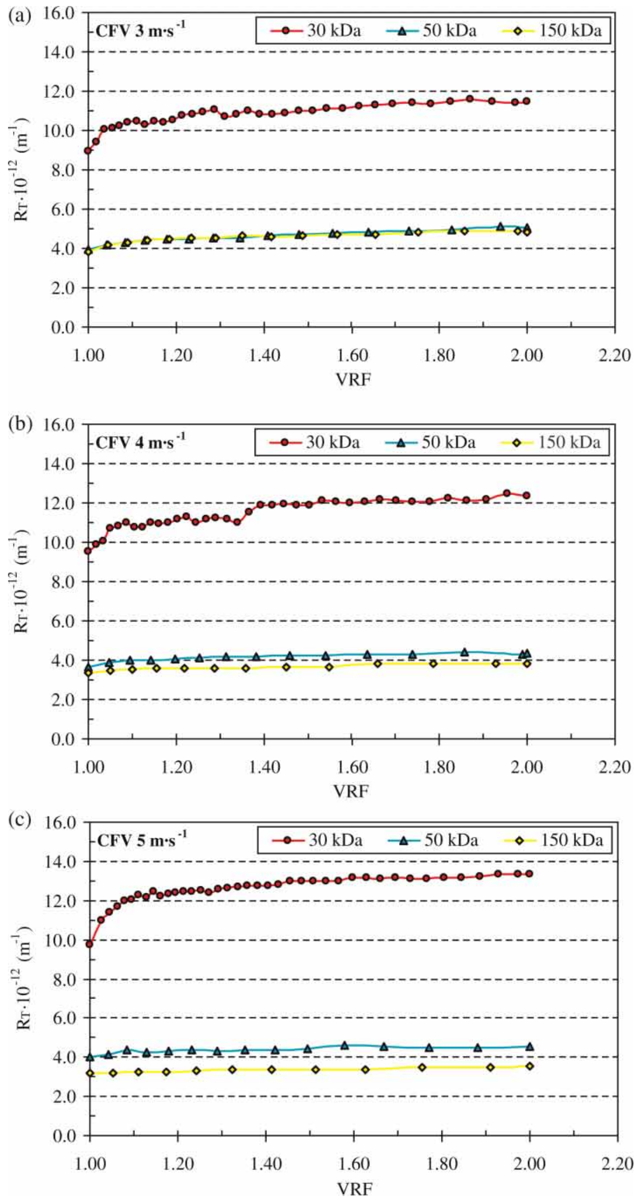

Figure 2 | Evolution of R_T as a function of VRF and MWCO for the three CFVs tested: (a) 3 $m\,s^{-1}$, (b) 4 $m\,s^{-1}$ and (c) 5 $m\,s^{-1}$.

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

Parameter	30 kDa 3 m s^{-1}	4 m s^{-1}	5 m s^{-1}	50 kDa 3 m s^{-1}	4 m s^{-1}	5 m s^{-1}	150 kDa 3 m s^{-1}	4 m s^{-1}	5 m s^{-1}
COD	82	84	79	72	72	77	65	65	63
TOC	70	63	60	68	61	63	67	57	58
Turbidity	>99	>99	>99	>99	>99	>99	>99	>99	>99
SAC									
436 nm	100	99	99	98	98	98	98	98	98
525 nm	100	100	100	99	100	100	99	97	100
620 nm	100	100	100	99	100	100	100	98	100

when VRF reached a value of 2. On the other hand, the increase for the 30 kDa membrane was around 37% at the same VRF value.

Effect of CFV and MWCO on membrane rejection

Table 2 indicates the total percentage removals obtained comparing the initial feed wastewater and the overall obtained permeate once the process reached a VRF value of 2. Table 2 shows that comparable removals were obtained regardless of the operating conditions (CFV and MWCO) of the process. Slightly higher removals were obtained for the 30 kDa membrane. The highest TOC removals were obtained at the lowest CFV tested for each particular membrane. This fact may be attributed to the higher permeate flux rate passing throughout the membrane for the highest CFV, which entails lower retention coefficients. While some differences are observed in the retention percentages of COD obtained for the different MWCO tested, no significant differences were noticed for the other measured parameters. Concerning the removals obtained for the monitored parameters, the combined process of MF/UF has proven to be a feasible pre-treatment in order to reduce wastewater volume and produce a permeate of enough quality to be used as the NF/RO stage influent in spite of the remaining COD and TOC values. In this way, previous studies have confirmed the ability of NF membranes to treat effluents with higher COD values, reducing this parameter from up to 2,000 ppm down to approximately 400 ppm in a colored effluent (De Vreese & Van der Bruggen 2007). Likewise, good results were also observed by Paraskeva *et al.* (2007) using RO membranes for olive mill wastewater fractionation in spite of its higher TOC values. In their work, TOC removals up to 99% from a feed stream with a TOC concentration up to 11,000 ppm were achieved.

According to the obtained results, the use of the tightest membrane would reduce process performance in terms of permeate flux and would not improve significantly the final effluent quality. On the other hand, the lowest ratio R_{of}/R_T was obtained for the 150 kDa membrane at 5 m s^{-1} (0.21), which means lower increase of R_T from the initial value. Nevertheless, the TOC and COD rejections for these operating conditions were the lowest. Therefore, the use of the 50 kDa membrane might be more appropriate in terms of both water quality and fouling ($R_{of}/R_T = 0.26$ at 4 m s^{-1}). In the same way, it would not be necessary to increase CFV higher than 4 m s^{-1} as it would not entail a process improvement.

CONCLUSIONS

In the treatment of raw textile effluents by tubular UF ceramic membranes, the influence exerted by the CFV differs depending on the different MWCOs used. The increase of CFV enhances the filtration process for the loosest MWCO. Similar results were obtained by other research groups (Ahmad *et al.* 2005; Lobo *et al.* 2006). On the contrary, when the MWCO tested is reduced to 50 kDa, an optimum CFV is observed (4 m s^{-1}), above which process performance decreased. When the tightest membrane (30 kDa) is tested, an increase in CFV produces an increase in the overall total resistance (R_T). This higher resistance may be attributed to a higher internal and cake layer fouling due to a selective deposition of smaller particles. Likewise, Zhao *et al.* (2003) observed that beyond a certain CFV value the R_T increased.

Almost complete color (>97%) and turbidity (>99%) removals were achieved after reaching a VRF value of 2 for the three molecular weight cut-offs at the three CFVs tested. No significant influence of the analyzed variables was observed for these parameters. The highest COD and TOC removals were obtained for the tightest membrane (up to 84 and 70%, respectively). The obtained yield in terms of organic matter percentage removal is close or even higher than that obtained by other treatments such as a combination of biological and membrane treatments (Yigit *et al*. 2009; Lu *et al*. 2010) or coagulation–flocculation coupled with UF membranes (Harrelkas *et al*. 2009).

For the analyzed raw textile effluents, the best overall results were obtained for the 50 kDa membrane operating at 4 m s^{-1}. A lower MWCO does not improve permeate quality significantly and higher CFV does not entail higher permeate flux either.

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