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Surfactants role in the enhancement of the treatment efficiency of the dyeing effluents with combination of membrane processes

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ABSTRACT

The industrial process of reactive dyeing of cotton is a sequence of several operations. Different types of surfactants were used several times during the dyeing process; anionic, nonionic, and cationic. The dyeing effluent is heavily charged with pollution especially color and salt. In a treatment at source approach, the dyeing effluent was isolated and then mixed with the other baths containing surfactants. A combined treatment involving microfiltration (MF), as pretreatment followed by nanofiltration (NF) as mean treatment, was investigated. The mixing of the dyeing bath with another effluent showed 100% of MF flux improvement, but the color retention still higher for the single dyeing bath. The NF treatment produces a completely discolored effluent with 100% of suspended solids retention, while the chlorides removal did not exceed 50%.

Keywords: Reactive dye; Surfactants; Microfiltration; Nanofiltration; Color removal; Chlorides retention

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1. Introduction

Textile industry is not only a big consumer of fresh water but also it uses huge amounts of complex chemical substances. Reactive dyes, due to their fixation characteristics and chemical degradation resistance, are widely used for cellulose as well as for cotton dyeing. Allègre et al. [1] cited that 1 kg of cotton requires 70–150 l of fresh water, 0.6 kg of NaCl and 40 g of reactive dye. As consequence, huge volumes of extremely polluted wastewaters are generated during various processing stages. Direct discharge of these effluents into the environment causes irreversible ecological problems, also increasing restrictive legislations referred to final disposal into the main sewage network, make industrial wastewater treatment a necessity an urgent need.

The current trends in the treatment of textile wastewater in Tunisia include conventional activated sludge and coagulation/flocculation. However, those systems require the input of wide range of chemicals, need an important installation space and poorly remove the widely used reactive dye [2]. So, there is

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an urgent need to develop more efficient and inexpensive methods that require fewer chemicals and energy consumptions and less installation spaces. Membrane technology gained a wider acceptance and became a promising technology that responds to the economical and space constraints.

The membrane processes that meet the legislative requirements are nanofiltration (NF) and reverse osmosis (RO), since they are able to retain small organic compounds as well as dye molecules [3]. However, a comparison between NF and RO showed that the permeate flux of NF was greater, while the energy consumption was less [4]. Because of the pore size of NF membrane (almost 10^{-9} m) as well as the substances properties in the dyeing effluents including important amounts of hydrolyzed dye, salt, suspended solids and auxiliaries, membrane fouling represents the major limitation for the use of single NF treatment. Fouling is susceptible to damage the membrane, to reduce its life time and leads to high operational costs [5]. To prevent membrane fouling and to enhance NF performances, feed pretreatment was necessary. Microfiltration (MF) is a membrane separation technique that can assume the prefiltration role with taking into account high-pollution removal and space constrain. Ellouze et al. [6] observed that the use of MF as pretreatment for NF when compared with C/F gives the best performances regarding to the retention of color (96.2% for MF and 92.8% for C/F) and salt (26% for MF and 15.7% for C/F). Rozzi et al. [7] found that in most cases, COD retention was near 70%, and color removal was more than 95% when MF is coupled with NF.

The dyeing bath compared with the other effluents from the different operations of the reactive dyeing cycle is the most charged with pollution, and it contains hydrolyzed reactive dyes and important amounts of salts and auxiliaries. In order to prevent this heavily colored effluent from contaminating the overall wastewater, treatment at source seems to be a good alternative. In this treatment scheme, the dyeing effluent is isolated, and then, it can be mixed with other effluents in order to be diluted and in an attempt to have favorable reactions between different baths components [8]. In this context, the relation between the use of surfactant and the enhancement of dye removal from textile wastewater was the goal of many studies. Kartal et al. [9] investigated the possible interactions between anionic or nonionic surfactant with anionic reactive dye and found that while nonionic surfactant formed a complex with the dye molecule, the anionic one did not; also, it was concluded that the use of two surfactants simultaneously inhibited the color removal due to the change of hydrophobic–hydrophilic balance. In the same context, Akbas et al. [10] studied the interactions between cationic or nonionic surfactant and anionic reactive dye; they found that the stability of the complex cationic surfactant–anionic dye was reduced when a nonionic surfactant was added. Zaghbani et al. [11] found that the use of anionic surfactants with cationic dyes enhanced the color rejection by an UF membrane which exceeded 97%.

NF membranes are generally negatively charged [12]. In this case, electrostatic interactions drive the membrane behavior with charged particles. Peeters et al. [13] found that salts rejection with negatively charged NF membrane is controlled by Donnan effect that is a potential difference created at the interface membrane solution to counteract the transport of anions through the membrane negatively charged.

The purpose of this study was to investigate the filtration behavior of MF and NF membranes in the treatment of real reactive dyeing wastewater in the presence of surfactants coming from the different effluents of the dyeing cycle. The possible interactions between the different species such as anionic reactive dye molecules and anionic, cationic, and nonionic surfactants and the impact of the different baths combinations on the filtration behavior, especially color removal by MF were studied. The efficiency of NF in salts removal especially chlorides was studied taking into account interactions between membrane and pollutants.

2. Materials and methods

2.1. Dyeing cycle and baths mixing

The effluents that constitute the object of this study were collected from a dyeing machine of cotton using reactive dyes. Data regarding the different operations, which constitute the dyeing cycle, the operating conditions, and auxiliaries added in each step, were collected in Table 1. It has to be noticed that after each operation, the effluent was drained out, and fresh water was used in the next step. The volume of water used in the process depends on the material weight; 1 kg of cotton requires about 10 l of fresh water in each bath, and therefore, the same volume of water is used in each operation.

Surfactants were used three times in the dyeing cycle, anionic surfactant in the preparation bath, nonionic in washing and cationic in softening. In order to study the impact of surfactants on the treatment of the dyeing bath using membrane processes, different mixtures were realized: The first three configurations

Table 1
Operating conditions of the dyeing process steps

Operation	pH	*T* (°C)	*t*(min)	Additives
Preparation (*P*)	6–7	30	5	Anionic surfactant Acetic acid
Dyeing (*D*)	9–11	60	110	Reactive dyes (anionic) Sodium chloride Sodium carbonate Sodium hydroxide
Neutralizing (*N*)	6–7	50	5	Acetic acid
Washing (*W*)	6–7	80	5	Nonionic surfactant
Softening (*S*)	5–6	40	20	Acetic acid Cationic surfactant

were obtained by mixing the dyeing bath with one other bath ($D+P$, $D+W$, $D+S$), and then, to study the effect of more than one surfactant on the treatment behavior, the configurations $D+P+W$, $D+P+S$, and $D+W+S$ were realized. In order to highlight the surfactants contribution on the treatment, the dyeing bath was treated separately.

2.2. Combined treatment

In the coupled treatment, MF and NF membranes were connected. The MF permeate was fed to the NF membrane. The concentrates of MF and NF are gathered and fed back into the MF feed tank (Fig. 1).

For the MF experiments, Membralox module (1P19-40/1R19-40) of 1,020 mm length was used. The membrane is a multichannel type (19 channels) made of porous ceramic based on alumina with an area of 0.24 m^2 and mean pore size of 0.2 μm.

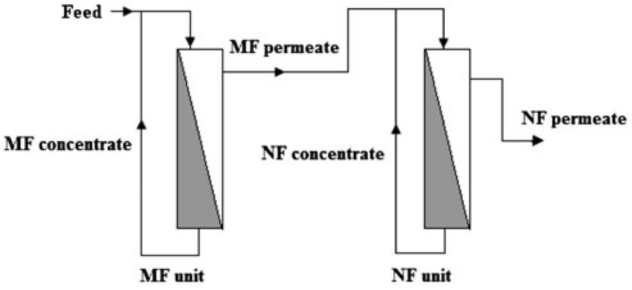

Fig. 1. Schematic presentation of the experimental set-up of MF/NF combination.

The NF membrane is a DESAL membrane product (DK2540F1073) of 1,016 mm length with spiral configuration and an active area of 2.5 m^2. The membrane weight cutoff (MWCO) is of 200 Da.

After every test, membranes were chemically cleaned in place, following a protocol recommended by the membrane maker: base cleaning with NaOH solution ($T=80$°C, $\Delta p=2$ bar for MF and $T=40$°C, $\Delta p=6$ for NF), then acid cleaning with HNO_3 solution ($T=60$°C, $\Delta p=2$ bar for MF and $T=30$°C, $\Delta p=6$ for NF).

All MF experiments were performed under 2 bar pressure and 50°C which is almost the temperature of the effluents at the outlet of the dyeing machine. The temperature of the effluent in the outlet of MF was close to 40°C, which was chosen as the operating temperature of NF membrane. The operational pressure was fixed after a series of experiments detailed in a previous work [3], 12 bar was considered the optimal TMP value for all the NF runs in this work.

2.3. Analytical measurements

Physicochemical parameters were determined according to the standard methods suggested by American Public Health Association [14]. Color measurements were determined with standard dilution multiple method using a UV–visible spectrophotometer (Perkin Elmer Lambda 20 UV/VIS), the cuvette used in all measurements had a width of 10 mm. For the effluents used in this study, peaks were observed with maximum wavelength of 560 nm, and all the samples were 100 times diluted. Chlorides amounts were calculated by $AgNO_3$ dosage following the Mohr method. Temperature and pH measurements were done using a pH-meter, Metrohm 744. Salinity and TDS determinations were done by means of a conductimeter, Tacussel model 123. COD was estimated by open reflux method, the protocol present a method derived from the standard AFNOR T90-101. The COD values were obtained using a Fisher Bioblock Scientific reactor COD 10,119 type COD-meter. Turbidity was measured using a turbidity-meter Hach RATIO 2100 N, and total hardness was calculated using a complexometric titration with EDTA.

For the evaluation of MF color rejection, the percent reduction of color was determined from the absorbance values in the feed and in the MF permeate (A_f and $A_{p,MF}$, respectively) as follows:

$$R(\%) = \left(1 - \frac{A_{\mathrm{p,MF}}}{A_f}\right) \times 100 \quad (1)$$

The mass percentages of color in the permeate, in the concentrate and adsorbed by the membrane, respectively, X_p, X_c, and ε were calculated as follows:

$$X_p(\%) = \frac{m_p}{m_f} \times 100 \quad (2)$$

$$X_c(\%) = \frac{m_c}{m_f} \times 100 \quad (3)$$

$$\varepsilon(\%) = \frac{m_f - m_p - m_c}{m_f} \times 100 \quad (4)$$

m_{f}, m_{p} and m_{c} are, respectively, the amounts of dye in the feed, in the permeate, and in the concentrate and were calculated from the dye concentrations C_f, C_p, and C_c and the total volumes of the feed, the permeate and the concentrate which are, respectively, 60, 45, and 15 l. The concentrations of dye were calculated from the Beer Lambert law connecting linearly the absorbance value to the concentration.

The chlorides removal after the NF treatment was calculated from the chloride amounts in the MF and NF permeates ($C_{\mathrm{p,MF}}$ and $C_{\mathrm{p,NF}}$, respectively) using the rejection parameter as follows:

$$R(\%) = \left(1 - \frac{c_{\mathrm{p,NF}}}{c_{\mathrm{p,MF}}}\right) \times 100 \quad (5)$$

3. Results and discussion

3.1. Effluents characterization

Before studying the treatment performances, it was necessary to identify the pollution level of the effluents. Table 2 represents the characterization of all realized configurations. It can be seen that the dyeing effluent was heavily loaded with salts and color compared with the other effluents. On the other hand, all these effluents represented a pH above the isoelectric point (IEP) of the MF membrane, which is between 6 and 7.5 for ceramic membrane [15]. This will lead to a negative charge of the membrane material.

3.2. MF performances

3.2.1. MF filtration flux

Fig. 2 illustrates the filtration flux vs. time for all baths mixtures. During the filtration tests, permeate fluxes decrease dramatically in the first 20 min then remains relatively constant, this behavior is attributed to the establishment of the membrane fouling. This phenomenon can be explained by the increased concentration close to the membrane surface due to the pollutant retention.

It is obvious that the treatment of single dyeing bath showed the lowest filtration performances, and this can be attributed to the dilution of this concentrated effluent when mixed with the other baths notably less concentrated with pollutants (Table 2).

When one surfactant was used, the filtration flux was enhanced at least by 90% since the stabilized permeate flux varies from 60 l/h m^2 for the dyeing effluent to a value between 100 and 120 l/h m^2 for the effluent coming from baths mixture. However, when two baths containing different surfactants were mixed with the dyeing effluent, an improvement in the stabilized flux value occurred for the combination $D+P+W$ containing anionic and nonionic surfactants, the permeate flux increased from 60 to 130 l/h m^2. The other configurations showed lower fouling intensity than the dyeing effluent filtration, but the stabilized flux did not show an important amelioration.

Table 2
Characterization of different baths combinations before the treatment

Configuration	Dilution ratio	T (°C)	pH	TDS (g/l)	S (g/l)	TH (°F)	Cl^- (g/l)	COD (g/l)	Color[a]	Turb (NTU)
D	1	23.8	11.49	102.8	107.2	220	44.37	3.2	11.4	16.2
$D+P$	1:1	24.5	10.37	23.4	21.3	120	14.2	1.7	4.5	9.7
$D+W$	1:1	25.5	11.25	35.1	36.4	260	19.88	1.5	4.6	11.9
$D+S$	1:1	25.7	10.58	33.2	34.4	172	18.85	2.3	5.1	9.5
$D+P+W$	1:1:1	25.1	9.71	19.93	20.1	208	9.23	1.2	3.4	9.79
$D+P+S$	1:1:1	25.5	10.03	24.13	23.7	251	13.65	1.7	3.1	9.7
$D+W+S$	1:1:1	24.3	9.93	23.1	22.9	180	12.78	2.6	4.4	8.4

[a] Absorbance measurements at 560 nm for samples 100 times diluted.

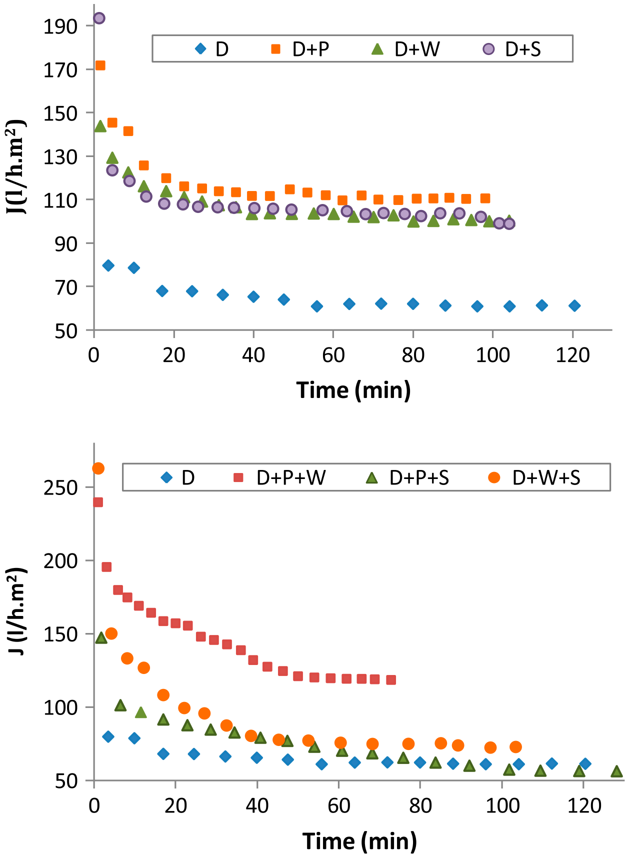

Fig. 2. Variation of the MF filtration flux versus time.

3.2.2. Color removal by MF

Fig. 3 represents the color removal by MF for all considered configurations. The dyeing bath treated separately showed the highest color removal, almost 55%. *D* + *P*, *D* + *S*, and *D* + *W* + *S* configurations showed also similar color retention by about 53% while the *D* + *W* and *D* + *P* + *W* configurations exhibited the lowest color removal by almost 30% only.

It was not possible to analyze these results without understanding the membrane behavior toward dyes particles. In order to study the fouling phenomena taking place on the membrane surface, a dye mass balance was realized (Table 3).

Fig. 3. Color removal by MF for different configurations.

The configuration *D* showed the highest amount of dye fixed on the membrane; 17.72% of initial dye which corresponds to about 10.2 g. On the other hand, the stabilized filtration flux, in this case, was also the lowest compared with the other mixtures: 60 l/h m^2 (Fig. 2). These results indicate that dye molecules accumulation on the membrane surface occurred and reduced the transport through the membrane. However, an eventual electrostatic repulsion between the dye molecules and the membrane, both negatively charged, also took place. In fact, the important amount of dye fixed on the membrane can be attributed to the high color contents (Table 2) which dominated the electrostatic repulsion and was the main cause of the high color removal.

D + *P* configuration gave almost the same rejection rate of dye as configuration *D* (53%); however, the dye amount fixed on the membrane was reduced to 3.03 g, and this can be explained by the dilution effect of the dye bath as well as the increase of repulsion forces applied on the dye molecule resulting from the membrane surface and the anionic surfactant.

D + *S* configuration also led to a significant color removal above 50% which was probably due to the high interaction between the cationic surfactant and the anionic dye. These results were in accordance with Zaghbani et al. [16] who found that the addition of cationic surfactant to reactive dye solution can enhance the dye removal by UF membrane by almost 70%. Following the mass balance, it can be seen that deposit of dye on the membrane surface took place for this configuration since the amount of adsorbed dye was about 3.46 g.

The addition of the washing effluent (*W*) to *D* + *P* configuration reduced notably the color removal which varied from 53 to 30% only. This can be attrib-

Table 3
Dye mass balance for MF process

Configuration	m_f (g)	X_p (%)	X_c (%)	ε (%)
D	57.49	33.60	48.68	17.72
D + *P*	22.69	36.08	50.56	13.37
D + *W*	23.20	50.93	45.11	3.97
D + *S*	25.72	41.93	44.61	13.47
D + *P* + *W*	17.15	52.58	45.14	2.29
D + *P* + *S*	15.63	45.64	43.29	11.07
D + *W* + *S*	22.19	36.79	43.75	19.46

m_f: estimated mass of dye in the feed. X_p: mass percentage of dye load in the permeate. X_c: mass proportion of dye load in the concentrate. ε: mass proportion of dye fixed on the membrane.

uted to the ability of nonionic surfactant present in the washing bath (*W*) to interact strongly with anionic surfactant (*P*) and to weaken the repulsion effect between the anionic surfactant and the dye molecule. Indeed, after the treatment of *D* + *P* + *W* configuration, only 0.39 g of dye was fixed on the membrane. This was in agreement with Kartal and Akbas results [9], who proved that the addition of anionic surfactant to a reactive dye and nonionic surfactant decreased the decolorization due to the ability of both surfactants to interact together.

The ability of the cationic surfactant to interact strongly with the reactive dye molecules or with the nonionic surfactant, contributed to weaken the repulsion forces between these compounds and the membrane. Thus, when the cationic surfactant was added to the reactive dye and nonionic surfactant mixture, the amount of dye fixed on the membrane varied from 0.92 to 4.32 g for *D* + *W* and *D* + *W* + *S* effluents, respectively. In the same context, Akbas and Kartal [10] reported that in a mixture of reactive dye, nonionic surfactant and cationic surfactant, the dye molecule can react with both surfactants, and also the two different surfactants can form a complex.

3.3. NF Performances

The characteristics of the MF permeate were given in Table 4, and it is obvious that the MF treatment provided relatively high retention rates regarding color, turbidity, and COD, but it did not show significant TDS, salinity or chlorides removals.

3.3.1. NF Filtration flux

Fig. 4 represents the variation of the NF filtration flux with time for different configurations. A typical behavior of a NF membrane was obtained; the fluxes dropped slightly in the first 15 min then remained relatively constant. This can be explained by the membrane fouling due to the increasing concentration of the feed and the particles accumulation on the vicinity of the membrane. First of all, it has to be noticed that the MF pretreatment influenced notably the NF behavior; indeed, the effluents performing the best MF filtration fluxes were also those giving the highest NF permeate fluxes. With a stabilized flux of 25 l/h m^2 for the single dyeing

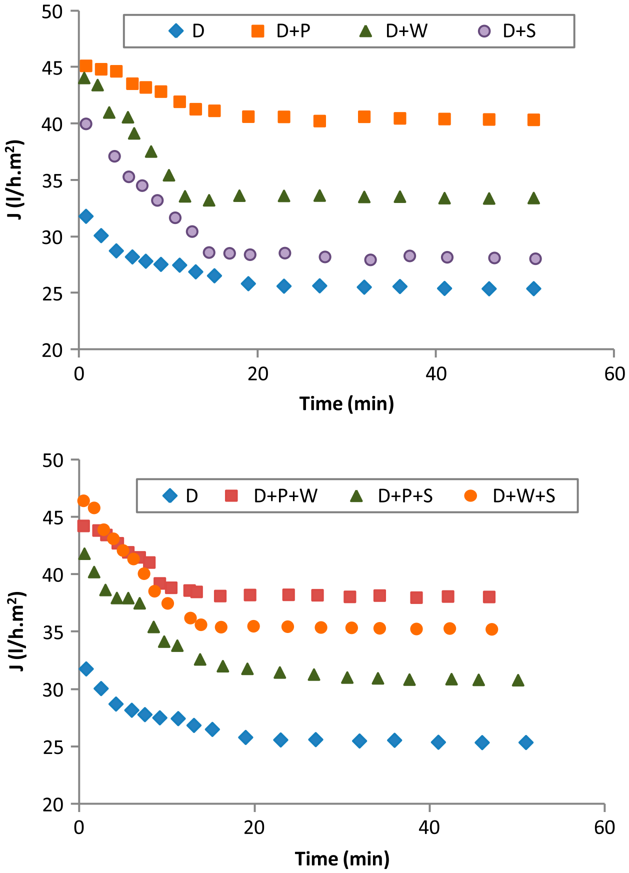

Fig. 4. The variation of the NF filtration flux versus time.

Table 4
Characterization of different baths combinations after MF treatment

Configuration	*T* (°C)	pH	TDS (g/l)	*S* (g/l)	TH (°F)	Cl^- (g/l)	COD (g/l)	Color[a]	Turb (NTU)
D	22.5	9.17	95.60	97.55	167	31.06	2.2	5.1	5.35
D + *P*	22.6	8.55	22.23	20.24	82	11.93	0.6	2.2	0.39
D + *W*	22.9	8.52	31.24	32.03	130	17.30	1.1	3.1	0.60
D + *S*	23.3	8.56	25.90	33.37	93	18.66	1.3	2.6	0.95
D + *P* + *W*	23.2	8.75	19.33	18.69	177	9.14	0.2	2.4	1.66
D + *P* + *S*	22.7	8.92	23.89	22.99	238	12.01	0.8	1.9	1.07
D + *W* + *S*	22.9	8.63	22.41	22.67	169	8.69	1.5	2.2	0.59

[a] Absorbance measurements at 560 nm for samples 100 times diluted.

Table 5
Characterization of different baths combinations after MF/NF treatment

Configuration	T (℃)	pH	TDS (g/l)	S (g/l)	TH (℉)	Cl^- (g/l)	COD (g/l)
D	23.2	9.53	88.9	81.19	46	21.13	0.267
$D+P$	22.9	8.82	17.78	14.60	12	6.85	0.098
$D+W$	23.5	8.74	25.3	22.63	21	13.47	0.124
$D+S$	23.3	8.52	26.8	22.65	10	15.06	0.023
$D+P+W$	23.2	8.71	17.11	16.33	18	6.24	0.031
$D+P+S$	22.8	8.76	13.21	10.80	59	7.92	0.125
$D+W+S$	23.1	8.96	15.95	13.08	18	6.11	0.219

effluent treatment, it can be clearly observed that the baths mixing enhanced the filtration performances, and this behavior is principally due to the effect of the effluent dilution as well as the feed contents. Indeed, the effluent $D+P$ which exhibited the highest stabilized flux (42 l/h m^2) and the lowest fouling intensity was among the effluents characterized by the lowest pollution concentration after the MF treatment (Table 4).

3.3.2. Chlorides removal by the NF membrane

Table 5 represents the characteristics of the NF permeate. Color and turbidity were completely removed but salts amounts still relatively high in the NF permeate. The important amounts of sodium chloride used to fix dyes on the substrate are responsible on the high salts concentration in the dyeing effluent. Therefore, the chlorides retention by the NF membrane was determined for each configuration (Fig. 5).

The chloride atom size is far from reaching 1 nm, the NF membrane pore size. So, the removal of chlorides by NF cannot be possible considering the difference between the pore diameter and the atom size. However, Fig. 5 shows a chlorides removal of at least 20%. Taking into account this behavior and the negative charge of the NF membrane, it has to be expected that the chloride elimination by the NF membrane was driven by Donnan effect and electrostatic repulsion occurred between the membrane and the anions [12,13]. The NF treatment of the dyeing bath showed a chlorides removal of 31%.

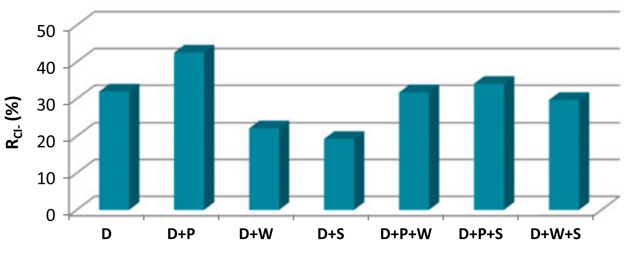

Fig. 5. Chlorides retention by the NF membrane.

The addition of one bath to the dyeing effluent showed that the $D+P$ effluent contained 6.85 g/l of chloride after NF treatment (42% of chloride retention), while $D+W$ and $D+S$ exhibited 13.46 and 15.06 g/l, respectively which corresponds to 22 and 19% of chloride removal. This behavior can be explained by the existence of the anionic surfactant after the MF treatment which increased of repulsion forces between the chloride molecules, the anionic surfactant (P) and the membrane.

The $D+P+W$, $D+P+S$, and $D+W+S$ configurations showed 31, 34 and 29%, respectively, of chloride retention, and the chloride contents after the combined treatment was 6.24, 7.921 and 6.11 g/l, respectively. The similarity between these results can indicate that only the dilution effect was considered, and there was not a significant contribution of surfactants in the chloride elimination.

4. Conclusion

The combined system of membrane processes MF/NF showed to be a promising approach for the treatment of dyeing wastewaters. It provided almost 100% of color and turbidity removal. The dilution of the heavily colored dyeing bath with effluents containing different types of surfactants, assuming an important role in the dyeing cycle, enhanced the membranes filtration efficiency due to the dilution effect as well as the surfactants action. Indeed, the mixing of the dyeing bath and effluents containing one surfactant enhanced the MF filtration flux by 90% at least. Due to the high color concentration in the dyeing effluent, the color removal rate by the MF membrane reached the maximum value when the dyeing effluent was treated separately. However, suitable mixture of the different baths is needed to obtain the best performances regarding permeate flux, treated effluent quality and also fouling control. The chlorides rejection by the NF membrane was driven by Donnan effect, but it was not sufficient to provide chlorides removal exceeding 50%.

Symbols

A	—	absorbance
COD	—	chemical oxygen demand
MF	—	microfiltration
NF	—	nanofiltration
R	—	retention
S	—	salinity
TDS	—	total dissolved solids
TH	—	total hardness
UF	—	ultrafiltration

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