Treatment of textile wastewater by a hybrid electrocoagulation/nanofiltration process

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ABSTRACT

Untreated effluents from textile industries are usually highly coloured and contain a considerable amount of contaminants and pollutants. Stringent environmental regulation for the control of textile effluents is enforced in several countries.

Previous studies showed that many techniques have been used for the treatment of textile wastewater, such as adsorption, biological treatment, oxidation, coagulation and/or flocculation, among them coagulation is one of the most commonly used techniques.

Electrocoagulation is a process consisting in creating metallic hydroxide flocks within the wastewater by the electrodissolution of soluble anodes, usually made of iron or aluminium. This method has been practiced for most of the 20th century with limited success. In recent years, however, it started to regain importance with the progress of the electrochemical processes and the increase in environmental restrictions in effluent wastewater.

This paper examines the use of electrocoagulation treatment process followed by nanofiltration process of a textile effluent sample.

The electrocoagulation process was studied under several conditions such as various current densities and effect of experimental tense. Efficiencies of COD and turbidity reductions and colour removal were studied for each experiment. The electrochemical treatment was indented primarily to remove colour and COD of wastewater while nanofiltration was used to further improve the removal efficiency of the colour, COD, conductivity, alkalinity and total dissolved solids (TDS).

The experimental results, throughout the present study, have indicated that electrocoagulation treatment followed by nanofiltration processes were very effective and were capable of elevating quality of the treated textile wastewater effluent.

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1. Introduction

At the main industrial processes, with suitable control and analysis of the environment, the majority of wastewater produced by industrial activity can be treated by biological purification plants. Nevertheless, textile industry effluents present two main problems: on one hand, they have high COD values and on the other hand, they contain organic compounds like phenols, dyes, tensioactives, etc., some of them very toxic and prevent their direct treatment in a biological plant. To solve this problem, some combinations of physical–chemical techniques have been tested, as chemical coagulation followed by sedimentation and absorption, ozonization and oxidation processes [1–4]. Due to high capital and operating costs of these methods, there is an urgent need to develop more efficient and inexpensive methods which require minimum chemical and energy consumptions, as well as minimum installation space when high land price are taken into account [5–7].

In recent years, investigations have been focused on the treatment of wastewaters using electrocoagulation (EC). Compared with traditional flocculation and coagulation, EC has in theory, the advantage of removing the smallest colloidal particles: the smallest charged particles have a greater probability of being coagulated because of the electric field that sets them in movement. It has also the advantage of producing a relatively low amount of sludge. Secondary pollution may be caused by chemical substance added at a high concentration when chemical coagulation is applied to treat dyeing wastewater. Excessively added coagulants can be avoided by EC, due to the generation of the coagulants by the electro-oxidation of a sacrificial anode [8].

EC is one of the simple and efficient electrochemical methods for the purification of many types of water and wastewaters [9]. In this technique, which is characterized by its simple equipment,

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easy operation and decreased amount of sludge, the generated coagulant leads, at an appropriate pH, to the insoluble metal hydroxide which is able to remove a large variety of pollutants [10]. These metal hydroxide species neutralize the electrostatic charges on suspended solids and oil droplets to facilitate agglomeration or coagulation and resultant separation from the aqueous phase [11,12]. A growing research interest is reported on the treatment of various wastewater types: metal processing wastewaters [13], semiconductor production wastewater [14], textile dyeing wastewaters [15–19], tannery wastewater pre-treatment [20–24], olive mill wastewater [9,10,25], urban wastewater [26], and organics removal from poultry slaughterhouse wastewaters [27]. EC has also been used to treat the landfill leachate by some researchers [28–31].

Other recent studies were focused on the treatment of wastewaters using membrane processes. In fact, membranes technologies provide an important solution in environmental fields such as pollution reduction and water reuse, recycling valuable components from the waste streams [32]. Both nanofiltration (NF) and reverse osmosis (RO) are good alternatives for textile wastewater treatment since high reductions in the aforementioned parameters (conductivity, COD and colour) can be reached. Nevertheless, untreated textile effluents cannot be used directly as an influent to nanofiltration or reverse osmosis membranes due to the high solids concentrations. Thus, it is necessary to carry out a very exhaustive pre-treatment in order to avoid fouling and membrane deterioration [33–35].

In our previous works, different membrane processes were used on the treatment of a biologically treated textile wastewater such as microfiltration (MF), ultrafiltration (UF) and NF [36]. UF process was also used as pre-treatment prior to NF process [37].

The purpose of this paper is to optimise the EC process using aluminium electrodes as a pre-treatment of NF process on the treatment of an untreated textile effluent sample supplied from a Tunisian factory.

2. Materials and methods

2.1. Textile wastewater characteristics

The study was conducted with a textile wastewater sample supplied from the SITEX textile factory in Kasr Hellal, Tunisia. This factory produces about 600,000 m^3 of wastewater per year (which represents the half of the region consumption), which is currently treated with a biological oxidation followed by aerobic stages with activated sludge. The characteristics of both biologically treated and untreated textile effluents are given in Table 1.

Table 1
Characteristics of the treated and untreated wastewater samples of the local textile industry.

Parameter	Untreated effluent	Biologically treated effluent
Conductivity ($\mu S\,cm^{-1}$)	15,190	8106
Turbidity (NTU)	252	5.93
COD ($mg\,L^{-1}$)	4,800	128
TDS ($mg\,L^{-1}$)	12,918	6148.8
pH	13.0	8.25
Colour[a]	1479.9	975.2
Cl^- ($mg\,L^{-1}$)	3739.7	634.3
SO_4^{2-} ($mg\,L^{-1}$)	3502.8	1326.0
Ca^{2+} ($mg\,L^{-1}$)	599.0	89.0
K^+ ($mg\,L^{-1}$)	235.6	106.7
Mg^{2+} ($mg\,L^{-1}$)	161.5	153.3
Na^+ ($mg\,L^{-1}$)	3,518	4339

[a] Integral of the absorbance curve in the whole visible range (400–800 nm).
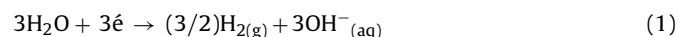
2.2. Analytical methods

The conductivities were measured by a PHYWE type conduct meter. The turbidity of the samples was measured by a Turb 555 IR type turbid meter. The COD values were obtained using a Fisher Bioblock Scientific reactor COD 10119 type COD-meter. pH was measured with a CONSORT C832 (multi-parameter analyser) type pH-meter. The colour intensity of feed and permeate samples were analyzed by PerkinElmer Lambda 20 spectrophotometer. The colour was measured using the integral of the absorbance curve in the whole visible range (400–800 nm). Anions were determined by ion chromatography using a Metrohm 761 Compact IC with conductivity detection. The anion chromatography measurements with chemical suppression were made with a Metrosep anion dual 2 column (4.6 mm × 75 mm) with a particle diameter of 6 μm. Cations amounts were determined by atomic spectroscopy using an analytical AAS Vario 6 spectrometer.

2.3. Principle and experimental procedure of EC

2.3.1. Description of EC process

Electrocoagulation is based on the in situ formation of the coagulant as the sacrificial anode corrodes due to applied current, while the simultaneous evolution of hydrogen at the cathode allows for pollutant removal by flotation. The mechanism of electrocoagulation is highly dependent on the chemistry of the aqueous medium, especially conductivity. In addition, other characteristics such as pH, particle size, and chemical constituent concentrations will also influence the electrocoagulation process. When aluminium is used as electrode materials, the reactions are as follows:

At the cathode:

$$3H_2O + 3\acute{e} \rightarrow (3/2)H_{2(g)} + 3OH^-_{(aq)} \tag{1}$$

At the anode:

$$Al_{(s)} \rightarrow Al^{3+}_{(aq)} + 3\acute{e} \tag{2}$$

In the solution:

$$Al^{3+}_{(aq)} + 3H_2O \rightarrow Al(OH)_3 + 3H^+_{(aq)} \tag{3}$$

Three main processes occur during EC: (i) electrolytic reactions at electrode surfaces, (ii) formation of coagulants in the aqueous phase, and (iii) adsorption of soluble or colloidal pollutants on coagulants, and removal by sedimentation or flotation.

$Al^{3+}_{(aq)}$ and OH^- ions generated by electrode reactions (1) and (2) react to form various monomeric species such as $Al(OH)^{2+}$, $Al(OH)_2^+$, $Al(OH)_4^-$ and polymeric species such as $Al_2(OH)_2^{4+}$, $Al_6(OH)_{15}^{9+}$, $Al_7(OH)_{17}^{4+}$, $Al_8(OH)_{20}^{4+}$, $Al_{13}O_4(OH)_{24}^{7+}$, and $Al_{13}(OH)_{34}^{5+}$, which transform finally into $Al(OH)_3$ according to complex precipitation kinetics [38]. Formation rates of the different species play also an important role in the decolourization process. Several interaction mechanisms are possible between dye molecules and hydrolysis products and the rates of these depend on pH of the medium and types of ions present. Two major interaction mechanisms are being considered in recent years; precipitation and adsorption, each being proposed for a separate pH range. Flocculation in the low pH range is explained as precipitation, while the higher pH range (>6.5) as adsorption [38–41]. At high pH values above 9, $Al(OH)_4^-$ is also present in the system. Freshly formed amorphous $Al(OH)_3$ "sweep flocks" have large surface areas which are beneficial for a rapid adsorption of soluble organic compounds and trapping of colloidal particles [39]. These flocks polymerize as
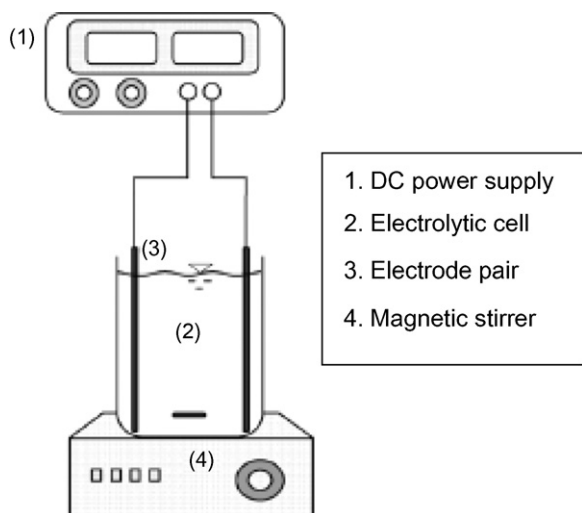
$$nAl(OH)_3 \rightarrow Al_n(OH)_{3n} \tag{4}$$

Fig. 1. The electrocoagulation experimental setup.

and they are easily removed from aqueous medium by sedimentation and by H_2 flotation. Secondary anodic reactions occur also during EC for example, in neutral and acidic chloride solutions, native and free chlorine and hypochlorite are formed which are strong oxidants [42,43].

2.3.2. Description of experimental apparatus and procedure

Two aluminium electrodes were used. Both aluminium cathode and anode were made from plates with the same dimensions of 60 mm × 50 mm × 1 mm. The total effective electrode area for each electrode was 30 cm². The distance separating the electrodes was fixed to 20 mm. They were connected to terminals of a direct current power supply (Statron, model 2224.1) characterized by the ranges 0–5 A for current and 0–25 V for voltage. A constant stirring speed of 700 rpm was applied during all experiments. The experimental setup is shown in Fig. 1.

Before each run, electrodes were stocked for 3 min in a sodium hydroxide solution (5 mol L^{-1}) to form Al(OH)$_3$. Then, they were washed by pure water and restocked for 5 min in a solution freshly prepared of HCl (3 mol L^{-1}) to obtain pure aluminium electrodes. A second wash with pure water was made.

The electrocoagulation process represents, in the present work, the first step of the textile effluent treatment (pre-treatment step) as shown in Fig. 3. Two major parameters were optimized: the experimental run time (t) and the current density (j).

The removal efficiency of COD, conductivity, turbidity and colour after EC treatment was determined using the following equation:

$$R(\%) = 100 \times \left(1 - \frac{X_{T_{sol}}}{X_{F_{sol}}}\right) \qquad (5)$$

where $X_{T_{sol}}$ and $X_{F_{sol}}$ represent the measured parameters (COD, conductivity, turbidity or colour), respectively, in the EC treated solution and in the feed solution.

2.4. Principle and experimental procedure of NF

2.4.1. NF membrane characteristics

A flat sheet polyamide/polysulfone based thin film composite membrane was used in NF process. This membrane is commercialised as HL membrane by Osmonics manufacture. Its effective surface is about 140 cm². The membrane permeability was determined using pure water. Flux values of pure water at different operating pressures were measured and were plotted against pressure difference in Fig. 2. The average value of membrane perme-
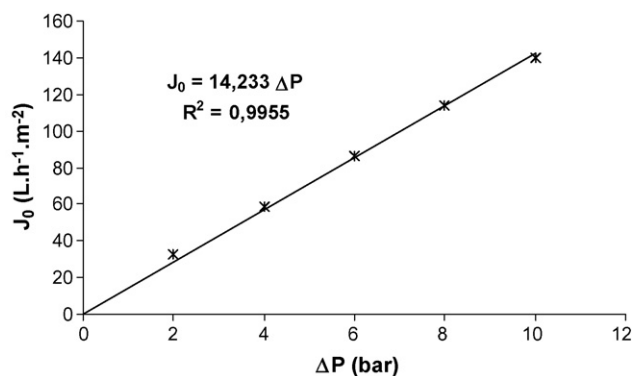

Fig. 2. Evolution of pure water flux with transmembrane pressure.

ability as measured was 14.233 L h^{-1} m^{-2} bar^{-1}, which remained almost constant through out the conduction of all experiments.

2.4.2. Conduction of NF experiments

The type of the used NF plant is SEPA CF II, Osmonics. The operating parameters were the volume reduction factor (VRF) (Eq. (6)) and transmembrane pressure (ΔP). The studied VRF range was [1,2] and the transmembrane pressure was varied from 2 to 12 bar.

During each experiment, cumulative permeate volumes were collected and analyzed. After thoroughly cleaning the system several times by pure water, the membrane permeability was re-evaluated. It was observed that the membrane permeability remained almost constant between successive runs.

$$VRF = \frac{V_f}{V_r} \qquad (6)$$

where V_f and V_r represent, respectively, the feed and retentate volumes.

As indicated in Section 2.3.2, the electrocoagulation process was followed by nanofiltration process in the treatment of the textile effluent sample. Fig. 3 shows the experimental setup of the hybrid EC/NF process.

3. Results and discussions

3.1. Optimization of EC parameters

The efficiency of EC process depends on several parameters such as: current density (j), electrolysis time (t), initial pH (pH$_i$), type of electrode material (Fe or Al) and distance separating the anode to the cathode (d). In order to enhance the process performance, the effects of current density and electrolysis time have been tested. In the present work, only the untreated effluent was used. The initial pH of this textile wastewater was about 13 as mentioned in Table 1. It has been established in several previous studies that aluminium electrode was selected as the most appropriate material in textile effluent treatment [3,20,44]. That is why, aluminium electrodes were selected in our work.

3.1.1. Effect of current density

The applied current density was varied from 3.33 to 100 mA cm^{-2}. Fig. 4 shows COD, turbidity, conductivity and colour removal efficiencies upon electrolysis time. Experimental
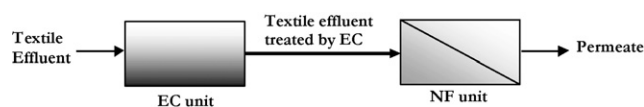

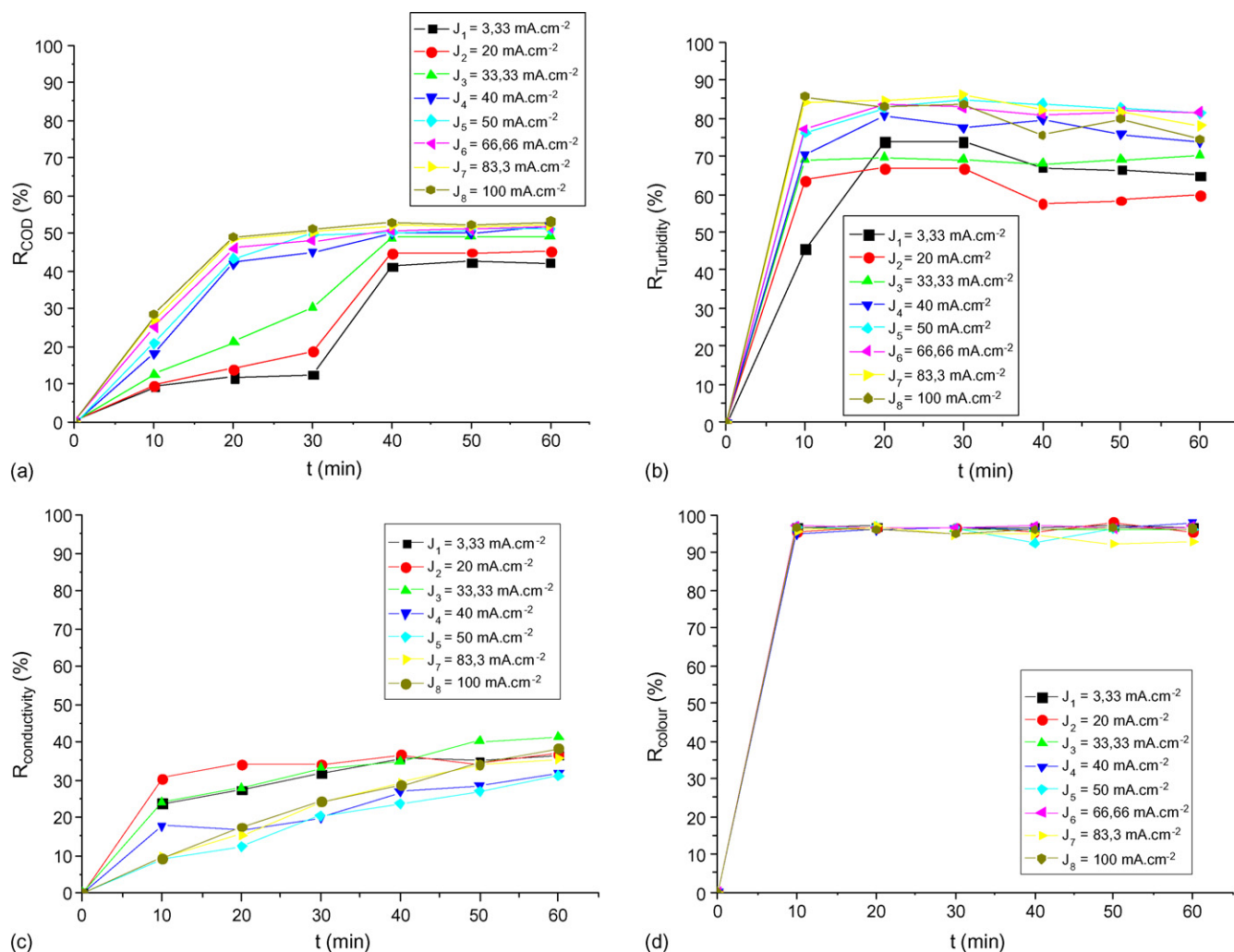**Fig. 3.** Schematic diagram of the experimental setup of the hybrid EC/NF process.

Fig. 4. Effect of current density on the analyzed parameters removal rate: (a) COD; (b) turbidity; (c) conductivity and (d) colour.

results show that COD removal efficiencies increase faster when current density increases (Fig. 4a). So, the current density has a strong effect on the kinetics of COD abatement: the curves reach the same asymptotic value (about 50%) as a function of electrolysis time. But, the highest current densities ($J > 40\,\mathrm{mA\,cm^{-2}}$) produced the quickest treatment with 50% COD removal efficiency. This
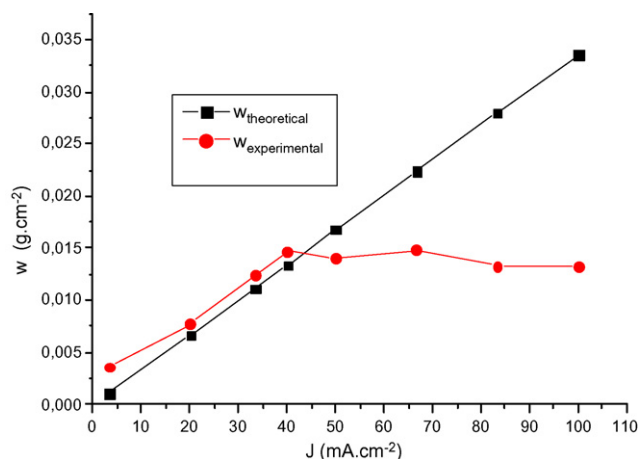

Fig. 5. Effect of current density on the anode consumption.

result is ascribed to the fact that at high current density, the extent of anodic dissolution of aluminium increases, resulting in a greater amount of precipitate for the removal of pollutants. Moreover, bubble generation rate increases and the bubble size decreases with increasing current density. These effects are both beneficial for high pollutant removal by H_2 flotation [45]. Fig. 4b shows that increasing current density has also a significant effect on the turbidity removal efficiency. In fact, the turbidity removal value was increased from 60% to 85% when current density was varied from 3.33 to 100 $\mathrm{mA\,cm^{-2}}$. More than 80% of turbidity removal efficiency was observed when only 40 $\mathrm{mA\,cm^{-2}}$ of current density was applied. Fig. 4c shows that no significant conductivity removal efficiencies were obtained (35% as maximum). However, more than 95% of colour removal was achieved for the whole range of applied current density (Fig. 4d). Fig. 5 shows the effect of current density on the anode consumption. The amount of aluminium oxidized after 60 min of electrolyze for each current intensity was calculated using Faraday's law (7) and also measured by weighting the electrode before and after each experiment (8). The theoretical and experimental anode consumption values are shown in Fig. 5.

$$w_{theoretical} = \frac{J\,t\,M}{n\,F} \tag{7}$$

$$w_{experimental} = w_{before} - w_{after} \tag{8}$$

where w is the quantity of anode material dissolved (g of Al $\mathrm{cm^{-2}}$), J the current density (A $\mathrm{cm^{-2}}$), t the run time (s), M the rel-
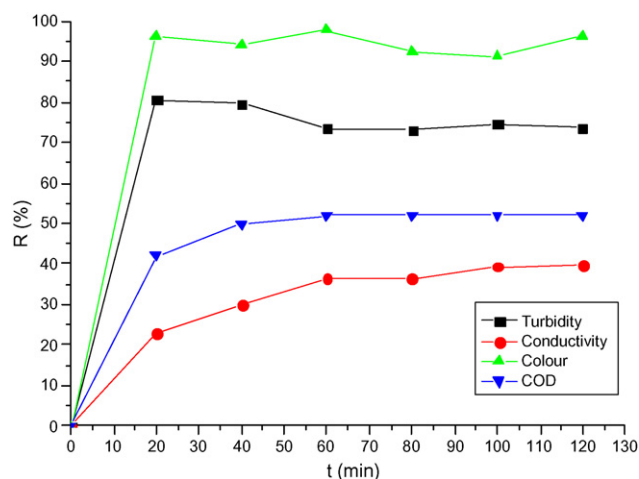
Fig. 6. Effect of electrolysis time on the analyzed parameters removal rate ($J = 40 \, \text{mA cm}^{-2}$).

Fig. 7. Effect of TMP on the retention rate of NaCl, Na$_2$SO$_4$ and CaCl$_2$ (10^{-3} M).

ative molar mass of aluminium, n the number of electrons in oxidation/reduction reaction ($n = 3$), F the Faraday's constant ($F = 96,500 \, \text{C mol}^{-1}$), w_{before} and w_{after} are, respectively, the experimental anode weight before and after electrolysis. The ratio between $w_{experimental}$ and $w_{theoretical}$ represents the EC yield. Results demonstrate that a good agreement between the calculated amount of aluminium oxidized and the experimental amount determined when current density was varied from 3.33 to 40 mA cm^{-2}. When current density exceeds 40 mA cm^{-2}, theoretical values became higher than experimental ones and a decrease of the EC yield was obtained. Taking into account all previous results, the current density was optimized to 40 mA cm^{-2}.

3.1.2. Effect of electrolysis time

The electrolysis time was varied from 0 to 120 min. Fig. 6 shows COD, turbidity, conductivity and colour removal efficiencies upon electrolysis time when the optimal current density was applied ($J = 40 \, \text{mA cm}^{-2}$). Experimental results show that removal efficiencies remained constant after 60 min of electrolysis time especially for COD and conductivity. These results suggest 60 min as an optimal electrolysis time for the treatment of the textile effluent.

3.2. Treatment of textile effluent by EC/NF hybrid process

3.2.1. Optimization of the transmembrane pressure (TMP)

The TMP was varied from 2 to 12 bar. Fig. 7 shows the retention rate of three salts (NaCl, Na$_2$SO$_4$ and CaCl$_2$) upon TMP using HL membrane previously characterized. The feed concentration of each salt solution was fixed to 10^{-3} M. Experimental results show that retention rates increased with increasing TMP and constant values were obtained for TMP > 10 bar. This result suggests 10 bar as an optimal TMP for the nanofiltration experiments.

3.2.2. Textile effluent treatment in optimum conditions

Three parameters were previously optimized: for the EC stage, the current density and electrolysis time were optimized respectively to 40 mA cm^{-2} and 60 min; for the NF stage, experiments were made at 10 bar as optimum TMP.

The NF process performance was controlled by measuring the permeate flows and the operating pressure during experiments. Permeate samples were collected for water quality analyses. The membrane permeability to water was measured before beginning experiments and it was verified after membranes cleaning.
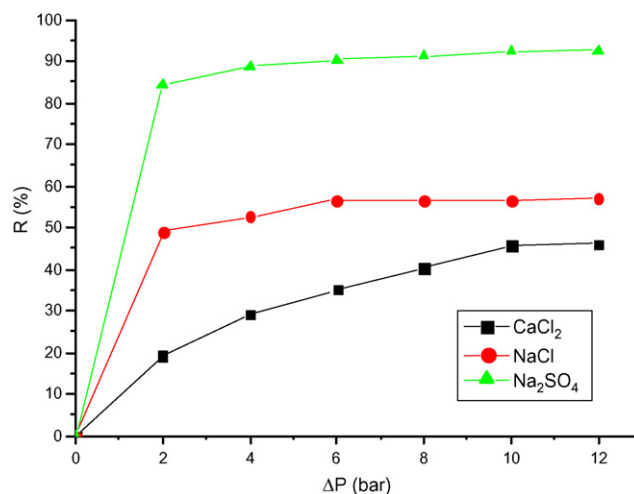
Influence of filtration time on permeate flux was studied on keeping a constant operating pressure ($\Delta P = 10$ bar) and with recycling the retentate to the feed tank at a temperature value equal to 30 °C. Results are shown in Fig. 8.

The study of the EC/NF combination performances at optimized conditions is shown in Figs. 9–11 which represent the evolution of analyzed parameters with the VRF.

Considerable retention rates were obtained for all analyzed parameters. In fact, experimental results show that more than 92% of retention rate was observed for turbidity, COD, sodium, magnesium, potassium, chloride and sulphate. A total decolourization was obtained for all VRF values ($R > 99\%$). Fig. 10 shows that calcium retention rate increases with VRF and reaches 89% at VRF = 1.66. Result details are given in Table 2. Taking into account previous observations, we can conclude that using EC as pre-treatment for NF process improved the quality of treated textile effluent. The present work demonstrates that biological treatment can be changed with EC process in textile effluent treatment. This result has an important economical impact. In fact, biological treatment is characterized with the addition of great amounts of chemicals. EC makes faster the textile effluent pre-treatment and no additional chemicals are required.

The effect of electrocoagulation in COD reduction and effluent decolourization can be explained by
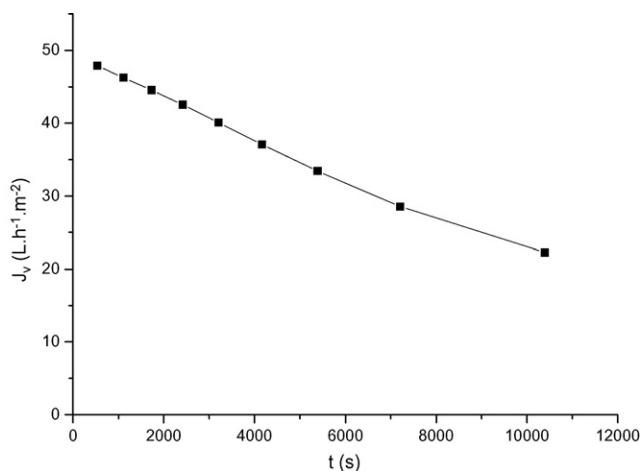

Fig. 8. Influence of filtration time on permeate flux at optimized conditions ($J = 40 \, \text{mA cm}^{-2}$, $t_{EC} = 60$ min, $\Delta P = 10$ bar and $\theta = 30$ °C).
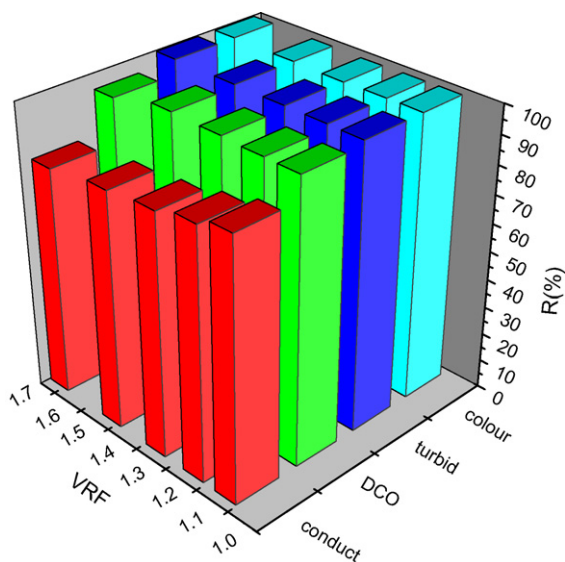
Fig. 9. Evolution of the retention rate of conductivity, turbidity, COD and colour with VRF after EC/NF textile effluent treatment at optimized conditions.
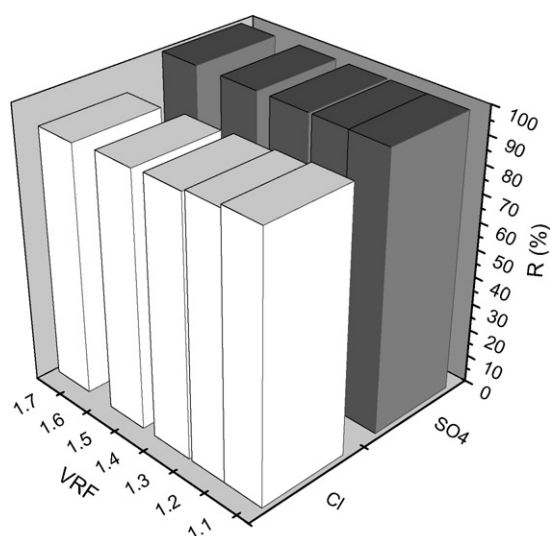

Fig. 11. Evolution of the retention rate of sulphate and chloride with VRF after EC/NF textile effluent treatment at optimized conditions.
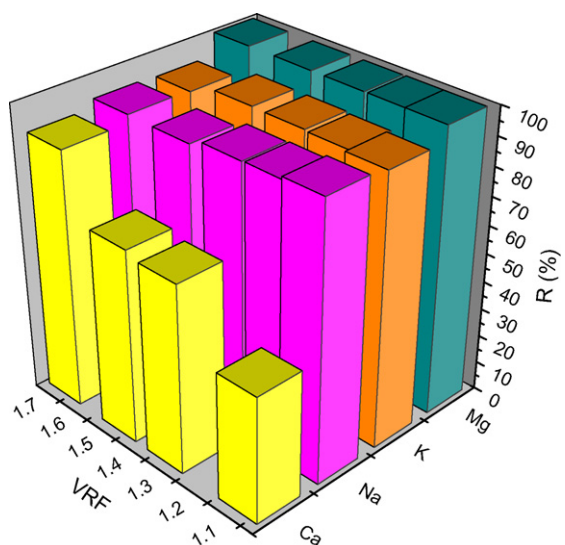

Fig. 10. Evolution of the retention rate of different cations with VRF after EC/NF textile effluent treatment at optimized conditions.

- Aluminium has only one oxidation state, so when an organic compound reacts with aluminium to form an insoluble compound it will react almost completely.
- The solubility of aluminium hydroxide [$Al(OH)_3$] has its minimum at a lower pH close to 4, so the textile effluent pH (13) promotes the COD reduction,
- When using aluminium electrodes there is oxygen evolution at the cathode which may help with the oxygen demand.
- Usually azo dyes are more accessible to chemical reduction and reactive dyes are more easily broken down by chemical oxidation. Due to low solubility, disperse dyes rarely participate in aqueous phase chemical reactions. Because electrochemical treatment provides oxidation, reduction, adsorption, coagulation, and flotation in one reactor, one would expect that the treatment might offer a universal process for decolorizing a matrix of textile dyes.

NF membranes retain low molecular weight solutes such as inorganic salts or small organic molecules such as glucose and pollutants such as pesticides, and dyes. NF typically has partial salt retention and rejects molecules from 500 to a few thousands Daltons. The retention of divalent ions (Ca^{2+} and Mg^{2+}) by NF process is generally higher than the retention of monovalent ions (Na^+, K^+, and Cl^-). This is because most NF polymers have formal charges, which exclude higher valence ions more than monovalent ions from passing through the membrane. These properties lead to the NF membranes to reduce COD and colour.

Table 2
Performance of EC/NF in treating textile wastewater.

Membrane	HL, Osmonics				
TMP (bar)	10				
VRF	1.09	1.19	1.32	1.47	1.66
Conductivity ($\mu S\,cm^{-1}$) (R (%))	882.7 (90.4)	1153 (87.4)	1405 (84.7)	1556 (83.0)	1827 (80.1)
Turbidity (NTU) (R (%))	0.481 (99.8)	0.094 (99.9)	0.145 (99.9)	0.08 (99.8)	0.237 (99.9)
Colour[a] (R (%))	1.94 (99.9)	2.43 (99.8)	6.49 (99.6)	2.54 (99.8)	1.05 (99.9)
COD (mg L^{-1}) (R (%))	80.0 (98.3)	73.6 (98.5)	51.2 (98.9)	36.8 (99.2)	240.0 (95.0)
Cl$^-$ (mg L^{-1}) (R (%))	188.7 (94.9)	234.5 (93.7)	267.6 (92.8)	299.7 (92.0)	358.0 (90.4)
SO$_4{}^{2-}$ (mg L^{-1}) (R (%))	0.0 (100.0)	0.0 (100.0)	0.0 (100.0)	0.0 (100.0)	0.0 (100.0)
Ca^{2+} (mg L^{-1}) (R (%))	332 .5 (44.5)	–	203.0 (66.1)	191.5 (68.0)	60.08 (90.0)
K$^+$ (mg L^{-1}) (R (%))	12.2 (94.8)	10.0 (95.7)	9.1 (96 .1)	8.9 (96.2)	18.1 (92.3)
Mg^{2+} (mg L^{-1}) (R (%))	0.0 (100.0)	0.0 (100.0)	0.0 (100.0)	0.0 (100.0)	0.0 (100.0)
Na$^+$ (mg L^{-1}) (R (%))	353.1 (96.1)	428.1 (95.2)	452.7 (95.0)	656.1 (92.7)	644.2 (92.8)

[a] Integral of the absorbance curve in the whole visible range (400–800 nm).

4. Conclusion

Process selection and operating conditions are important issues to optimize technically and economically the textile effluent treatment. NF process was successfully used to improve permeate quality of biologically treated textile wastewater, but this process presented some limitations in the treatment of raw effluent because of membrane fouling problems. So, NF process required an efficient pre-treatment step.

The results of this study show that EC process seems to be an efficient method in the pre-treatment of the textile wastewater when optimal conditions are satisfied. In fact, experimental results show that more than 92% of retention rate was observed for turbidity, COD, sodium, magnesium, potassium, chloride and sulphate. A total decolourization was obtained for all VRF values ($R > 99\%$). Based on the experiments, the hybrid EC/NF process is suitable for producing reusable water quality.

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