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ELECTROFLOCCULATION FOR TEXTILE WASTEWATER TREATMENT

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Abstract - This work reports on the viability of the electroflocculation (EF) process for chemical oxygen demand (COD), turbidity and color removal from a raw effluent originated from a particular textile industry related to hemp manufacture. Firstly, the following operational parameters were optimized: current density; initial pH; electrolysis time; material of the electrode (iron, aluminum or iron-aluminum); and interelectrode distance. Additionally, the effects of these parameters on specific electrical energy consumption (SEEC) were studied under the optimum conditions. The best removal efficiencies obtained were 93% for color, 99% for turbidity and up to 87% for COD using an aluminum electrode, the initial pH was 5, the cell time operation was 30 min and current density was 15 A/m^2. These results indicate that, under the studied operational conditions, electroflocculation of these efluents may constitute a viable alternative for COD, turbidity and color removal.

Keywords: Textile effluent; Dye; Electroflocculation.

INTRODUCTION

The textile industry is one of many industries that utilizes large volumes of water in the manufacturing process. This water, used in the dying and finishing processes, ends up as wastewater, which needs to be treated before its final discharge. Frequent changes of the dyestuff employed in the process cause considerable variation in the wastewater characteristics, such as intense color, high chemical oxygen demand (COD), dissolved solids values and highly fluctuating pH, the last being especially troublesome because pH tolerance of conventional biological and chemical treatment systems is very limited. Hence, without continuous pH adjustment, normal operation of the treatment process is impossible (Gürses et al, 2002).

Color is one of the most important water quality parameters. During the dying process, about 5 - 20% of the dye are lost due to its partial adsorption on the fibers (Paschoal and Tremiliosi-Filho, 2005). Dyes are manufactured to have high chemical resistance because they are normally chemical species that are very difficult to degrade (aromatic dyes). Moreover, dye solutions usually contain antibacteria and antifungi agents, which are used to give the fibers more resistance to biological degradation (O`Neil et al, 1999).

Even at relatively low concentrations, the intense color associated with the dye affects not only the aesthetics, but also the transparency of waters, thus interfering in photosynthesis, and the solubilization of gases in lakes, rivers and other surface water bodies. It damages both the aquatic flora and fauna. Furthermore, colored effluents may contain considerable amounts of toxic compounds, especially azo dyes, that are known to be highly carcinogenic (Daneshvar et al, 2007; Kunz et al, 2002).

Color removal from these effluents before being discharged constitutes a great challenge for the textile industry due to strict water quality parameters imposed

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*To whom correspondence should be addressed by environmental agencies. Hence, conventional treatment processes have been applied, namely: adsorption, precipitation, chemical degradation, photochemical degradation, biodegradation, flocculation, and so on (Paschoal and Tremiliosi-Filho, 2005). Over the recent years, there has been a growing interest in the development of efficient electrochemical processes for the removal of toxic organic wastewater contaminants. Such processes have been successfully tested to treat various industrial wastewaters, including dye wastewaters (Xiong et al, 2001).

The electrochemical process can be a viable alternative. It consists of electroflocculation (EF), carried out in an electrochemical reactor, which consists of a cell in which the sacrificial electrodes, — made either of aluminum or iron — are the site where Al^{3+} or Fe^{3+} ions are generated due to a given applied potential. This stage is known as the anodic stage, in which the metallic aluminum or iron is oxidized. Once formed, these cations immediately undergo further spontaneous reactions to produce the corresponding hydroxides and/or polyhydroxides. These compounds have a strong affinity for dispersed/dissolved substances, as well as counter-ions to cause coagulation/adsorption phenomena (Crespilho and Rezende, 2004).

The most widely used electrode materials in electrocoagulation process are aluminum and iron. In the case of aluminum, the main reactions are:

Anode:

$$Al_{(s)} \rightarrow Al^{3+} + 3e^- \qquad (1)$$

Cathode:

$$3H_2O + 3e- \rightarrow 3/2\ H_{2(g)} + 3OH^-, \text{ or}$$

$$2H_2O + O_2 + 4e^- = 4OH \qquad (2)$$

Al^{3+} and OH^- ions generated by electrode reactions (1) and (2) react to form various monomeric species, which finally transform into $Al(OH)_{3(s)}$ according to complex precipitation kinetics.

$$Al^{3+} + 3H_2O \rightarrow Al(OH)_3 + 3H^+ \qquad (3)$$

Freshly formed amorphous $Al(OH)_{3(s)}$ “sweep flocs” have large surface areas, which are beneficial for a rapid adsorption of soluble organic compounds and trapping of colloidal particles. Finally, these flocs are removed easily from aqueous medium by sedimentation or H_2 flotation (Daneshvar et al, 2006).

The mechanism of removal of pollutants by an EC process with iron electrodes is shown below.

Upon oxidation in an electrolytic system iron produces iron hydroxide, $Fe(OH)_n$ where $n = 2$ or 3. Two mechanisms have been proposed for the production of $Fe(OH)_n$ (Mollah et al, 2001; Xu and Zhu, 2004).

Mechanism 1:

Anode:

$$4Fe_{(s)} \rightarrow 4Fe^{2+}{}_{(aq)} + 8e^- \qquad (4)$$

$$4Fe^{2+}{}_{(aq)} + 10H_2O_{(l)} + O_{2(g)} \rightarrow 4Fe\left(OH\right)_{3(s)} + 8H^+{}_{(aq)} \qquad (5)$$

Cathode:

$$8H^+{}_{(aq)} + 8e^- \rightarrow 4H_{2(g)} \qquad (6)$$

Overall:

$$4Fe_{(s)} + 10H_2O_{(l)} + O_{2(g)} \rightarrow 4Fe\left(OH\right)_{3(s)} + 4H_{2(g)} \qquad (7)$$

Mechanism 2:

Anode:

$$Fe_{(s)} \rightarrow Fe^{2+}{}_{(aq)} + 2e^- \qquad (8)$$

$$Fe^{2+}{}_{(aq)} + 2OH^-{}_{(aq)} \rightarrow Fe(OH)_{2(s)} \qquad (9)$$

Cathode:

$$2H_2O_{(l)} + 2e^- \rightarrow H_{2(g)} + 2OH^-{}_{(aq)} \qquad (10)$$

Overall:

$$Fe_{(s)} + 2H_2O_{(l)} \rightarrow Fe(OH)_{2(s)} + H_{2(g)} \qquad (11)$$

The $Fe(OH)_{n(s)}$ remains in the aqueous phase as a gelatinous suspension, which can remove the pollutants from the wastewater by either complexation or electrostatic attraction followed by coagulation. In the surface complexation mode, the pollutant acts as a ligand (L) to chemically bind hydrous iron:

$$L{-}H_{(aq)} + (OH)OFe_{(s)} \rightarrow L{-}OFe_{(s)} + H2O_{(l)} \qquad (12)$$

The prehydrolysis of Fe^{3+}cations also leads to the formation of reactive clusters for wastewater treatment (Xu and Zhu, 2004).

Besides the formation of the coagulating agent, the EF also generates micro gas bubbles that are responsible for the flotation of flocculated particles. When the effluent also contains chloride ions, Cl_2 will be formed at the anode and could react with water to generate hypochlorite ions. This fact can be considered to be a great advantage for EF, because chlorine generation can be optimized for both the disinfection of the water and the elimination of undesirable odors (Mollah et al, 2001).

The aim of the present work was to study the technical viability of the electroflocculation process for the removal of COD, turbidity and color from an effluent generated by a textile industry of hemp manufacture.

MATERIALS AND METHODS

Efluent samples, supplied by a textile industry located in the city of Rio de Janeiro, Brazil, were taken from a tank with 1000 dm^3 capacity, containing a mixture of waste dye solutions, whose main characteristics are show in Table 1.

Table 1: Characterization of the textile wastewater

Parameters	Range of values
COD (mgO_2/L)	1179 - 2553
Temperature (ºC)	41 - 47
pH	9.9 - 11.6
Turbidity (NTU)	8.0 - 75.3
Color (Abs)	0.503 - 0.842
Conductivity (mS/cm)	2.2 - 4.61
Flow Rate (m^3/day)	20 - 90

The experimental set-up (Figure 1), developed for the Peltron Brazilian Co., an environmental engineering company, consisted of a glass container with 100 cm^3 capacity, in which beehive type electrodes, several electrodes located side by side, were vertically inserted. This arrangement of electrodes was built with 8 intercalated Al and/or Fe plates, 4 of them corresponding to the cathodes and the remaining to the anodes, whose dimensions were 10 cm x 5 cm x 0.15 cm, separated by spacers with a thickness of 0.5 cm each, thus allowing one to vary the distance between them. The experiments were carried out with the raw effluent at room temperature and with constant agitation (approximately 200 rpm). The electrodes were connected to a DC power supply with galvanostatic operational options for controlling the current density.

After the electrolysis, the reactor content was filtered and analyses were made with samples of the filtrate. In order to reduce cathode passivation as well as to duplicate the electrode lifespan, in all experiments, inversion of polarity was carried out every 5 minutes by means of a manual device (Mollah,. 2001).

The process efficiency was measured in terms of the following parameters: color, turbidity, and Chemical Oxygen Demand (COD), which was measured by the procedure described in "*Standard Methods for the Examination of Water and Wastewater*" (APHA 1995). Digestion of samples was carried out through in a DRY BLOCK TE 021 and the reminiscent color was evaluated by measurements of absorbance of the solutions, at the wavelength of the effluent. The equipment used was a FEMTO 600S spectrophotometer. The evaluation of the turbidity removal efficiency was made with a TECNOPON TB 1000 turbidimeter. Conductivity was measured with a DIGIMED DM3 conductivimeter and pH, adjusted with 3 mol.L^{-1} H_2SO_4 solution, with an OAKTON ION 6 ACON SERIES pHmeter.

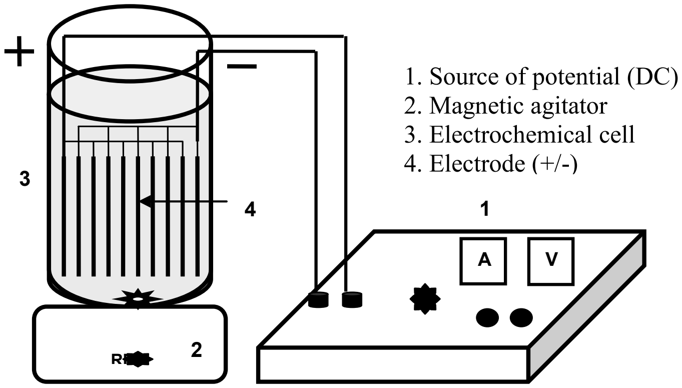


Figure 1: Experimental Set-Up

RESULTS AND DISCUSSION

The Effect of Initial pH

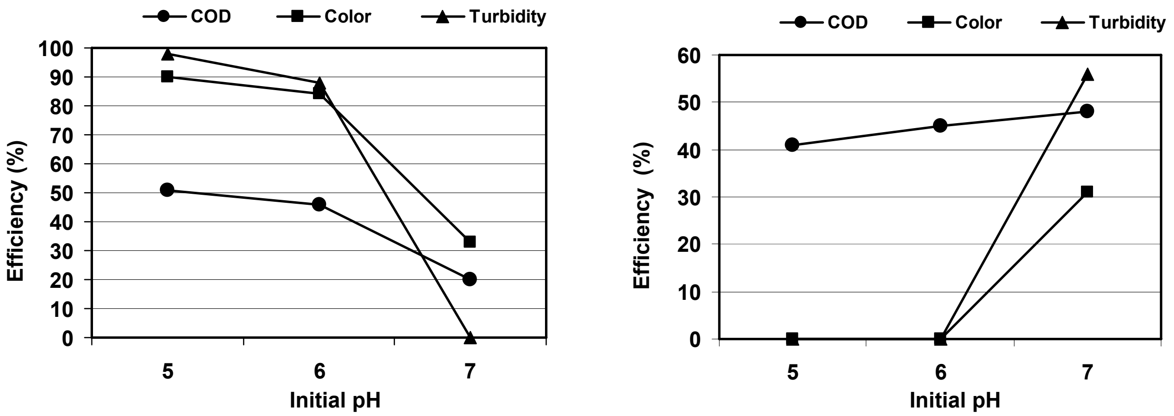
As pointed out by several authors (Lin and Chan, 1997; Chen et al., 2000; Gurses et al., 2002; Kobia et al., 2003), pH is an important operating factor influencing the electrocoagulation process. The pH of the medium changes during the process, depending on the type of electrode material and initial pH. On the other hand, the electrocoagulation process exhibits some buffering capacity, especially in alkaline medium, which prevents large changes in pH and a decrease of the pollutant removal efficiency (Bayramoglu et al 2004).

The effect of the initial value of pH on COD, turbidity and color removal efficiencies from wastewater is presented in Figure 2a for the aluminum electrode with application of a current density of 15 A/m^2 for 30 min. The color and turbidity removal efficiencies were determined at pH values in the range of 5 and 6, in which, however, lower COD removal (50%) was observed. The removal efficiency for these parameters drops dramatically above pH 6. Thus, it may be concluded that the initial pH has a significant effect on the removal efficiency of these parameters. On the other hand, in acidic media, higher removal efficiencies are obtained.

During the electrolysis, the pH of the medium increased, probably due to the liberation of $H_{2(g)}$ at the cathode and the formation of $OH^-_{(aq)}$ Ions. Hence, at higher initial pH, different Al hydroxide forms were present during the electrolytic process, for example: between pH 5 and 6, the dominant aluminum species are $Al(OH)_2^+$ and $Al(OH)_3$, which acts as a coagulating agent. At pH 7, the removal efficiency of this contaminant was lower due to the formation of soluble $[Al(OH)_4^-]$ aluminum complexes (Holt, 2002).

In the case of iron electrodes (Figure 2b), at different pH values (range 5-7), color, turbidity and COD removal efficiencies were lower compared with the aluminum electrode. However, at neutral pH, the removal efficiencies were higher for the case of the iron electrode. The formation of Fe^{2+}(aq) ions and their posterior oxidation to Fe^{3+}(aq) cause the precipitation of iron (III) hydroxide; it can be emphasized that this oxidation by oxygen becomes higher as the pH increases. In this experiment with initial pH 7, the pH of medium increased to 8.5. At this pH value, iron cations form hydroxocomplexes that act as particle destabilization agents and promote removal of the pollutant due to the formation of a solid phase - sludge (Diaz et al, 2003).

Thus, the higher efficiency value for the aluminum electrodes (Figure 2a) occurred again with an initial pH of 5, probably due to the higher production of $Al(OH)_3$. In the case of the iron electrode, the higher value of the removal efficiency occurred at an initial pH of 7. This is probably due to the fact that a higher production of $Fe(OH)_3$ occurs, and this compound may be responsible for the removal of the major part of the impurities in the textile wastewater.



(a) (b)

Figure 2: Effect of initial pH on COD, color and turbidity removal by (a) aluminum electrodes and (b) iron electrodes, 30 min operation and current density of 15 A/m^2 for both electrodes.

The Effect of Interelectrode Distance

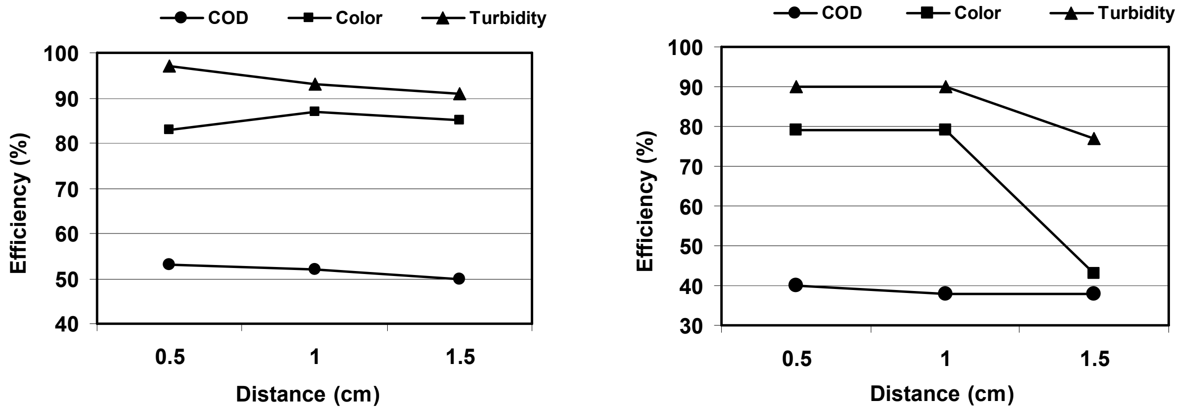
The greater the interelectrode distance, the greater should be the difference in applied potential, because the solution presents higher resistivity to the electrical current. According to the characteristics of the effluent, the process efficiency can be improved by varying the distance between the electrodes (Crespilho and Rezende, 2004). This is an important variable with regards to operational costs. In the case of high values of the conductivity of the effluent, it is recommended that, when the conductivity of the effluent is relatively high, greater interelectrode distance be used. In the case of moderate values, it is recommended to use a smaller distance, as this will reduce the energy consumption.

When the interelectrode distance increases, the removal efficiencies also increase. This change probably occurs due to electrostatic effects, since they depend upon this distance; when it increases, the ion movement is slower and there would be greater opportunity to generate aggregate flakes. Moreover, these flakes are able to adsorb more molecules (Daneshvar et al., 2004).

In the experiment with the variation of the interelectrode distance (Figure 3), the distance between the aluminum electrodes did not cause a significant increase in the removal efficiency of contaminants. On the other hand, the distance between iron electrodes influenced the electrofloculation process: the worst removal efficiency of the contaminants occurred with the distance between iron electrodes of 1.5 cm. When the interelectrode distance increases, the electrical resistance of the solution also increases, and the ions produced move more slowly. Hence, the voltage for obtaining the optimum current density increases, thus increasing the power consumption.

The Effect of Current Density

With the increase of the current density from 10 A/m^2 to 15 A/m^2 (Table 2), higher values of removal efficiencies were obtained. The higher removal efficiency of contaminants with increased current density was due to the higher amount of ions produced on the electrodes that promote destabilization of the pollutant molecules and, finally, the aggregation of the induced flocs, while increasing hydrogen evolution. However, the increase of the current density causes higher consumption of the anode material.



(a) (b)

Figure 3: Effect of distance between the electrodes on COD, color and turbidity removal by (a) aluminum electrodes, initial pH 5 and 30 min operation, and (b) iron electrodes, initial pH 7 and 30 min operation; current density 15 A/m^2 for both electrodes.

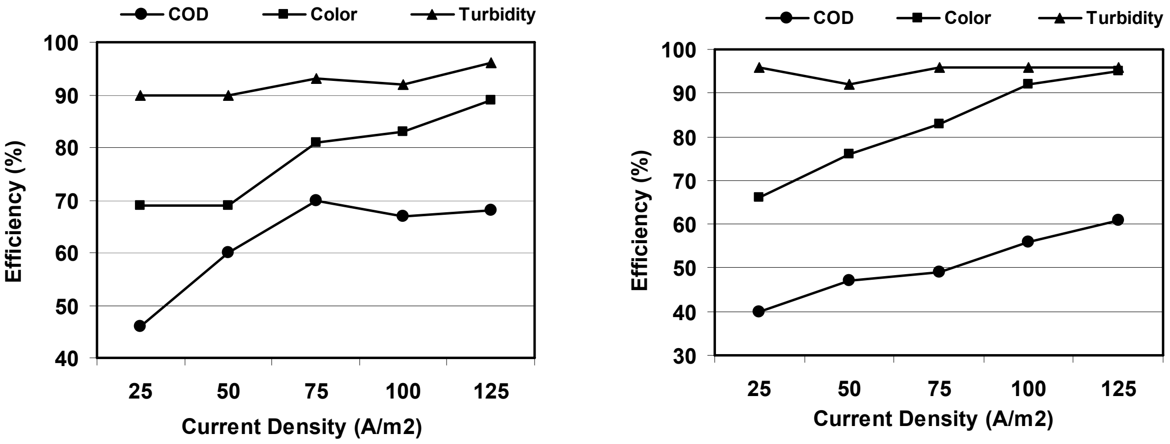
Table 2: Effect of current density on efficiency of electrofloculation of textile effluents

Parameter	Al[a]		Fe[b]	
	10 A/m^2	**15 A/m^2**	**10 A/m^2**	**15 A/m^2**
COD	51%	87%	48%	71%
Color	86%	90%	31%	84%
Turbidity	98%	98%	56%	98%

a: initial pH 5; time: 30 minutes; b: initial pH 7; time: 30 minutes

Using current densities of the order of 25 to 125 A/m^2, preliminary results indicate that the operation time can be substantially reduced from 30 to 10 min, resulting in even higher values of removal efficiencies of COD, color and turbidity (Figure 4). It is important to point out that the electroflocculation cell suffered modification aiming at increasing its power capacity, making it able to carry out assays at higher values of current density and decrease the electrolysis time. It may be seen that higher current density values probably result in a higher pollutant removal efficiency.

Using the iron electrode, the results of removal efficiencies are shown in Figure 4b. Varying the current density from 25 to 125 A/m^2, the best removal efficiencies of COD, color and turbidity using the iron electrode occurred at this largest value. In contrast to the aluminum electrode results shown in Figure 4a, in this case it is valid to apply the highest value of current density. Although implying higher energy and electrode consumption, under these conditions higher removals of color (95%), turbidity (96%) and COD (61%) are achieved. In Figures 5 and 6, tests run with raw effluent are shown for both aluminum and iron electrodes with differents current densities.



(a) (b)

Figure 4: Effect of the variation of the current density using (a) aluminum electrode, initial pH 5; and (b) iron electrode, initial pH 7, with 10 min of electroflocculation.

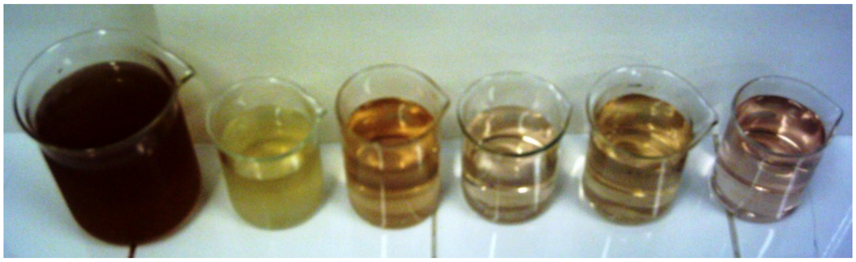


Figure 5: Effect of the variation of the current density (from left to right - raw effluent; treated effluent with aluminum electrode with 25; 50; 75; 100 and 125 A/m^2, respectively)

Figure 6: Effect of the variation of the current density (from left to right – raw effluent; treated effluent with iron electrode with 25; 50; 75; 100 and 125 A/m^2, respectively).

The increase of the supplied current density from 25 to 125 A/m^2 led to better values of COD and turbidity removal, but this is not economically interesting due to the higher electrode and energy consumption.

An electroflocculation using the hybrid electrode (Fe+Al) was developed with the purpose of a simultaneous release of both iron and aluminum ions into the solution and to assess its effect on the treated effluent. This hybrid electrode was constructed with 8 plates, 4 being aluminum and 4 iron plates. The experiment was run at an initial pH of 6, which is a intermediate value of pH used for the iron and aluminum electrodes, i.e., pH 5 and pH 7, respectively.

With variation of the current density from 25 to 125 A/m^2, as shown in Figure 7, it can be seen that the best removal efficiencies of COD (56 and 55%), color (81 and 88%) and turbidity (96 and 97%) are obtained in the range of 100 and 125 A/m^2,. As far as pollutant removal is concerned, the results obtained with the hybrid electrode are very promising.

In Figure 8 it is shown raw and treated effluent using hybrid electrode.

The mass of aluminum electrode consumed during electrolysis with application of a current density of 75 A/m^2 was 0.168 g.L^{-1}, and the consumption of electric energy 0.00208 kwh.L^{-1}, whereas the mass of iron electrode consumed during electrolysis with application of a current density of 125 A/m^2 was 0.870 g.L^{-1}, and the consumption of electric energy was 0.005 kWh.L^{-1}.

The consumed masses of hybrid electrode during electrolysis with application of current densities of 100 and 125 A/m^2 were 0.458 and 0.575 g.L^{-1} respectively, and the consumptions of electric energy were 0.00416 and 0.00625 kWh.L^{-1}, respectively.

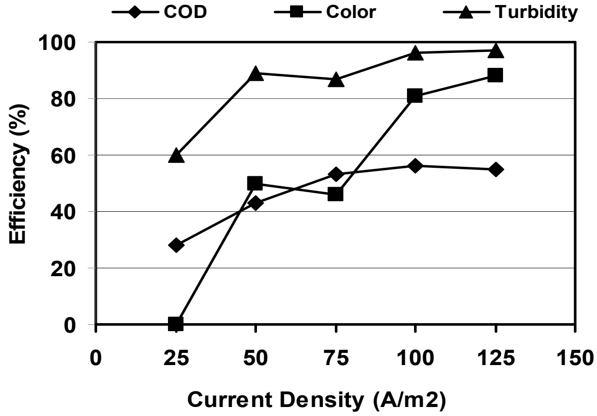


Figure 7: Effect of the variation of the current density, using a hybrid electrode, initial pH 6 and 10 minutes of operation.

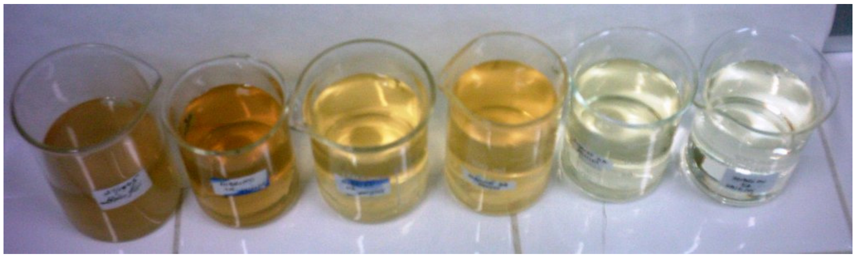


Figure 8: Effect of the variation of the current density (from left to right – raw effluent; treated effluent with hybrid electrode with 25; 50; 75; 100 and 125 A/m^2, respectively)

Evaluation of Residual Al and Fe in the Sample of Raw Sewage and After Electroflocculation

Using, respectively, aluminum and iron electrodes, concentrations of these ions, measured with ICPS (Inductively Coupled Plasma Spectrophotometer), both in raw sewage and after electroflocculation samples are shown in Table 3 and Table 4.

For the case of aluminum electrodes, both aluminum and iron concentrations in electroflocculated samples were lower than in the raw sewage samples. This appears to show that electroflocculation also removes both ions present in the solution. For the run with the highest value of the applied current density (125 A/m^2), 0.23 mg/L was obtained, which is higher than the legislation prescribes. See CONAMA 357/2005.

From this table, it can be seen that, for all runs, the residual aluminum and iron concentrations after electroflocculation were lower than the concentration measured in the raw sewage, thus indicating that both ions were also removed from the solution.

For the tests using hybrid electrodes, namely aluminum and iron, the results are presented in Table 5.

It can be seen that only in 2 cases, the residual iron concentration was well below that measured in raw sewage samples and the residual iron concentration fits well the prescribed iron concentration for disposal in the environment, which is 5 mg/L. On the other hand, the residual aluminum concentration that was obtained for a current density of 125 A/m^2, namely 7.2 mg/L, is well above the prescribed concentration, namely 0.2 mg/L.

Table 3: Aluminum and Iron Concentration in Raw Sewage and Electroflocculated using Aluminum Electrodes

Conditions of EF	Al (mg/L) residual	Fe (mg/L) residual
	0.61[a]	0.43[b]
75 A/m^2 / t = 10 min / initial pH 5	0.14	0.05
100 A/m^2 / t = 10 min / initial pH 5	0.20	0.18
125 A/m^2 / t = 10 min / initial pH 5	0.23	0.03

a: raw effluent with residual Al
b: raw effluent with residual Fe

Table 4: Aluminum and Iron Concentration in Raw Sewage and Electroflocculated using Iron Electrodes

Conditions of EF	Al (mg/L) residual	Fe (mg/L) residual
	0.61[a]	0.43[b]
100 A/m^2 / t = 10 min / initial pH 7	0.08	0.36
125 A/m^2 / t = 10 min / initial pH 7	0.06	0.20

a: raw effluent with residual Al
b: raw effluent with residual Fe

Table 5: Aluminum and Iron Concentration in Raw Sewage and Electroflocculated using Aluminum and Iron Hybrid Electrodes.

Conditions of EF	Al (mg/L) residual	Fe (mg/L) residual
	0.49[a]	0.63[b]
100 A/m^2 / t = 10 min / initial pH 6	0.95	0.07
125 A/m^2 / t = 10 min / initial pH 6	7.19	0.10

a: raw effluent with residual Al
b: raw effluent with residual Fe

Table 6: Comparison between the EF Process used in the Present Work and the Physical-chemical Process used in the Textile Industry

Parameter	Raw Effluent (mg/L)	Electroflocculation treatment (mg/L)		Physical-chemical treatment (mg/L)
		Electrode of Al	Electrode of Fe	Settling tank
COD	(a) 1428	515	643	600
	(b) 1770	832	973	708
	(c) 1340	616	683	603
	(d) 1348	404	526	620

Comparison of the EF Treatment Process with the Process Currently Used in the Textile Industry

Table 6 presents the results obtained with the EF process and that currently used in the textile industry (physical-chemical process).

As can be seen from Table 6, the electroflocculation process, using an aluminum electrode, presented better results [(a) 515 mg/L] as compared with the physico-chemical process [(d) 404 mg/L]. In the runs using iron electrodes, this was only true for case (d), where the COD removal was 526 mg/L, a result above that prescribed in the legislation. This means that a subsequent treatment should be applied in order to adjust the wastewater characteristics to those prescribed in the legislation for discharge into bodies of water or for reuse.

CONCLUSION

The quality parameters of wastewaters of a textile industry of hemp manufacture are highly variable, high amplitude of variation of the organic load, as measured in terms of COD, color and turbidity. Because these variations interfere in the process efficiency, equalization of the effluent was recommended.

The system of batch electroflocculation presented the higher removal efficiencies for COD, color, and turbidity using aluminum electrodes at pH 5 and iron electrodes at pH 7.

With respect to the materials used for the manufacture of the electrodes, one cannot determine a priori the choice in accordance with the type of effluent. This option obligatorily will have to pass through preliminary studies in a pilot reactor to determine which material will lead to the highest pollutant removal efficiency.

The efficiency of pollutant removal by the electroflocculation process depends on the initial pH, the distance between the electrodes, the material of the electrodes, and the current density.

A major problem for treating textile industry effluent is the great temporal variation of the pollutant loads that are generated by the industry, due to the variability of staple fibers and the dyes employed in the process.

Best results for the efficiency of pollutant removal (COD 70%, color 81% and turbidity 93%) using aluminum electrodes were obtained under the following conditions: current of 75 A/m^2, contact time of 10 minutes, distance between the electrodes of 0.5 cm and initial pH 5. With iron electrodes, the efficiencies were COD 61%, color 95% and turbidity 96% with a current of 125 A/m^2, time of 10 minutes, distance between the electrodes of 0.5 cm and initial pH 7.

These results illustrate the effectiveness of the electroflocculation (EF) process in the treatment of textile effluents. Moreover, considering that EF generates a coagulating agent *in situ* that forms flakes of particulate material and, at the same time, carries out flotation of the generated residue, this process is particularly interesting from the point of view of relative simplicity of automation and low cost.

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