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EFFECT OF ELECTROCOAGULATION ON ZETA POTENTIAL DURING TREATMENT OF ACIDIC TEXTILE WASTEWATER

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ABSTRACT

The study focused on determining the optimal conditions for better treatment by electrocoagulation in "batch" mode using an iron anode to treat acidic dye discharges. Particular attention is paid to the effect of electrocoagulation on the behavior of flocs through the measurement of their zeta potential. The results obtained made it possible to advance hypotheses on the mechanism governing the purification of acidic textile wastewater by electrocoagulation. The influence of various parameters on the quality of treatment and on the value of the zeta potential such as the initial pH of the solution, the electrolysis time and the electrolysis current density, were studied. Indeed, for a pH = 7, the elimination of the color reaches 100% while the chemical oxygen demand (COD) only reaches 43% for an electrolysis time of 5 minutes and a current density of 24 mA/cm².

KEYWORDS: Electrocoagulation, textile wastewater, iron electrode, zeta potential, mechanism.

1. INTRODUCTION

Wastewater from the textile industry contains a variety of organic and mineral substances such as cellulose fibers, dyes, softeners, mineral substances (sulfates, chlorides, ammonium

...). The mixture of these substances makes the effluent very complex with a high organic and mineral load which can generate potential nuisances for the environment.

Liquid textile discharges are generally suitable for physicochemical or biological treatment. Biological treatment is often inefficient for the removal of dyes that have a high polymerized structure with low biodegradability. However, several physicochemical techniques such as chemical coagulation (Verma et al. 2012; Freitas et al. 2015), adsorption on adsorbent materials (Aljeboree et al. 2017; Sharma et al. 2019) and membrane processes (Lafi et al. 2018) are also used for the treatment of textile effluents. These techniques are limited by the high concentration of dyes in the rejects. Another major disadvantage in the use of chemical coagulation is the addition of new compounds to the rejects. More recently, ozonation (Lafi et al. 2018) and photo-oxidation (Somensi et al. 2010; Lucas and Peres 2007; Sapkal et al. 2012) have been proposed as an alternative solution because they are considered very effective. But the high cost of these methods leads one to think of other processes. Indeed, electrochemical methods have shown their effectiveness in the treatment of dye discharges (Orts et al. 2018; Samarghandi et al. 2020; Körbahti and Tanyolaç 2009). For dyes, which have high water solubility and low molecular weight, traditional electrochemical methods are no longer effective.

The electrocoagulation technique is one of the modern electrochemical technologies in wastewater treatment.This process, characterized by simple equipment, easy operation and low levels of sludge, uses electrode, usually constituted by iron or aluminium, to releases coagulant.The coagulant is dissolved from the anode, with a simultaneous formation of hydroxyl ions and hydrogen gas occurring at the cathode. This method has been successfully applied for the treatment of various industrial wastewaters (Mollah et al. 2010; Akbal and Camcidotless 2011; Khandegar and Saroha 2013; Tezcan Un et al. 2014; Harinarayanan Nampoothiri et al. 2016; Wang et al. 2016; Nandi and Patel 2017; Huang et al. 2017; Nidheesh and Singh 2017; Maher et al. 2019; Ulu et al. 2019; Xu et al. 2019).

In this study, we tried to treat acidic dyeing effluent by electrocoagulation using the iron electrode. A detailed experimental investigation has been carried out to observe the effects of different operational parameters such as time of electrolysis, current density and pH. Further, the zeta potential of formed flocs has been measured to determine the governing mechanism of removal of pollutants (organic matter and dyes) and the type of iron hydroxide formed.

2. MATERIEL AND METHODS

The experiments are carried outwith a volume of 100 mL of acidic dyeing effluent recovered directly from the reactor of textile dyeing operation, from the commercial textile industry located in Casablanca, Morocco. The main characteristics of this water are grouped in the following table (Table 1).

Table 1: Characteristics of acidic dyeing effluent.

Parameter	Value
pH	4.53
Temperature (°C)	100
Conductivity (ms cm^{-1})	12.7
COD (mg/L)	1912.5
A_{436} nm	0.604
A_{525} nm	0.351
A_{620} nm	0.224

The experimental setup that we adopted in this study includes two iron electrodes (anode and cathode). The surface of the anode is 6.4 cm². The reference electrode is a saturated calomel electrode. A potentiostat (VoltaLab, PGZ 100) is used to apply the desired current. A magnetic stirrer is used to homogenize the solution during electrolysis. The pH of the solution is adjusted by adding HCl or NaOH with high purity.

The chemical coagulation has been carried out by using $FeSO_4.7 H_2O$ and $FeCl_3.6 H_2O$. At the end of the experiment, the solution is filtered before analysis. Chemical oxygen demand (COD) is measured using COD reactor with direct reading spectrophotometer (AL 32, Aqualitic). Iron concentration is determined by atomic absorption spectroscopy (AAS, Varian model AA- 20). Apparent colour of samples is determined by measuring the absorbance at 436, 525 and 620 nm (Zaroual et al. 2006) by using Heλiosγ UV–vis spectrometer. A Malvern zeta potential analyzer (model zetasizer 3000 HS, Malvern Instrument)is used to determine zeta potential of flocs formed during electrolysis.

3. RESULTS AND DISCUSSION

Several operating parameters affect the electrocoagulation process such as time of the electrolysis, current density and pH. Current density and electrolysis time determines the coagulant production rate, total production of coagulant (Fe^{2+} or Fe^{3+} ions), size and growth of the flocs, which can affect the efficiency ofelectrocaugualation. The pH solution plays a key role in the performance of the electrocoagulation. The maximum removal efficiency is

obtained at an optimum pH for a particular pollutant. The precipitation of a pollutant begins at a particular pH (Khandegar and Saroha 2013).

3.1. Effect of electrolysis time

The electrocoagulation experiments are carried out with a current density of 16 mA/cm² at different time intervals. At the end of each experiment, the coloring of the solution became more intense and there was no precipitate formation. The quality of treatment is controlled by measuring the absorbance of effluent at three wavelengths 436 nm, 525 nm and 620 nm (Figure 1-a). The results show that the absorbance of the solution increases with the electrolysis time. Moreover, during electrolysis the final pH of the solution and the iron concentration (Figure 1-b), increases with time.
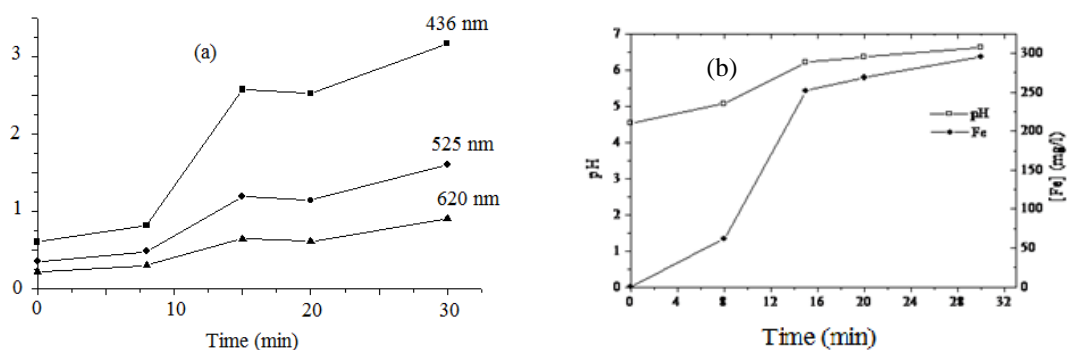
Figure 1: Evolution of (a) the absorbance, (b) of iron concentration and pH of the effluent as a function of the electrolysis time at i = 16 mA/cm².

3.3. Effect of current density

To study the effect of the current density to degrade textile effluents, other electrocoagulation experiments were performed at an electrolysis time of 15 minutes and varying current densities. The coloring of the solutions obtained after the electrolysis is more intense and no precipitate formation was observed. Figure (2-a) shows the evolution of the absorbance as a function of current density. The absorbance of the solution also increases with electrolysis time despite the pH rise of the effluent (Figure 2-a). Furthermore, we note an increase in the concentration of dissolved iron in the solution (Figure 2-b).
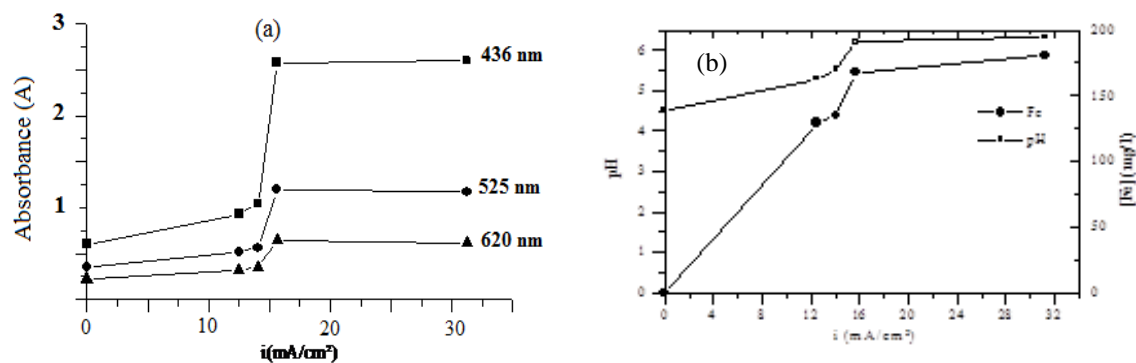
Figure 2: Evolution of (a): the absorbance (b): iron concentration and pH as a function of current density at time = 15 min.

The results obtained show that the absorbance of the solution increases with electrolysis time and current density. This behavior can be explained by the presence of iron ions in the solution or by another secondary oxidation reaction that took place. To verify these hypotheses, we performed chemical coagulation tests with ferrous ions and ferric ions using iron sulfate ($FeSO_4.7\ H_2O$) and ferric chloride ($FeCl_3.6\ H_2O$), in addition to electrochemical oxidation on a platinum electrode. The chemical coagulation is realized with an amount of iron ions equivalent to the amount of dissolved iron ions for a time of 15 minutes and a current density of 31mA/cm². This quantity is determined by the weighing anode or by applying faraday's law. The electrochemical oxidation, was performed for 15 minutes of electrolysis time and a current density of 31mA/cm². The results obtained are grouped in the following table (Table 2).

Table 2: Treatment results of electrocoagulation, chemical coagulation and electrochemical oxidation.

|	Effluent without treatment	Electrocoagulation	Chemical Coagulation with $FeSO_4.7H_2O$	Chemical Coagulation with $FeCl_3.6\ H_2O$	Electrochemical oxidation by platinum electrode
Final pH	4.53	6.6	6.2	2.3	6.6
A 436 nm	0.604	3.175	3. 212	0.406	0.866
A 525 nm	0.351	1.608	1.411	0.164	0.606
A 620 nm	0.224	0.908	0.659	0.07	0.454

Examining this table, we find that chemical coagulation with ferric ions leads to a decrease in pH with a decrease in absorbance. While the addition of ferrous ions and electrochemical oxidation leads to an increase in pH to a value similar to that of electrocoagulation tests and an increase in absorbance. The increase of the absorbance with the ferrous ions is greater than

with the electrochemical oxidation. This absorbance is comparable to that of electrocoagulation, which makes it possible to conclude that the increase in absorbance observed during the electrocoagulation tests is due to the presence of ferrous ions in the medium, probably in a complexed form.

3.3. Effect of the initial pH of the solution

Several works have shown that pH is a sensitive operation parameter in the removal of pollutants by electrocoagulation, which could significantly influence the direction of various chemical reactions. Since the type of iron hydroxide species formed in the solution and the surface charge of the particle depend on the pH.

To evaluate the effect of the initial pH on the elimination of dyes and organic matter by electrocoagulation, the experiments were performed at a current density of 31 mA / cm² for 15 minutes at different initial pH from 2 to 10. The evolution of the iron ions concentration and the solution pH with initial pH value is given in figure (3).
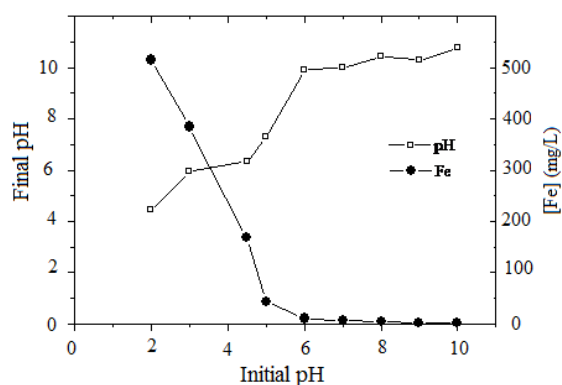
Figure 3: Evolution of iron concentration and final pH versus initial pH (i= 31 mA/cm^2, t = 15min).

Figure (3) shows that the dissolved iron ions remain in the solution, which is in agreement with a study of Stephenson and Duff (Stephenson and Duff 1996). From a pH equal to 6, the solution becomes free of iron. On the other hand, after electrolysis, the increase in the pH of the solutionis greater in an acid medium (two units) than in basic medium (one unit)which agrees with the literature (Harinarayanan Nampoothiri et al. 2016). This increase in pH can be explained by the occurrence of water electrolysis resulting in hydrogen evolution and production of OH$^-$ ions. Moreover, some anions such as NO_3^-, HCO_3^-, SO_4^{2-} and Cl^- that are present in wastewater can as well replace the hydroxide in $Fe(OH)_2$ or $Fe(OH)_3$ and release

OH⁻ which is also one of the factors of pH increase.The evolution of the COD was followed and the absorbance as a function of the initial pH. The results obtained are illustrated in Figure (4).
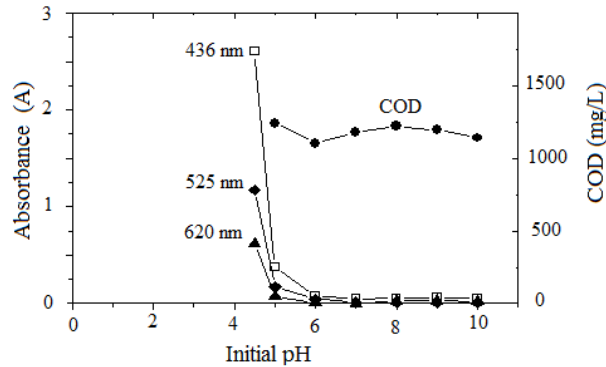
Figure 4: Evolution of the absorbance and the COD as a function of the initial pH of the solution pH (i= 31 mA/cm^2, t = 15min).

It can be seen that the initial pH has a significant effect on dye removal efficiency. At the initial pH of effluent, the minimum removal of dyes is observed. Beyond pH = 6 the total elimination of dyes is reached. Similar observation is reported by other investigations (Nandi and Patel 2017). However, the reduction of the COD is independent of the initial pH which makes it possible to say that in addition to the electrocoagulation, the COD can be eliminated by the electrochemical oxidation.

To demonstrate that the reduction of the coloring matter is done by the electrocoagulation and not by the change of pH solution, the absorbance for the three wavelengths at different pH was measured. The results obtained are given in figure 5 showed that the absorbance of the solution is independent of pH. Consequently, the color removal is essentially due to electrocoagulation.
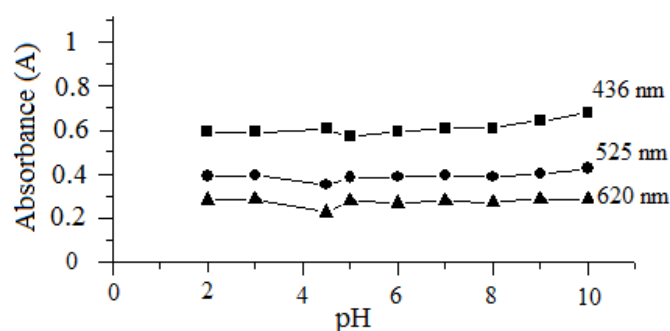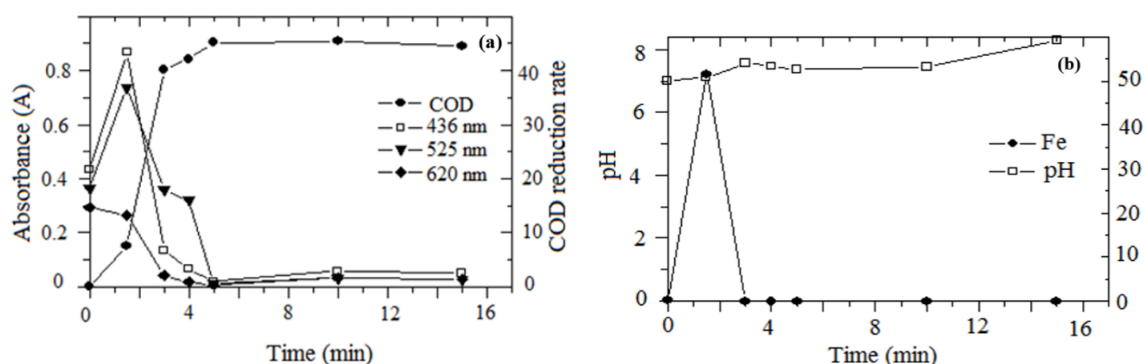

Figure 5: Evolution of the absorbance as a function of the solution pH.

3.4. Determination of optimum conditions at neutral pH

3.4.1. Electrolysis time

To determine the optimum electrocoagulation time for the acidic dyeing effluent, we carried out tests at different times and at a current density of 31mA/cm² by adjusting, beforehand, the initial pH of the solution pH at 7. We followed, on the one hand, the evolution of the COD abatement rate and the absorbance (Figure 6-a) and on the other hand, the pH variation and the iron content in the solution (Figure 6-b).

Figure 6: Evolution of (a)COD reduction rate and the absorbance, (b) Evolution of iron concentration and pH as a function of electrolysis time at initial pH = 7 (i = 31 mA/cm²).

Figure (6-a) shows that the absorbance of treated effluent presents peaks for all three wavelengths at low time. From time of 5 minutes the absorbance becomes zero. The same result is obtained by Do and Chen (Stephenson and Duff 1996) in the treatment of reactive and dispersed dyes. The degree of reduction of the COD increases with electrolysis time and stabilizes at a value of 43% from a time of 5 minutes. In Figure (6-b) we observe an increase in the solution pH with the electrolysis time and high iron content at low time, which explains the increase of the absorbance at this time. This content becomes zero from time of 3 minutes.

3.4.2. Current density of electrolysis

To determine the optimum current density, electrocoagulation experiments are carried out as previously at the initial pH of the solution equal to 7 during an electrolysis time of 5 minutes (optimum time obtained) and at different current density. The quality of treatment is monitoring by measuring the COD abatement rate and absorbance (Figure 7-a). From Figure 11 we notice that the absorbance of the solution is important at low current density and cancels when the current density increases (from i ≥24 mA/cm²). We also find that the COD

percentage reduction increases with the current density and stabilize at an average value of 43% when the current density is greater or equal to 24 mA/cm². In figure (7-b) we represent the evolution of pH and dissolved iron with the density of the current. This figure shows, on the one hand, an increase in pH with the current density, on the other hand high content of iron at low current, which explains the increase of the absorbance at these densities. The iron charge becomes zero as the density of the current increases.
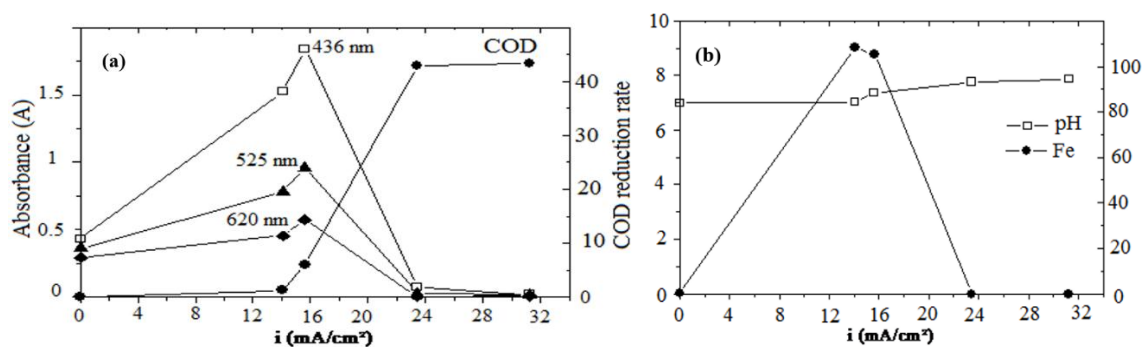

Figure 7: Evolution of: (a) COD reduction rate and of the absorbance, (b) concentration of iron and pH according to the density of the current at time = 5 min.

3.4.3. Zeta potential Measurement

There are several different mechanisms of aggregation that can occur during electrocoagulation and determining which mechanism governs the electrocoagulation process is difficult. The most commonly used technique for characterizing the stability colloid suspensions is the measurement of particle zeta potential. The zeta potential is an important measure of the electrical potential at the floc surface, and the magnitude and sign of the zeta potential control the electrostatic interactions betweenthe floc surface and polar species, other charged interfaces and particles in suspension (Al Mahrouqi et al. 2017).

To understand and know the mechanism governing the removal ofthe pollutant from acidic textile wastewater byelectrocoagulation, the zeta potential of the flocs formed during the treatment was measured.
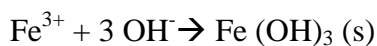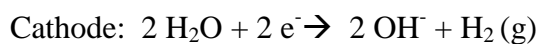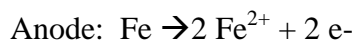
The results obtained are illustrated in figure (8a-b). Figure 8 shows that the formed flocs are charged negatively.This charge increases with the electrolysis time. After 5 minutes this charge stabilizes and the zeta potentialvalue is -22 mV (Figure 8-a). However, zeta potential reaches this value and remains stable from a current density of 24 mA / cm² (Figure 8-b).
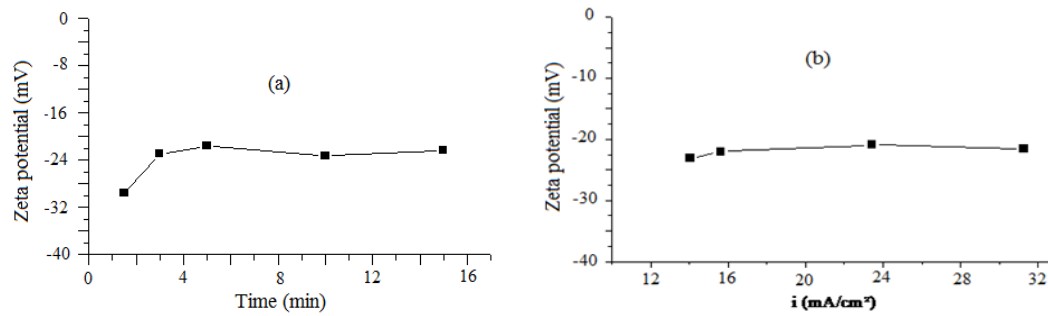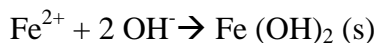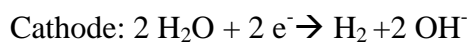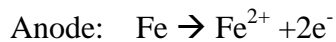
Figure 8: Evolution of the zeta potential of the formed flocs: (a) with the electrolysis time, (b) with the current density.

Generally, during the electrocoagulation process with an iron anode, two mechanisms are proposed for the formation of iron hydroxide:

Mechanism I:

Anode: Fe $\rightarrow$ 2 Fe^{2+} + 2 e-

Fe^{2+} + 1/2 O$_2$ + H$^+$ $\rightarrow$ Fe^{3+} + OH$^-$

Cathode: 2 H$_2$O + 2 e$^-$ $\rightarrow$ 2 OH$^-$ + H$_2$ (g)

Fe^{3+} + 3 OH$^-$ $\rightarrow$ Fe (OH)$_3$ (s)

Mechanism II:

Anode: Fe $\rightarrow$ Fe^{2+} +2e$^-$

Cathode: 2 H$_2$O + 2 e$^-$ $\rightarrow$ H$_2$ +2 OH$^-$

Fe^{2+} + 2 OH$^-$ $\rightarrow$ Fe (OH)$_2$ (s)

During the electrolysis the solution becomes green. After a settling time, we obtain a total clarification of the solution with the formation of green mud. The surface layer of this sludge becomes yellow - brown as the decantation time progresses. The green color of the mud allows us to say that we probably had the formation of Fe (II) hydroxide (mechanism II) which oxidizes, at the surface, with time by the oxygen of the air in Fe (III) hydroxide (yellow - brown color, mechanism I). In previous work (Zaroual et al. 2006), we have demonstrated that at pH = 8, the value of the zeta potential of Fe (II) hydroxide is -35 mV while for the Fe(III) hydroxide is - 4 mV. After the electrolysis, the pH of the solution is about 8. The zeta potential values for iron hydroxide flocs formed during the electrocoagulation tests are comparable with those of Fe (II) hydroxide at pH = 8. Therefore, it is likely that we have the formation of Fe (II) hydroxide during the electrocoagulation experiments. Before reaching the optimum condition (5 min, 24 mA/cm²), we have a change in the charge of the flocs formed with electrolysis time and current density (it becomes more

positive). Hence, the repulsive electrostatic force is decreased while the van der Waals force is dominant. This later has an action of approaching and flocculating the pollutants by an increase of the negative zeta potentials. When the electrolysis time, or the current density, exceeds 5 minutes or 24 mA/cm² respectively, the zeta potential remains stable. Also, we notice the stabilization of the absorbance and the COD. For all electrocoagulation experiments, the pH final is near or equal to 8. Further, the value of zeta potential is negative what suggests that $Fe(OH)_4^-$ species should be formed in the bulk. The same trend of zeta potential is observed by A. Raschitor and al (Raschitor et al. 2014).

The iron peak observed at low electrolysis time (Figure 5-b) (or low current density (Figure 6-b) can be explained by the fact that this time (or this current density) is not sufficient to lead to the total precipitation of iron. But when the electrolysis time (or the density of the current) increases, the iron content decreases until zero because the pH of the medium (pH = 8) is favorable to the total precipitation of iron in the form of $Fe(OH)_2$ solid.

4. CONCLUSION

In this work, we have studied the possibility of treating acidic dyeing effluent, containing a variety of textile dyes and complex chemicals, by electrocoagulation in a batch system. The results obtained show that the electrocoagulation treatment efficiency is controlled by the electrolysis current density, the electrolysis time and the pH of the solution. The investigations revealed that virtually complete color and 43% COD removal efficiency was achieved in the electrocoagulation process employing iron as the electrode at its optimized operating conditions. The measure of zeta potential allows determining the type of iron hydroxide formed as well as the electrocoagulation mechanism.

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