

## Electrocoagulation/Electroflotation of real textile effluent: Improvement of the process in non-conventional pilot external loop airlift reactor

Mohammed Elhafdi<sup>a,b,\*</sup>, Mohamed Benchikhi<sup>c</sup>, Aabderrahim Dassa<sup>b</sup>, Hachemi Chenik<sup>a,b</sup>, Aabdelfahid Essadki<sup>a</sup>, Mohamed Azzit<sup>b</sup>

<sup>(a)</sup> Laboratoire d'Environnement, Procédés, Electrochimie (LEPE), Université Hassan II, Ecole supérieure de Technologie, BP.8012 Oasis, Casablanca, Maroc

<sup>(b)</sup> Laboratoire d'Interface, Matériaux et Environnement (LIME), Faculté des Sciences Ain Chock, B.P 5366 Maarif, Casablanca 20000, Morocco. Casablanca, Maroc.

<sup>(c)</sup> Laboratoire Physico-chimie des Matériaux Inorganiques (LPCMI), Faculté des sciences Ain chock, B.P 5366 Maarif, Casablanca 20000, Maroc.

<sup>(a)\*Corresponding author:</sup>

elhafdi.moh@gmail.com

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### Abstract

A pilot external-loop airlift reactor (ELAR) of 150 liters was designed and used as a non – conventional reactor to carry out Electrocoagulation/Electroflotation to treat real textile effluents containing disperse and reactive dyes. The designed reactor ensure the recovery of sludge by electroflotation (EF) in which complete flotation of the pollutants is achieved without additional mechanical power in the pilot external-loop airlift reactor (ELAR), using only the overall liquid recirculation induced by H<sub>2</sub> microbubbles generated by water electrolysis without filtration process. Aluminum, iron electrodes and combined aluminum – iron electrodes were tested. The obtained results were interesting as they would help managing the Electrocoagulation/Electroflotation process in pilot external – loop airlift reactor to remove real textile effluent. The treatment of the mixtures of the real textile dyeing industry is better when using a combination of the electrodes of iron and aluminum providing a better treatment efficiency of 80% and a lower specific energy consumption (50 kWh/kg dye). In order to analyze the by-product of the electrocoagulation (EC) and the treated effluent, different techniques were used to elucidate the role of different kind of anodes especially when the combined iron – aluminum were used simultaneously as sacrificial anodes.

**Keywords:** ; Pilot external-loop airlift reactor; Electrocoagulation; Electroflotation; Real textile effluents

## 1.Introduction

Pollutant flows have become for a long time very difficult for the environment to eliminate naturally. Thus, the management of the water cycle is essential for a sustainable water supply. It became necessary for research teams to work out an integrated solution that takes into account all requirements and meets the global demand for water [1], this may be achieved through the increase of water reuse and recycling. Purification of water intended for human consumption is more and more required. Cleaning wastewater from industrial effluents before discharging is also a challenging work. In fact, innovative, cheap and effective techniques have to be developed. Industrial effluents when they are biodegradable are generally biologically treated, but for many industries this remains impossible especially because of the toxicity of the non-biodegradable pollutants. However, industrial water treatment technologies must be able to operate over a wide range of pollutants. Also, small technologies and those of outsourced processing are often preferable to a centralized treatment. As centralized processing requires expensive transport networks from the point of view of the investment operation. Furthermore, the mixture of different water qualities decreases the treatment ability of a mixture of pollutants. Thus, small local water treatment facilities are required for much industrial pollution. Chemical coagulation is still one of the heavily used techniques but some of its drawbacks are induced by the presence of metal salts, acidification of the treated water and the large amount of sludge disposal. The color removal in textile discharges has been extensively studied: coagulation-flocculation [2-4], biological treatment [5], advanced chemical oxidation such as UV, H<sub>2</sub>O<sub>2</sub>, and O<sub>3</sub> oxidations [6-8], adsorption on adsorbent materials such as activated carbon [9] or membrane processes were effective, but in most cases very costly. An interesting alternative to these methods is the electrocoagulation (EC). EC is an electrochemical method for treating polluted water which has been successfully applied for treatment of soluble or colloidal pollutants, such as wastewater containing heavy metals, emulsions, suspensions, etc., but also drinking water for lead or fluoride removal. EC is considered one of the most effective methods of treatment of wastewater, especially textile effluents [10-14]. A typical EC unit includes an EC cell/reactor, a separator for settling or flotation, and often a filtration step. Indeed, the benefits of EC include simplicity, efficiency, environmental compatibility, safety, selectivity, flexibility and cost effectiveness [13-25]. In particular, the main points involve the reduction of sludge generation, the minimization of chemicals addition and little space requirements due to shorter residence time. During electrocoagulation, the coagulant is released into the solution in situ, using the electrochemical dissolution of a sacrificial anode. When feeding of current electrodes, there is a simultaneous dissolution of the anode metal and hydrogen (H<sub>2</sub>) gas near the cathode. Aluminum and iron metals are generally used as the anode. Their dissolution results in the formation of metal hydroxides, metal oxy-hydroxides and polymeric hydroxides. These compounds generally have better coagulation efficiency than that of the chemicals used in conventional techniques. They can destabilize the colloidal suspension neutralizing or precipitating polluting species dissolved in the liquid, and finally transform easily removable flocks by precipitation, filtration or flotation. Generally, flotation is easier to achieve than filtration and precipitation. For this, electrocoagulation can be assisted by the injection of compressed air or performed only by hydrogen H<sub>2</sub> produced by the cathode. The main deficiency is the lack of dominant reactor designs and scale-up purposes. Most papers use laboratory-scale EC cells but the separation step by floatation/sedimentation is not studied. So an innovative reactor has to be developed in order to optimize the cost of this process. The first configuration of external loop airlift reactor to realize electrocoagulation and electroflotation (EC/EF) was proposed by Essadki et al. [26] in which the capacity is 20 Liters. Hydrogen bubbles produced at the cathode are used to float the flocks, the density difference between the riser section containing the liquid-gas mixture and the downcomer containing only the liquid causes a liquid flow that performs agitation. Another study [27] for defluoridation was conducted for comparison, using (EC/EF) in two reactors in a batch mode: stirred reactor in a conventional cell and an external-loop airlift reactor. The same reactor was used by Balla et al. [28] to

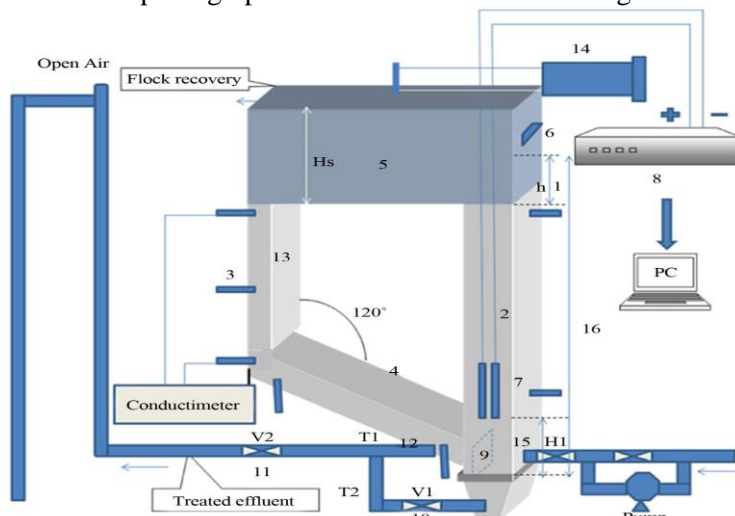
study the efficiency of EC/EF in removing a color from synthetic and real textile wastewater by using aluminum and iron electrodes in an external-loop airlift reactor of 20 L. In the same airlift reactor, Essadki et al. [29] performed a detailed study of the residence time distribution (RTD). A pilot external-loop airlift reactor of 150 L was designed to operate in batch and continuous modes. The objective is to analyze the potential of external –loop reactors to perform Electrocoagulation /Electroflotation in real conditions. Physicochemical characterization of the obtained flock during this operation using iron, aluminum and the combination of both anodes is carried out to comprehend the role of each kind of anode, the aim of this work is to give information in order to help managing electrocoagulation by using optimal conditions in industrial environment.

## 2.Methodology

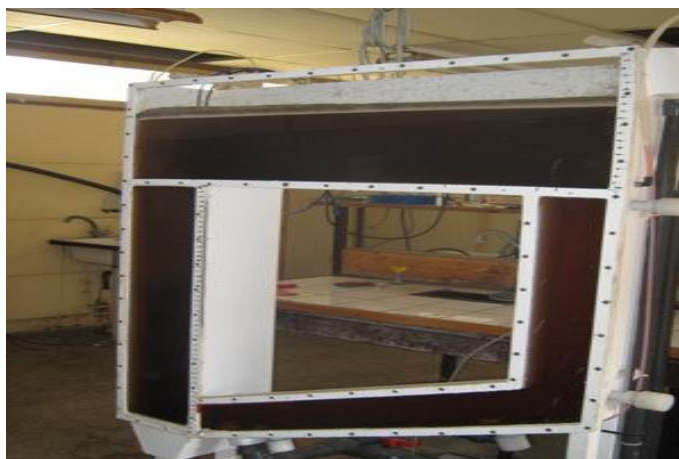
### 2.1. Designed reactor

A pilot external loop airlift reactor was designed by taking into account the simulation results. Thus, the stability of the flock to realize electroflotation was performed when the electrodes are positioned in a certain axial position of the riser section. The maximum capacity of the reactor is 150 liters (L). Thus, depending on the height  $h$  of the liquid in the separator zone, the volume of the treated effluent is ranged between 100 to 150 liters.

**Figure 1** shows the detailed reactor. A photograph of this reactor is shown in Figure 2.



**Figure 1:** External-loop airlift reactor (1: level of the effluent in the separator, 2: riser section, 3: conductivity probe, 4: connecting column, 5: separation section, 6: slot, 7: electrodes, 8: generator, 9: region closed to the liquid passage, 10: valve V1, 11: valve V2, 12: tube T1, 13: downcomer section, 14: scraping system, 15: H1: electrodes position).



**Figure 2:** photography of the External-loop airlift reactor : 150 L

The electrodes (aluminum and/or iron) were placed in the riser. The shape of each electrode is rectangular (232 mm × 165 mm × 1 mm) which corresponds to an electrode surface area of  $S = 1050 \text{ cm}^2$ . The distance between electrodes was  $e = 20 \text{ mm}$ , which is a typical value in electrocoagulation (EC) cells. The axial position of the electrode could also be varied in the column. The distance (H1) between the bottom of the electrodes and the bottom of the riser was chosen to be  $H1 = 54 \text{ cm}$  according to the induced liquid velocity that permit the stability of the flock. EC was conducted in the intensiostat mode, using a digital DC power “Power supply EA-9080 PSI”. Two options are considered: a batch operation and a continuous operation. The choice of one of the options can be achieved by handling valves. The effluent is recovered without flock since the later is formed by electroflotation in the separator section and recovered by overflow (6) or by a scraping system (14). So the filtration is avoided decreasing the cost of the process. In addition, since the liquid is induced electrochemically by inserting electrodes in an optimal position in the riser, the power of mixing is not needed.

## 2.2 Chemicals and analytical methods

Spectrophotometer (UV-Visible spectrometer Helios  $\gamma$ , type Gamma) was used to determine dye concentration by its absorbance characteristics in the UV-vis range (200-800 nm). The maximum intensity of absorbance was obtained by using the wavelengths  $\lambda_{\text{max}} = 436, 525$  and  $620 \text{ nm}$  covering the absorbance of various types constituting the mixture. The standard closed reflux colorimetric method was used to measure Chemical Oxygen Demand (COD). Initial pH was measured with a pH meter (PHM 220 Radiometer analytical). Conductivity was determined by a conductivity meter (“Cyberscan 510-cond” “Conductimeter Consort K 120”). Absorbance was measured every 10 min to follow the efficiency evolution. Colour and COD removal efficiencies ( $Y_{\text{COL}}$ ,  $Y_{\text{COD}}$ ) were expressed as follows:

$$Y_{\text{COL}}(\%) = \frac{A_0 - A}{A_0} \quad (1)$$

$$Y_{\text{COD}}(\%) = \frac{(COD)_0 - COD}{(COD)_0} \quad (2)$$

The specific electrical energy consumption per kg dye removed ( $E_{\text{dye}}$ ) was calculated as follows:

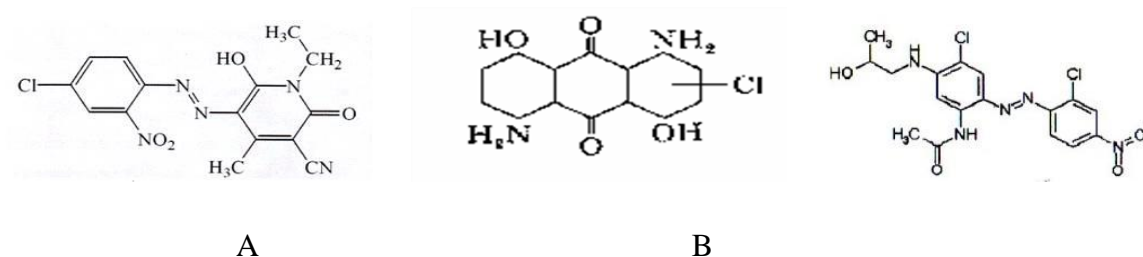
$$E_{\text{dye}} = \frac{U I t}{1000 V (C_0 Y_{\text{COL}})} \quad (3)$$

Where  $E$ : energy consumption (kWh/kg dye);  $I$ : current intensity (A);  $U$  voltage (V);  $t$ : time (h);  $V$ : volume ( $\text{m}^3$ ),  $C_0$ (kg/ $\text{m}^3$ ): initial dye concentration. In Equation (1),  $A_0$  and  $A$  are respectively the initial absorbance and the absorbance at a certain time. In Equation (2),  $(COD)_0$  and  $COD$  are respectively the initial  $(COD)_0$  and the  $COD$  at a certain time expressed in kg/L. The real wastewaters have been collected from two containers. One containing disperse dyes (acidic pH), and a second one collected from containing reactive dyes (basic pH). The reactive dyes contained Yellow SPD, Red S3B 195, Blue BRF 150%. The disperse dyes contained Yellow Terasil 4G, Red disperse and Blue Terasil 150%. The real wastewaters were provided from ITEX, Textile industry (Casablanca, Morocco). The characteristic of the real wastewaters are shown in Table 1.

**Table 1:** Characteristics of the real dyes used in ITEX (Casablanca, Morocco)

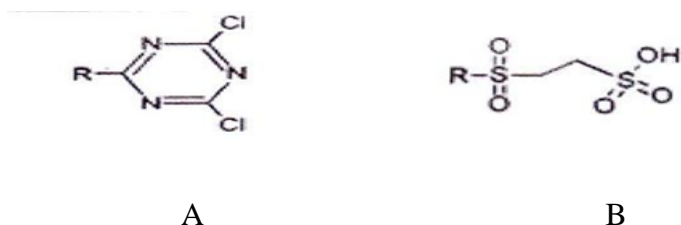
Parameters	Values
Temperature (°C)	29.5
pH	11.46
Conductivity at 20°C (μS/cm)	1040
BDO5 (mg/L)	405
COD (mg/L)	951
Total Nitrogen (mg/L)	8.1
Fe (mg/L)	1.15
Zn (mg/L)	0.16
Pb (mg/L)	0.037

Disperse dyes are acidic dyes partially soluble in water, used to dye polyester. The chemical structures of the dyes used are represented in Figure 3.



**Figure 3.** Molecular structure of the constituents of the dye: (A) Yellow terasil 4G,; (B) Blue terasil 3R02 (C) Red terasil 343 150%

The reactive dyes are soluble and basic in-water type, used for dyeing cotton. The reactive dyes used are bi-functional, they comprise two main groups, the dichlorotriazine and vinyl sulfone of structure (Figure 4).



**Figure 4:** Molecular structure of the reactive dye: (A) DICHLOROTRIAZINE; (B) VINYL SULFONE.

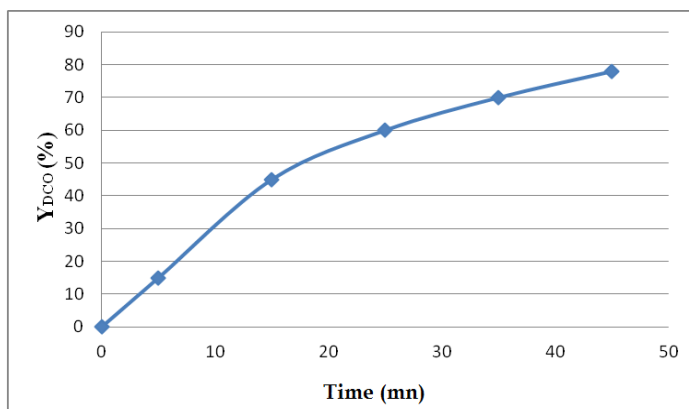
The sludge was recovered at the end of the electrolysis. Each sample was dried at 105°C for 2 h in order to remove water from the samples before weighing to estimate the amount of dried sludge formed by EF. The mineralogical structure of the dried sludge was analyzed using an X-ray diffractometer (DRX, Philips X'Pert Pro) with a Cu(K<sub>α</sub>) source. This technique is able to identify crystalline phases and to show the presence of amorphous compounds. The morphology of the solid particles of the sludge was analyzed by scanning electron microscopy (SEM). Energy dispersive X-ray analysis (EDX) was coupled to SEM and allowed a simultaneous access to an estimation of the

elemental composition of the sludge. A more precise quantitative analysis was carried out using a similar method, but based on a wavelength dispersive X-ray fluorescence analyzer (XRF-WDS, Philips X'Pert Pro) that is able to measure elemental concentration from ppm to 100%. This technique gives a superior order of magnitude and a better spectral resolution, sensitivity and ability to determine concentrations of light elements in a more efficient way than with EDX. The dried sludge was then calcined using 500°C as the oven temperature. The same analysis was done with the calcined flock. Inductively Coupled Plasma coupled with Optical Emission Spectroscopy (ICP-OES) analysis was also used to determine the total soluble aluminum or iron content firstly in water, and then in the solubilized flock.

### 3. Results and discussion

#### 3.1 Treatment of real reactive dyes

The studied dyes were collected directly from machines, just at the end of the bath dyeing cotton cloth. At this stage, the baths are not diluted, so they have a high degree of pollution and maximum coloration, with an initial COD of 1230 mg O<sub>2</sub>/l. Discharges cotton dye baths by the reactive dye, are basic (pH = 11.6) and have high conductivity ( $\kappa$  = 25 mS/cm), because during the dyeing process, salts such as sodium chloride and sodium carbonate are added. When using aluminum electrodes, the efficiency does not exceed 40% (not presented here). It tends to stabilize after 60 minutes of electrolysis. Whereas, when the iron was used as an anode electrode, the rate of reduction in COD value reached 75% after 45 min of electrolysis as shown in Figure 5.



**Figure 5:** Evolution of the COD abatement versus time (real effluent: reactive dye). Initial pH = 11.6, conductivity = 25.3 mS/cm, current density = 31.4 mA/cm<sup>2</sup>, iron anode.

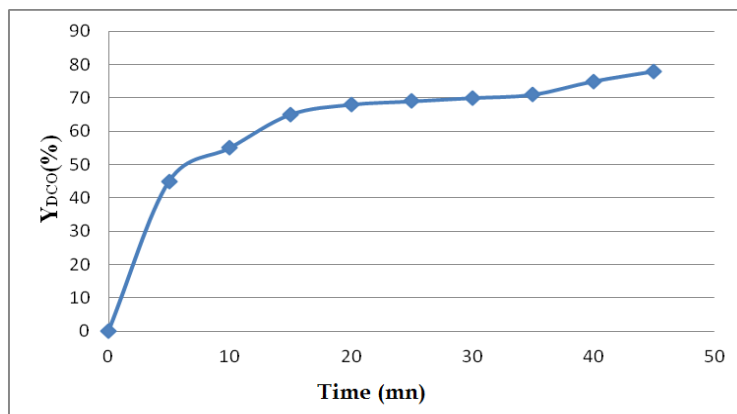
It is noted that the use of iron electrodes is more effective than the aluminum electrodes for the discharge of reactive dyes. This is probably due to the high pH of the solution (11.6). Thus, according to the speciation diagram of two metals (aluminum and iron), there is more Fe(OH)<sub>3</sub> than Al(OH)<sub>3</sub> in the same pH [30]. This confirms the adsorption character of electrocoagulation in which the adsorbents are Fe(OH)<sub>3</sub> or Al(OH)<sub>3</sub>.

#### 3.2 Treatment of real effluent of disperse dyes

Disperse dyes are used on the polyester. As above, a representative sample is taken directly to the valves of the machine, just at the end of the polyester fabric dye bath. Discharges of disperse dyes have characteristics that do not facilitate treatment by electroflotation. These unfavorable parameters are acidity and conductivity of the medium (pH = 3.8 and low conductivity,  $\kappa$  = 1.1 mS/cm). Hence, the need to adjust these two parameters before treatment is crucial. This adjustment can lead to a conductive solution and a level of acidity that allows favorable conditions for



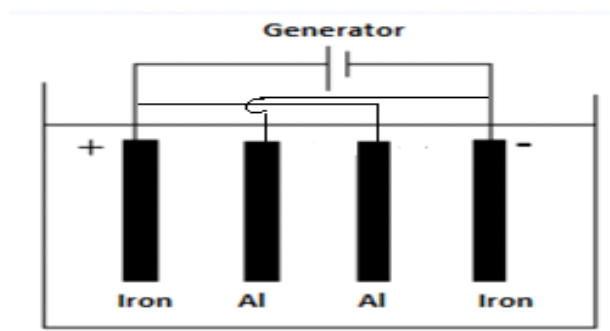
EC/EF. It is noted that, in the case of discharge only containing disperse dyes, the use of an aluminum anode allows better treatment than the use of an iron anode. As shown in Figure 6, the reduction in COD value reached 80% after an electrolysis time of 45 min.



**Figure 6:** Evolution of the COD abatement versus time (real effluent: disperse dye). Initial pH = 6.4, conductivity = 2.1 mS/cm, current density = 31.4 mA/cm<sup>2</sup>, aluminum anode.

### 3.3 Treatment of mixture real textile effluent

Treatment of real discharges textile, consisting only of the reactants or disperse dyestuffs, is difficult. That is the reason of performing the treatment of the overall mixture in order to compensate for the low pH and conductivity (□) values of the disperse dyes effluents. The treatment of the mixture containing disperse and reactive, were carried out using an iron anode, an aluminum anode or a combination of anodes made of iron and aluminum connected with a monopolar mode. Monopolar electrodes in parallel connections (Figure 7) were chosen because they give a good efficiency as suggested by preliminary experiments.

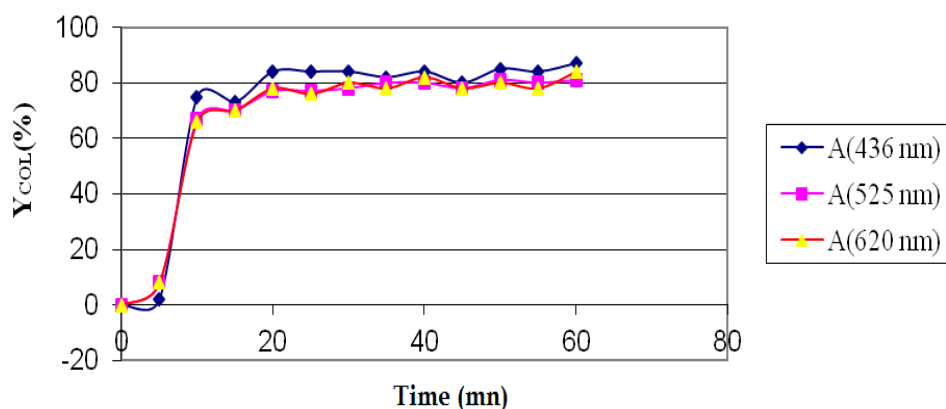


**Figure 7:** Monopolar electrodes in parallel connections : iron/aluminum.

**Figure 8** shows the evolution of the efficiency of decolorization of the real effluent using a combination of iron-aluminum anodes. It is observed that the efficiency reached 80% after an electrolysis time of 20 minutes and then stabilized to the same value. It is noted also that the efficiency used for a wavelength of 436 nm is greater than that corresponding to when one uses both other wavelengths. This can be explained by the fact that the kinetics of decolorization are not the same for each component of the dye. However, the rate of reduction of the COD increases with time to reach 80% after an electrolysis time of 60 minutes.

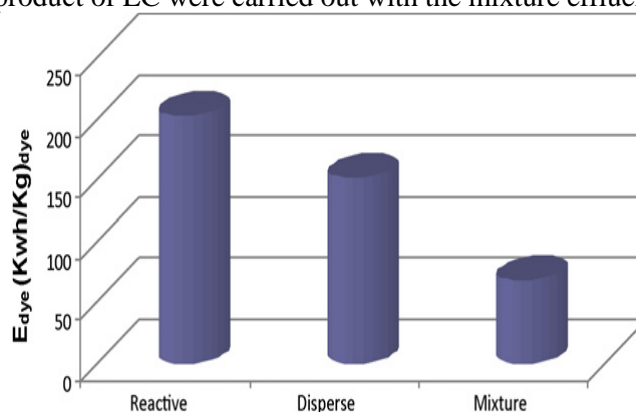
The use of a combination of the electrodes of iron and aluminum is allowed to have an electroflotation processing efficiency better than in the case where the anode consists only of iron or aluminum [31-32].

The specific energy consumption was also measured. Figure 9 shows the energy consumed for the three types of effluents: disperse, reactive and mixed. This figure shows that specific energy consumed is lower for the case of a mixture (50 kWh/kg dye), followed by the dispersed (120 kWh/kg dye) type and reactive (170 kWh/kg dye) type. For industrial processing, real mixture would be very beneficial. This is due to the compensation phenomenon that occurs between the reactive dyes (very high initial conductivity) and the disperse dyes (initial pH and initial conductivity very low).



**Figure 8:** Evolution of the decolorization efficiency of real textile effluent versus time (combined iron - aluminum anodes,  $pH_i$  (initial) = 6.90,  $k = 6.50$  mS/cm,  $j = 31.4$  mA/cm<sup>2</sup>).

The characterization of the by-product of EC were carried out with the mixture effluent (disperse and reactive dyes).



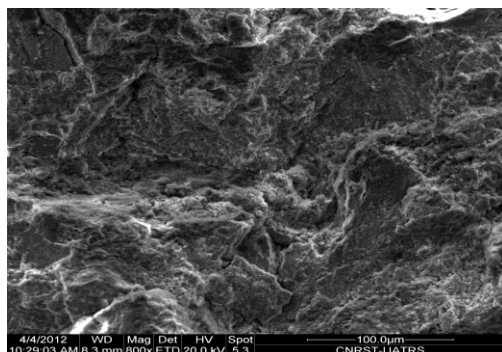
**Figure 9:** Specific electrical energy consumption per kg dye removed ( $E_{dye}$ ) in optimal conditions for real effluents: reactive, disperse and mixture dyes.

### 3.4 Characterization of the by-products and the treated solution obtained from the EC

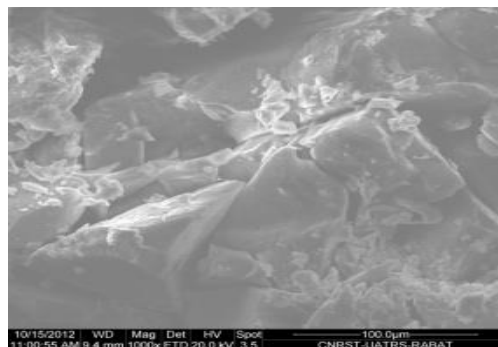
In order to understand the nature of removal of dye, the EC flocks were characterized with X-Ray Fluorescence (XRF), X-Ray Diffractometry (XRD), Scanning Electron Microscopy (SEM) coupled with EDX. ICP technique was also used for either sludge or liquid to determine the amount of aluminum or/and iron. The flocks formed are recovered to make investigations in terms of characterization and analysis. After electroflotation, the flocks are drained by suction filtration water. The solid is dried in an oven at 105°C to measure its humidity and then analyzed. It was then calcined at 500°C to assess the organic and mineral matter content. First, the recovered flocks are examined under a scanning electron microscope after drying at 105°C and after calcination at 500°C. SEM images of the dried



and calcined flocks correspond respectively to Figure 10-a and 10-b. The observation of these images reveals a fibrous, amorphous structure of the dried flock. The calcined flocks have shapes in regular geometric structures [33].



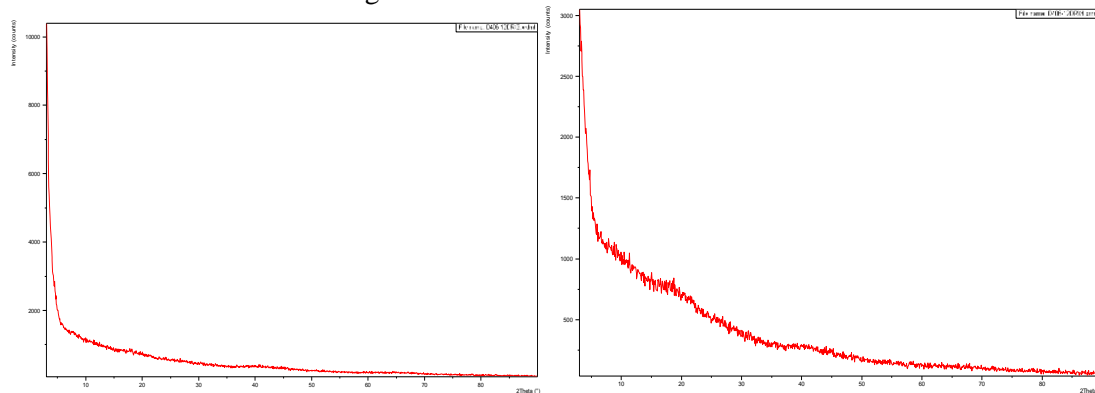
a: Dried sludge



b: Calcined sludge

**Figure 10:** SEM micrographs

XRD diffractograms showed that in both cases, insoluble amorphous compounds were formed and no crystalline phase could be identified as observed in Figure 11-a and 11-b.

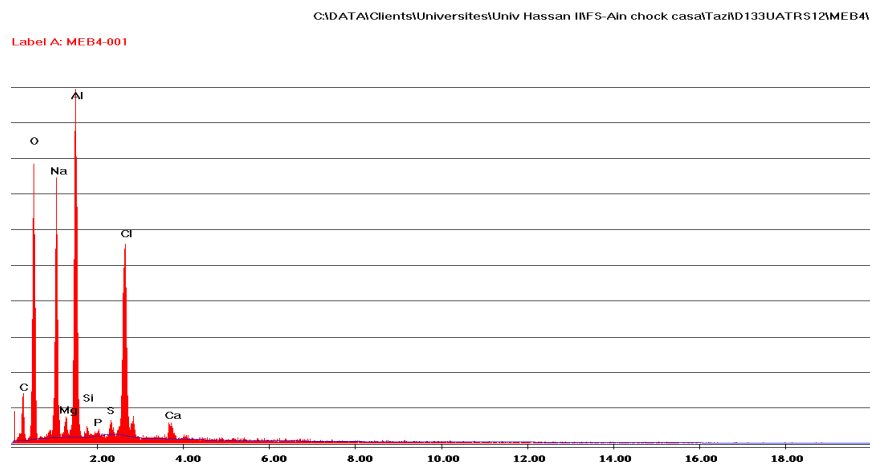


**a**

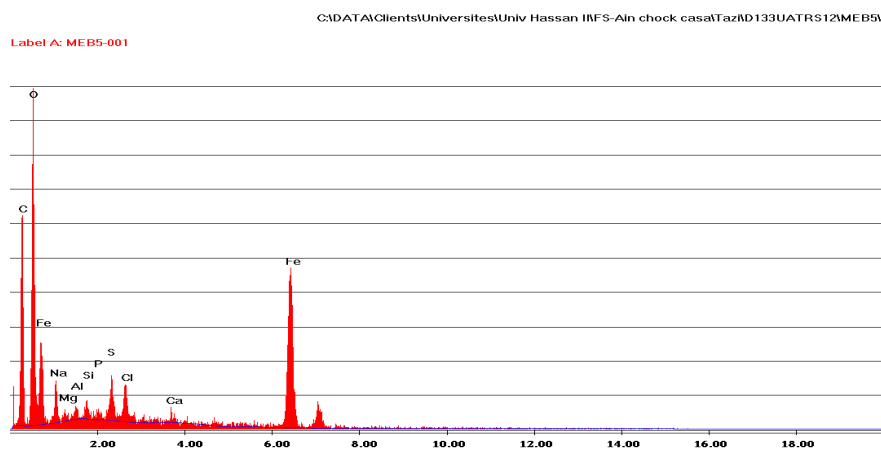
**b**

**Figure 11:** Diffractograms obtained by XRD for: (a) dried sludge et (b) calcined sludge

Then, two solid samples of flocks were collected for analysis. One sample (A) corresponds to the case of treatment using aluminum electrodes, whereas, the other sample (B) corresponds to the case of treatment using iron electrodes. The analytical result by EDX elemental analysis for dried flocks shows that they consist of an organic material characterized by the presence of carbon and an inorganic material represented by various other elements (Figures 12 and 13). The elemental analysis by XRF is presented in Tables 2 and 3. The origin of the organic matter is from the dyes used in the textile industry.



**Figure 12:** EDX spectrum of dried sludge in the case of aluminum anode (initial pH = 6.90,  $k = 6.50 \text{ mS/cm}$ ,  $j = 31.4 \text{ mA/cm}^2$ ).



**Figure 13:** EDX spectrum of dried sludge in the case of iron anode (initial pH = 6.90,  $k = 6.50 \text{ mS/cm}$ ,  $j = 31.4 \text{ mA/cm}^2$ ).

**Table 2:** Estimation of the composition of the sludge by XRF-WDS obtained at the end of the operation ( $t = 45 \text{ min}$ ) for dried sludge in the case of aluminum anode: (initial pH = 6.90,  $k = 6.50 \text{ mS/cm}$ ,  $j = 31.4 \text{ mA/cm}^2$ )

Elements	Wt(%)	Atoms (%)
C	21.37	32.15
O	34.14	38.57
Na	13.42	10.56
Mg	1.05	0.78
Al	15.76	10.55
Si	0.72	0.46
P	0.47	0.27
S	0.83	0.46
Cl	10.84	5.54
Ca	1.40	0.63
Total	100	100

**Table 3:** Estimation of the composition of the dried sludge by XRF-WDS obtained at the end of the operation ( $t = 45$  min) for dried sludge in the case of iron anode: (initial pH = 6.90,  $k = 6.50$  mS/cm,  $j = 31.4$  mA/cm<sup>2</sup>).

Elements	Wt %	Atoms %
C	41.66	58.32
O	28.94	30.41
Na	2.65	1.93
Mg	0.43	0.3
Al	0.57	0.35
Si	0.52	0.32
P	0.44	0.24
S	1.59	0.83
Cl	1.46	0.69
Ca	0.58	0.24
Fe	21.21	6.39
Total	100	100

The analysis of the calcined flocks obtained by X-ray fluorescence (XRF) of sample A are shown in Table 4 and those of sample B are shown in Table 5. The examination of the two samples analysis results shows that the weight of the metal from the dissolution of the anode is an important part of the total mass in the sludge. It represents almost the 1/3 of the calcined flocks weight. The flocks from the treatment contain, in addition to the organic matter of textile colorants, the mineral material consisting of various components initially in release and which are removed during the electroflotation process.

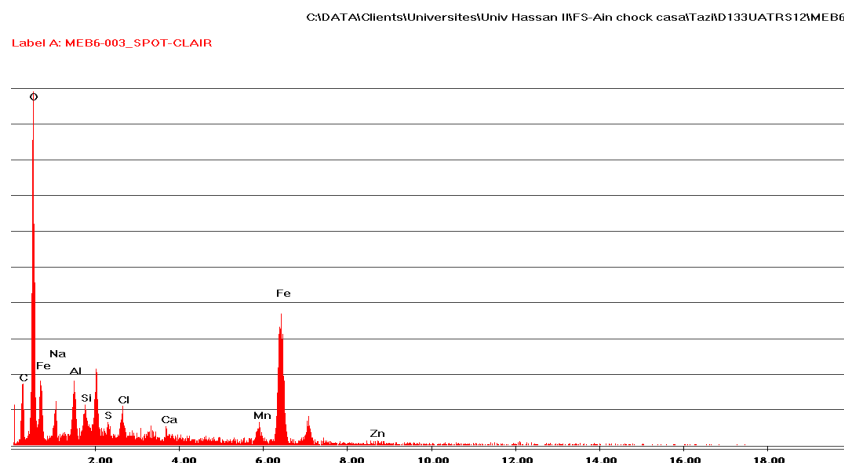
**Table 4:** Estimation of the composition of the calcined sludge by XRF-WDS obtained at the end of the operation ( $t = 45$  min) for dried sludge in the case of aluminum anode: (initial pH = 6.90,  $k = 6.50$  mS/cm,  $j = 31.4$  mA/cm<sup>2</sup>).

Compound	Al <sub>2</sub> O <sub>3</sub>	Fire loss	Na <sub>2</sub> O	SO <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Cr <sub>2</sub> O <sub>3</sub>	MgO
Wt %	39.6	21.7	12.1	10.9	6.44	3.12	2.19	1.65
Element	O	Al	Na	C	Fe	S	Cr	Si
Wt %	49.6	21	9.01	5.93	4.5	4.37	1.5	1.46

**Table 5:** Estimation of the composition of the calcined sludge by XRF-WDS obtained at the end of the operation ( $t = 45$ min) for dried sludge in the case of iron anode: (initial pH = 6.90,  $k = 6.50$  mS/cm,  $j = 31.4$  mA/cm<sup>2</sup>).

Compound	Fe <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	Fire loss	SO <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	ZnO	P <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>
Wt %	43.5	21.6	17.5	3.77	2.81	2.4	1.98	1.62	1.56
Element	O	Fe	Na	C	Zn	S	Al	P	Cr
Wt %	44.7	30.4	9.44	4.77	1.93	1.51	1.49	1.47	1.11

In the case of the combination of aluminum and iron electrodes, the presence of these metals in the flock is demonstrated in Table 6 describing XRF analysis. This shows that the aluminum and iron are oxidized simultaneously during the electrolysis to generate coagulant species ( $\text{Fe}^{3+}$  and  $\text{Al}^{3+}$ ). It is also noted that the percentage of iron in the flocks is greater than that of aluminum for the current density of  $31.4 \text{ mA/cm}^2$  used. Figure 14 shows EDX elemental analysis. It indicates that the  $\text{Fe}^{3+}$  ions have a more important coagulant character than  $\text{Al}^{3+}$  ions one. In the case of a combined anode Iron-Al, iron tends to oxidize more easily than aluminum even if the latter is thermodynamically more electropositive especially as aluminum tends to passivate in the pH range of neutrality.



**Figure 14:** EDX spectrum of dried sludge in the case of combined aluminum - iron anodes (initial pH = 6.90,  $k = 6.50 \text{ mS/cm}$ ,  $j = 31.4 \text{ mA/cm}^2$ )

**Table 6:** Estimation of the composition of the dried sludge by XRF-WDS obtained at the end of the operation ( $t = 45 \text{ min}$ ) in the case of combined iron-aluminum anode : (initial pH = 6.90,  $k = 6.50 \text{ mS/cm}$ ,  $j = 31.4 \text{ mA/cm}^2$ )

Elements	Wt (%)	Atom (%)
C	26.065	37.2575
O	39.6825	44.5025
Na	6.9475	5.18
Al	9.005	5.9975
Si	0.795	0.535
S	0.87	0.4725
Cl	1.15	0.625
Ca	0.385	0.1875
Mn	0.735	0.2725
Fe	13.1775	4.64
Zn	1.195	0.33
Total	100	100

The ICP was also used to analyze and determine the amount of aluminum and iron contained in the flock. Thus, the sludge was solubilized using HCl. The ICP analysis of the amounts of iron and aluminum in each sample is given in

Table 7. The observation of the results shows that the latter are slightly different than those found by X-ray fluorescence and by EDX. The results of analysis by ICP of the treated effluent using both iron anode and aluminum anodes are given in Table 8. The results show that the treated water contains iron and aluminum in amounts respecting national standards.

**Table 7 :** ICP analysis of solubilized flocks in water.

Sample	Type of anode used	Type of solubilized flocks	Content of (% Wt)	Content of (% Wt)
1	Fe	dried	0.6	22.7
2	Al	dried	20.4	1
3	Fe	Calcined	1	41.5
4	Al	Calcined	32.7	2

**Table 8 :** ICP analysis of treated effluent

Sample	Anode used	Content (mg/l)	Moroccan standards (mg/l)
1	Al	1.82	10
2	Fe	1.985	3

## 4.Conclusion

EC/EF has been successfully realized in a pilot external- loop airlift reactor with the aim of achieving a complete flotation to recover the sludge. The advantage of electroflotation in external loop is that the electrical energy cost is offset by the lack of mobile mechanical or pneumatic for agitation with good mixing induced by only tiny bubbles generated electrochemically by water electrolysis. Another advantage is the lack of filtration that is an investment and a non-negligible maintenance cost. The removal by EC of the mixture of real liquid effluents for dyeing cotton and polyester fabrics is easier and the consumed energy is lesser in comparison to a separate treatment of each kind of dye. In fact, the mixture presents compensation phenomena between its various components. This would approach the initial pH conditions and initial conductivity most favorable to operate economically. In addition, the treatment of the mixture of the real textile dyeing industry is better when using a combination of the electrodes of iron and aluminum, which allowed better treatment efficiency, unlike the case where the anode consists only of iron or aluminum. In this study, treatment with EC/EF using a combination of mixed iron anodes and aluminum showed that the iron is preferentially dissolved in the aluminum probably due to the passivation phenomenon limiting the dissolution of the latter. The analysis by ICP showed that the treated water is in agreement with the national standards.

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