

# Evaluation of Electrochemical and Fenton Processes in the Treatment of Mature Landfill Leachates

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## ABSTRACT

In this study, the effects of various operating conditions (reaction time, current intensity, pH and H<sub>2</sub>O<sub>2</sub> concentration) in treatment of old municipal landfill leachates by using electrochemical processes (electroflocculation, electro-Fenton and combined electroflocculation+electro-Fenton) was compared to Fenton's reagent oxidation process. The results of this work showed that, E-Fenton technology may be used efficiently for treatment of old municipal landfill leachates by using appropriate operating conditions. The best removal efficiency was obtained when the following operating conditions were used: reaction time of 30 minutes, current intensity of 2A, H<sub>2</sub>O<sub>2</sub> concentration of 0.5% and pH=3. In these conditions, the removal efficiencies achieved for chemical oxygen demand, colour and turbidity were, respectively: 70±2%, 92±2% and 92±2%. The results obtained by the present study revealed that the E-Fenton electrochemical process was efficient in the removal of high concentrations of chemical oxygen demand, colour and turbidity and could be an environmentally promising alternative for the treatment of mature landfills leachates. Furthermore, this process showed lower energy cost and consumption of electrodes

**Keywords:** Landfill Leachate, Electro-Fenton, Electroflocculation, Alternating Current and Fenton

## I. INTRODUCTION

Sanitary landfilling, although environmentally inadequate, is the most common alternative to treat old landfill leachate, mainly in countries like China, Brazil, India and others. Leachate is a liquid that has been seeped through the solid waste disposed in a landfill and contains extracted, dissolved or suspended materials [1]. Therefore, developing treatment technologies for landfill leachate requires further research to obtaining an efficient and economically viable process, aiming to reduce its potential to harm water resources, soil and biota.

In old landfills, the leachate is characterized by a high chemical oxygen demand (COD) due to presence of non-biodegradable and high toxic organic compounds [2-6], which requires the use of technologies, that can even be combined to increase their efficiency, to minimize the amount of pollutants to an acceptable level before its discharge into water flow [7, 8]. Among these

technologies, stands out Electroflocculation (EF), Fenton, Electro-Fenton (E-Fenton) and Electroflocculation-Electro-Fenton (EF+E-Fenton) combined processes.

Electroflocculation (EF) is a process wherein flocculating metal ions are electrolytically added to the effluent by an anode and gas micro bubbles are released by a cathode. The flocculating metal ions adhere to water pollutants increasing their size, and the gas micro bubbles capture the flocculated pollutants that float to the surface, from where they can be easily removed. By an appropriate choice of electrode materials, this process can remove a wide variety of pollutants without needing chemicals or filters [9 – 14]. When using direct current (DC) technology, the anode oxidation causes formation of an impermeable oxide layer over the cathode which increases the electrode resistivity. Along the time, the efficiency of EF process declines. This problem can be minimized by addition of sacrificial electrodes in parallel configuration to the electrolytic cell [15, 16]. Furthermore, to reduce the cathode passivation and

extend electrodes lifetime, current direction can be inverted at regular time intervals. Thus, cathode and anode can be switched periodically. Many researchers have preferred using alternating current in EF process. It's assumed that the cyclic energization between the anode–cathode in alternating current (AC) system simulates the manual polarity reversion. It delays the cathode passivation and the anode oxidation, ensuring a reasonable electrode life [17].

Advanced oxidation processes (AOP), such as Fenton, are also widely used to treat high COD levels wastewater. This processes are very efficient methods for organic substances mineralization. The Fenton process employs ferrous ions and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) under acidic pH conditions [18 – 21]. E-Fenton process has been used for treating old landfill leachates [9]. Usually, there are two different types of applications for E-Fenton: the first, called direct reaction, in which ferrous ions and hydrogen peroxide are electrochemically generated in a reactor (the anode and cathode, respectively) and the second, called indirect reaction, where ferrous ions are generated "in situ" from oxidation of an iron sacrificial anode, while hydrogen peroxide is added to the electrochemical cell. Then, Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> react like the Fenton process generating hydroxyl radicals (OH<sup>•</sup>) which are highly reactive [22]. The indirect reaction has been mostly used to decrease reaction time in the treatment of effluents with high organic contents [10, 11, 13]. In comparison to classical Fenton process, E-Fenton offers significant advantages such as lower generation of ferrous ions in cathode and sludge production [9, 11, 23].

The present study aims evaluate and compare the efficiencies of Electroflocculation (EF), Fenton, Electro-Fenton (E-Fenton) and combined Electroflocculation + Electro-Fenton (EF+E-Fenton) processes in the treatment of mature landfill leachates.

## II. METHODS AND MATERIAL

### Mature Landfill leachate characteristics and sampling

Leachate samples from Jardim Gramacho Metropolitan Landfill (Rio de Janeiro state, Brazil) was collected

manually in plastic containers and immediately transported to laboratory, characterized, and kept cooled to 4°C, per the recommended by Standard Methods for the Examination of Water and Wastewater [24]. The physicochemical leachate characteristics are presented in Table 1.

**Table 1** – Characterization of old landfill leachate studied.

Parameter	Measured values (Mean ± SD)
pH	8.7±0.2
COD (mg/L)	2,880±250
Conductivity (µS/cm)	12,040±400
TDS (mg/L)	9,610±350
Salinity (mg/L)	6,565±270
Temperature (°C)	23±2
Color	1.6±0.3
Turbidity (NTU)	42±8
NH <sub>4</sub> <sup>+</sup> (mg/L)	3,306±640
NO <sub>3</sub> <sup>-</sup> (mg/L)	273±80
NO <sub>2</sub> <sup>-</sup> (mg/L)	367±45
Cl <sup>-</sup> (mg/L)	3,590±860
Br <sup>-</sup> (mg/L)	134±12
PO <sub>4</sub> <sup>-</sup> (mg/L)	195±20
SO <sub>4</sub> <sup>-</sup> (mg/L)	925±45
Na (mg/L)	3,079±900
Fe (mg/L)	0.4±0.3
Ca (mg/L)	1,964±100
Ni (mg/L)	0.5±0.1
Pb (mg/L)	0.3±0.2
Cu (mg/L)	0.06±0.03
Cd (mg/L)	0.02±0.01

### Experimental procedure

First, leachate samples were kept at room temperature and shaken for settled solids re-suspension. Then, aliquots of the samples were transferred to electrolytic reactor.

### Electrolytic cell

The electrolytic cell consists in an alternating current source (AC) (Tecnopeltron Co. Ltd, Brazil), a digital magnetic mixer (Heidolph MR 3002-4), an electrolytic reactor (Pyrex glass, volume of 1.5 L), an iron anode and cathode plates (dimensions: 10 x 5 x 0.3 cm) and an unipolar electrode with seven rectangular iron plates (electrode gap = 1 cm) vertically placed [12]. In all tests, the frequency of 60 Hz was used. At the end of each test, the electrolytic reactor and electrodes were cleaned with 5% (v/v) hydrochloric acid solution for at least 15 min., rubbed with a soaker and rinsed with tap water.

### Electroflocculation tests

Experiments were conducted using six different initial pH conditions (from 3.0 to 7.0 and 8.7, which corresponds to the pH of the raw leachate). pH values were adjusted by using  $\text{H}_2\text{SO}_4$  and  $\text{NaOH}$  10 mol  $\text{L}^{-1}$  reagent grade solutions (Merck). Then, the iron electrodes were vertically placed into electrolytic reactor and leachate was thoroughly stirred with a magnetic stirrer. For each pH value, EF tests were performed using current intensities of 2 A, 3 A and 5 A and 30 minutes of reaction time.

### E-Fenton tests

Leachate was submitted to E-Fenton treatment, in electrolytic cell with the addition of 0.50% (v/v) hydrogen peroxide solution. These tests were performed using the reaction times of 10, 20, 30 and 45 minutes.

To study the effects of different  $\text{H}_2\text{O}_2$  concentrations, leachate samples were submitted to E-Fenton treatment in electrolytic cell, with different  $\text{H}_2\text{O}_2$  concentrations (0.25, 0.50, 0.75 and 1.00% v/v), applied in continuous mode using a peristaltic pump (Model D100C, China). These tests were performed using the current intensity of 2 A (best condition obtained from EF trials) and pH value was adjusted to 3.0 [3, 8, 25]. Hydrogen peroxide solutions were prepared using a 30% (v/v)  $\text{H}_2\text{O}_2$  solution (Merck). In the end of each test, reaction was suppressed by using 10 mol  $\text{L}^{-1}$   $\text{NaOH}$  solution, increasing pH into values above 7.0 in order to avoid any residual  $\text{H}_2\text{O}_2$  that could affect COD measurements [26].

### Combined Process Electro flocculation + E-Fenton (EF+E-Fenton) tests

Leachate samples treated by EF (using optimal experimental conditions:  $t = 30$  min;  $\text{pH} = 6.0$ ;  $i = 2$  A) were filtrated, pH value set to 3.0, and then were submitted to E-Fenton treatment, in electrolytic cell, with 0.50% (v/v) hydrogen peroxide. All tests were performed using current intensity of 2 A.

To study the reaction time effect, the leachate treated by EF (in the optimal experimental conditions) was filtrated and submitted to E-Fenton treatment, in electrolytic cell, with 0.50% (v/v) hydrogen peroxide concentration and tests were performed using reaction times of 10, 20, 30 and 45 minutes.

In addition, to study the different  $\text{H}_2\text{O}_2$  concentrations effect, the leachate treated by EF (in the optimal experimental conditions) was filtrated and submitted to E-Fenton, at the electrolytic cell. Tests were performed using different hydrogen peroxide concentrations 0.25, 0.50, 0.75 and 1.00% (v/v) applied in continuous mode using a peristaltic pump (Model D100C, China).

At the end of each test, the reaction was suppressed by using  $\text{NaOH}$  10 mol  $\text{L}^{-1}$  to increase pH into values above 7.0.

### Fenton Tests

Fenton experiments were performed in bench-scale by using a Jar-Testing Apparatus (Model JT 102.6 Milan, Brazil) equipped with 2000 mL beakers. Batch Fenton treatment experiments were conducted using 1000 mL of each leachate sample. Each experiment started by stirring the leachate at 100 rpm, while pH was adjusted to 3.0 to 5.0 levels using  $\text{H}_2\text{SO}_4$  solution (10 mol  $\text{L}^{-1}$ ).

To prepare the Fenton reagent,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  crystals were weighed and dissolved in leachate using the Jar-Test beaker. This solution was stirred by approximately 1 min. and then, hydrogen peroxide was applied in a single step. After 30 minutes, the pH was adjusted into values up to 7 by adding 10 mol  $\text{L}^{-1}$   $\text{NaOH}$  solution. Experimental pH and reagent dosage ratio  $\text{H}_2\text{O}_2/\text{Fe}^{2+}$  was selected based on pH range (3.0–5.0) and optimum medium  $\text{H}_2\text{O}_2/\text{Fe}^{2+}$  (1.8 p/p) [27].

### Analytical Methods

Salinity, pH, electrical conductivity (CE), total dissolved solids (TDS) and temperature were measured using a multipara meter (Oakton PCS Tester 35), chemical oxygen demand (COD) was determined using closed reflux method based on the Standard Methods [24]. Color was evaluated by measurements of absorbance at a wavelength of 400 nm using a spectrophotometer (HACH DR5000) and turbidity was performed with a turbidimeter (Tecnozon TB 1000). The concentrations, nitrogen-ammonium ( $\text{NH}_4^+$ ), nitrate-nitrogen ( $\text{NO}_3^-$ ), nitrite-nitrogen ( $\text{NO}_2^-$ ), chloride ( $\text{Cl}^-$ ), bromide ( $\text{Br}^-$ ), phosphate ( $\text{PO}_4^-$ ) and sulfate ( $\text{SO}_4^-$ ) were determined by anion chromatograph (ICS 3000 Dionex Inc.), equipped with a IonPac<sup>®</sup> CS16 analytical (3×250mm) cation column preceded by two pre-columns and a cation suppressor CSRS 300 (2 mm) and also a AS23 analytical (2×250 mm) anion column preceded by a pre-column and a 300 ASRS anion suppressor (2 mm). Metals concentrations (Fe, Ni, Pb, Cu and Cd) were measured using an atomic absorption spectrophotometer (AA 240 Varian Inc.) [24]. All assays were performed in triplicate.

The removal efficiencies of COD and color was obtained using Equation 1

$$\text{Removal efficiency \%} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

Where  $C_i$  and  $C_f$  refer to the initial and final values of these parameters.

### Energy and mass electrode consumption for laboratory scale Electrolytic treatment of landfill leachate

The energy consumption in the process was calculated by Equation 2:

$$E_{c_{kWh/m^3}} = \frac{V \cdot i \cdot t}{v} \quad (2)$$

Where  $V$  is operating voltage (volt),  $i$  is the operating current (ampere),  $t$  (or  $t_{E_c}$ ) time of reaction (minutes) and  $v$  is volume of the effluent ( $\text{m}^3$ ).

The experimental mass consumption of electrodes (expressed as kg consumed per  $\text{m}^3$  of treated effluent)

was determined by direct measurement of the electrodes mass before and after each experiment on an analytical balance (Mettler AL 204) after washing with distilled water and drying for 30 minutes in an oven.

The theoretical mass consumption of the electrodes, according to Faraday's law, is directly related to the applied current and defined by Equation 3

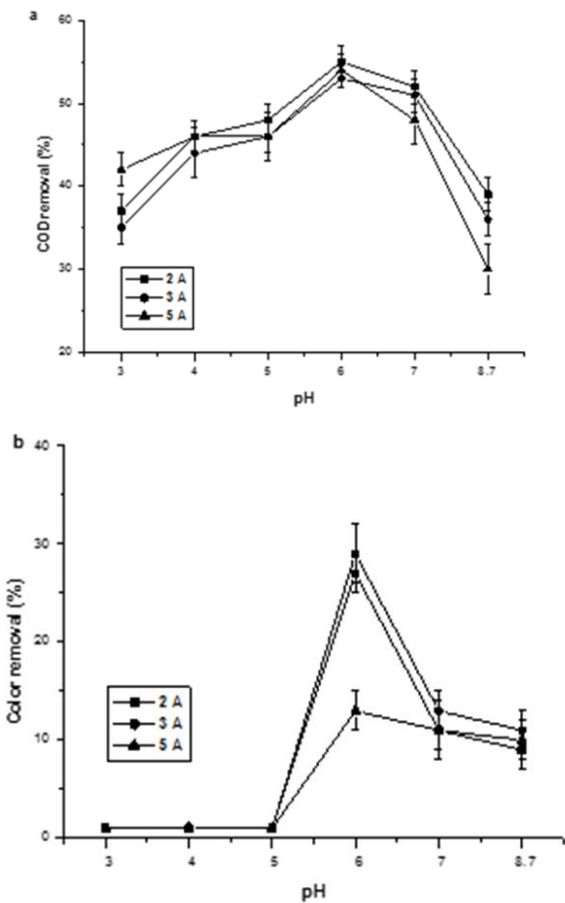
$$m_{el} = \frac{i \cdot t \cdot M}{n \cdot F} \quad (3)$$

Where:  $m_{el}$  is maximum amount of electrode consumed (g);  $i$  is applied current (A);  $t$  is time of electrolysis (s),  $M$  is mass of iron ( $\text{g mol}^{-1}$ );  $n$  is the number of electrons involved in oxidation of anode element;  $F$  is Faraday Constant ( $96500 \text{ C mol}^{-1}$ ).

## III. RESULTS AND DISCUSSION

### Evaluation of pH and current intensity on EF process

Several authors have reported that pH is an important parameter that affects EF efficiency since this parameter influence the speciation of iron ions. Consequently, pH interferes in pollutants flocculation process [28 – 30]. Thus, EF tests were performed varying pH from 3.0 to 7.0 and 8.7 for raw leachate, using different current intensities (2 A, 3 A and 5 A). When the initial pH used was 6, the best COD removal efficiency was observed after 30 minutes of electrolysis (Fig. 1). However, despite the satisfactory COD removal efficiency ( $56 \pm 2\%$ ), color removal efficiency was lower ( $30 \pm 3\%$ ). Furthermore, no turbidity removal was observed in these tests.



**Figure 1 :** Effect of current intensity in different pH values on EF treatment (t = 30 min.) COD (a) and color (b).

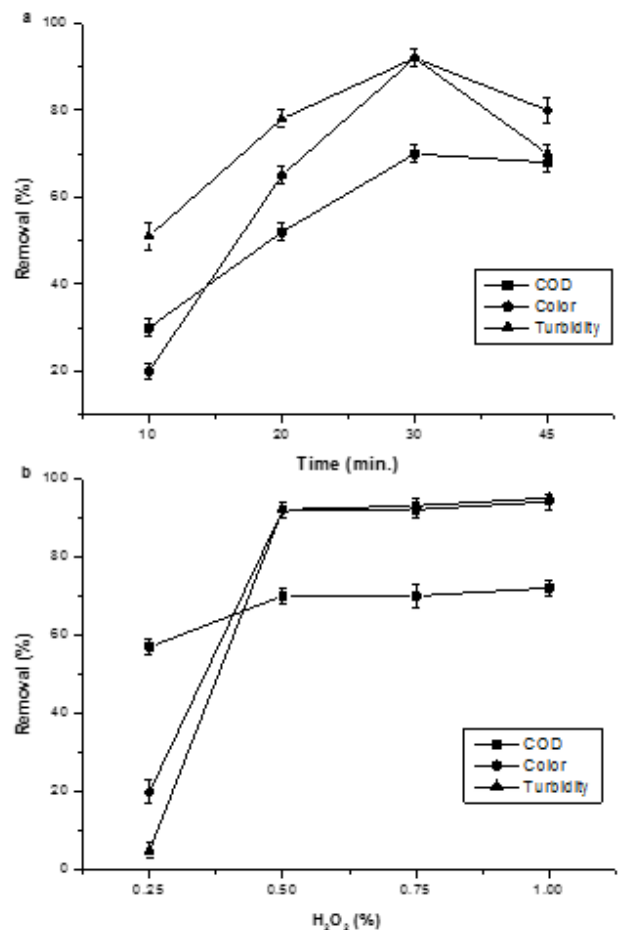
According to Claro et al [31], in 30 min. of reaction-time, the solution becomes reddish due to the presence of  $Fe^{3+}$  ions, generated by the oxidation of  $Fe^{2+}$  ions. Thus with precipitation of  $Fe^{3+}$  ions into  $Fe(OH)_3$ , color values of treated effluent becomes higher than color values of non-treated effluent. This fact happens because the iron electrodes does not cease the flakes production while electrolysis occurs and the gas bubbles generated promote the rise of the flakes (electrofloculation). With the increasing amount of suspended matter, the turbidity of treated effluent also increases.

Besides pH, the optimum current intensity must be determined for each effluent since it directly affects the electrolytic treatment performance and causes an unnecessary increase of energy consumption, as well as electrode consumption [22]. Thus, based on the results presented in Fig. 1b, the optimum current intensity was defined as 2 A, considering that using the current

intensity of 5 A, the high ferrous ions concentration induced the darkening of the solution. This observation was confirmed by a higher electrode consumption generating ferric ions which absorb at  $\lambda=400$  nm.

### Effect of reaction time and $H_2O_2$ concentrations in E-Fenton treatment

The performance of E-Fenton treatment at different reaction times lasting 10, 20, 30 and 45 minutes each, was evaluated in order to compare the removal efficiency of COD, color and turbidity. It was observed that maximum removal of COD, color and turbidity removals occurred in 30 minutes of reaction were, respectively:  $70\pm 2\%$ ,  $92\pm 2\%$  and  $92\pm 2\%$  (Fig. 2).



**Figure 2 :** Effect of reaction time (a) and  $H_2O_2$  concentration (b) on E-Fenton treatments in the removal efficiencies of COD, color and turbidity.

Concerning to 45 minutes time (Fig. 2a), it was observed that there was a decrease in removal efficiencies. Lin and Chang [32] noted that, in reactions that last long time, after total consumption of hydrogen peroxide,

there was a decrease in color and turbidity removal efficiencies due to the increment of ferric ions electrochemically generated.

The influence of different  $H_2O_2$  concentrations (0.25, 0.50, 0.75 and 1.00% v/v), in E-Fenton treatment was assessed using a reaction time of 30 minutes.

The results presented on Fig.2b indicates that peroxide dosage of 0.5% (v/v) guarantees higher COD, color and turbidity removal efficiency presenting a lower reagent consumption when compared to the dosages of 0.75% (v/v) and 1.00% (v/v), that showed equivalent removal rates for these parameters.

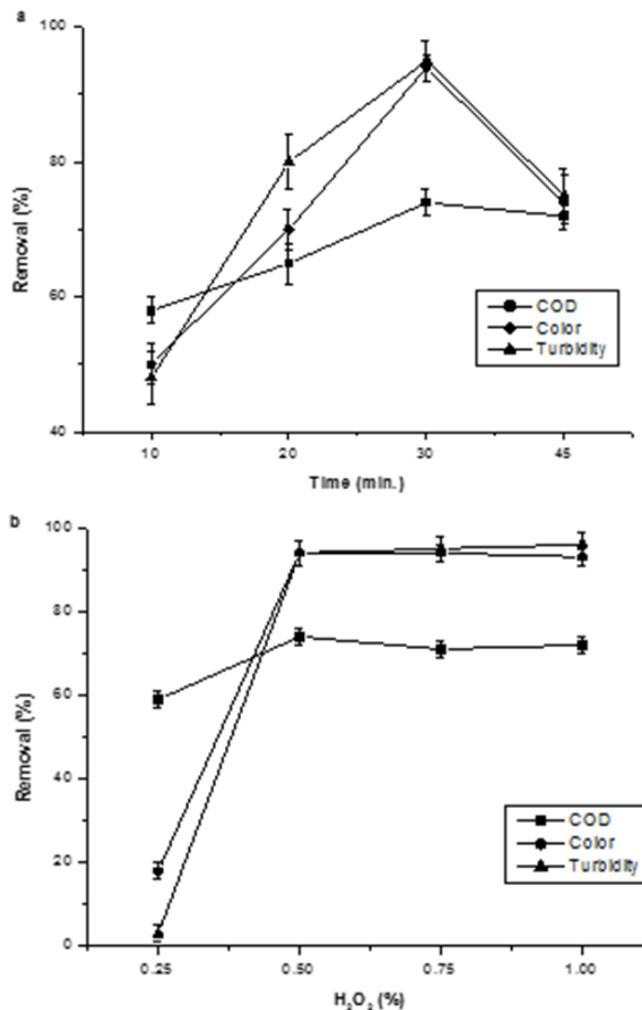
The low removal efficiency in E-Fenton treatment found in 0.25% (v/v)  $H_2O_2$  dosage, may be associated to the fact that  $Fe^{2+}$  ions in excess, compete with organic compounds for the hydroxyl radicals ( $OH^\bullet$ ), which decreases the efficiency in removing effluent color and turbidity.<sup>3</sup>

#### Effect of reaction time and $H_2O_2$ concentrations on EF+E-Fenton process

The results for removal efficiencies of COD, color and turbidity in EF+E-Fenton process using different reaction times (10, 20, 30 and 45 minutes), are presented in figure 3. The best results obtained were  $74\pm 2\%$ ,  $94\pm 2\%$  and  $94\pm 3\%$ , (Fig. 3a) and maximum COD, color and turbidity removals occurred at 30 minutes of reaction time.

Similarly to E-Fenton, in EF+E-Fenton process, after 45 minutes of reaction time (Fig. 3a), a decrease in removal efficiencies was observed. This way, in order to find the optimum hydrogen peroxide concentration for achieving the best removal efficiencies, different  $H_2O_2$  concentrations (0.25, 0.50, 0.75 and 1.00% v/v) were tested in 30 minutes of reaction time.

The results shown in Fig. 3b shows that peroxide dosage of 0.5% (v/v) was the best for obtaining the higher COD, color and turbidity removal efficiency with a lower reagent consumption when compared to the dosages of 0.75% (v/v) and 1.00% (v/v).

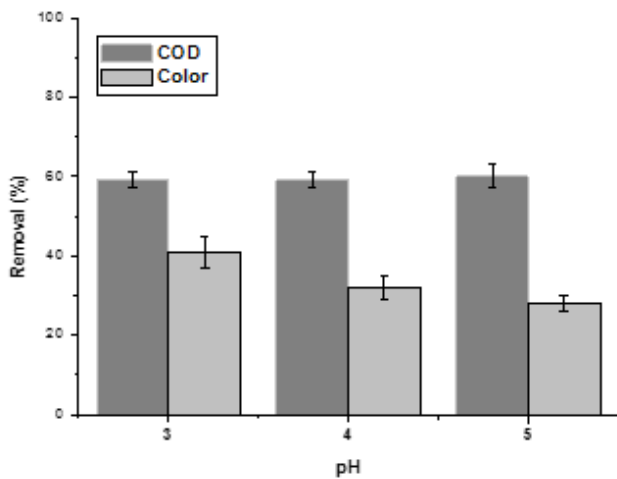


**Figure 3 :** Effect of time reaction (a) and  $H_2O_2$  concentrations (b) on EF + E-Fenton treatments in the removal efficiencies.

Similarly to E-Fenton treatment, the lower color and turbidity removal efficiencies for EF+E-Fenton treatment was observed when using  $H_2O_2$  dosage of 0,25% (v/v), probably caused by  $Fe^{2+}$  ions in excess in competition with organic compounds for hydroxyl radicals ( $OH^\bullet$ ).

#### Effect of initial pH on Fenton process

Initial pH values investigated were 3.0, 4.0 and 5.0,  $H_2O_2$  dosage used was 0.5%, and  $H_2O_2/Fe^{2+}$  ratio was 1.8 (w/w) [27]. The best removal efficiencies for COD ( $59\pm 2\%$ ) and color ( $41\pm 4\%$ ) by oxidation were obtained at pH=3.0 (Fig. 4), which is in accordance with classic Fenton process [20].



**Figure 4 :** Effects of initial pH on the COD and color removal by the Fenton process (t = 30 min., 0.5% H<sub>2</sub>O<sub>2</sub> dosage and H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> 1.8 (w/w)).

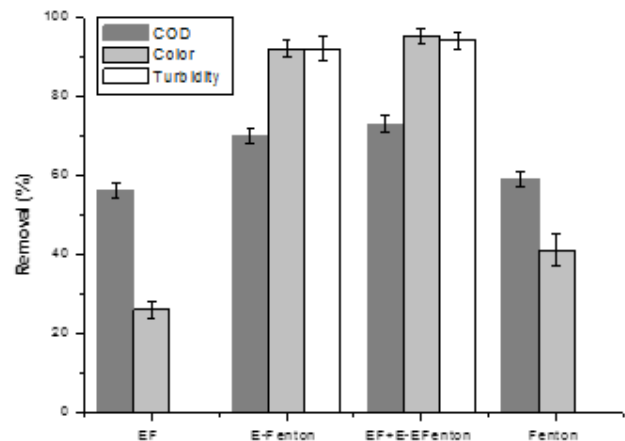
Singh and Tang [27] reported that in Fenton process, COD removal by oxidation scarcely occurs at pH above 7. In pH values higher than 5.0, the removal efficiencies decreases as result of the accelerating decomposition of hydrogen peroxide into water and oxygen, as well as the deactivation of ferrous ions into ferric hydroxo complexes, and the decrease oxidation potential of hydroxyl radicals. In addition, under alkaline conditions, the decomposition of H<sub>2</sub>O<sub>2</sub> to generate •OH radical is inhibited due to absence of H<sup>+</sup> ion.

### Energy and Electrode Mass Consumption

In optimized conditions for EF treatment (initial pH 6; i = 2 A; U = 0.75 V; reaction time: 30 min.) and E-Fenton (initial pH 3; i = 2 A; U = 0.75 V; reaction time: 30 min.; [H<sub>2</sub>O<sub>2</sub>] 0.5%), the leachate treatment has consumed 0.75 kW h m<sup>-3</sup> of energy. Regarding the EF+E-Fenton process, in optimized condition (EF phase: initial pH 6; i = 2 A; U = 0.75 V; reaction time: 30 min.; E-Fenton phase: initial pH 3; i = 2 A; U = 0.75 V; reaction time: 30 min.; [H<sub>2</sub>O<sub>2</sub>] 0.5%) the energy consumption was 1.5 kW h m<sup>-3</sup>. The theoretical electrode mass consumption in EF and E-Fenton in optimized conditions was 1.04 kg m<sup>-3</sup>. In EF+E-Fenton process the consumption was 2.09 kg m<sup>-3</sup>. Experimentally, the truthful electrode mass consumption, gravimetrically determined, was 0.8 kg m<sup>-3</sup> to EF and E-Fenton and 1.6 kg m<sup>-3</sup> to EF+E-Fenton.

The results achieved in this study shows that in EF+E-Fenton combined treatment, the energy and the electrode mass consumption were higher than in EF and E-Fenton

treatments. However, comparing the results of EF, E-Fenton, EF+E-Fenton and Fenton process in their best operating conditions (Fig. 5).



**Figure 5 :** Comparison of removal efficiencies of COD, color and turbidity among the different treatments of the landfill leachate: EF, E-Fenton, EF+E-Fenton and Fenton process.

It was observed that EF+E-Fenton and E-Fenton have no significant differences on COD, color and turbidity removal efficiencies, indicating that E-Fenton process is the most appropriate method for treatment of old landfill leachate

### IV.CONCLUSION

This study results reveals that the parameters: initial pH, reaction time, hydrogen peroxide concentration and current intensity, are important in assessing the performance of EF, E-Fenton and combined EF+E-Fenton processes.

Fenton process results for COD removals were similar to that obtained for EF (electrofloculation). A small difference in color removal efficiency was noticed, pointing that Fenton had a better performance for color removal.

According to the results of this work, the recommended operation conditions for treating the old municipal landfill leachate were: initial pH 6, i = 2 A and 30 minutes treatment time in EF process. Using these conditions, 56±2% of COD and 26±2% color were removed.

E-Fenton process was more effective than EF process removing, approximately,  $70\pm 2\%$  of COD,  $92\pm 2\%$  of color and  $92\pm 3\%$  of turbidity in its optimum operation conditions (initial pH=3,  $i = 2$  A,  $H_2O_2 = 0.5\%$  concentration and 30 min. reaction time).

Combined EF+E-Fenton and E-Fenton had no significant differences on COD, color and turbidity removal efficiency in its optimum operating conditions. However in combined treatment (EF+E-Fenton), electric energy and electrode mass consumption was higher, indicating that from an environmental stand point, E-Fenton process, is a promising alternative for treatment of old municipal landfill leachates, reducing its potential to harm water resources, soil and biota. Moreover E-Fenton requires lower costs in energy and electrode maintenance.

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