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Evaluation of the electrode wear and the residual concentration of iron in a system of electrocoagulation

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ABSTRACT

In this study, we evaluated the wear and tear experienced by electrodes and the residual concentration of iron in a system of electrocoagulation using effluent from a dairy industry. The experimental module was composed of iron electrodes, a 10 A source of direct current, and a 1,000 mL beaker. The tests were conducted with and without polarity inversion through the following variables: pH 4.5, time 60 min, and an electric current intensity of 1.5 A. In polarity inversion tests, the mass consumed in the electrode showed an average value of 0.2412 g/L of iron and residual iron presented a mean value of 2.10 mg/L. The treatment efficiency was 99% for turbidity, 95% for COD, and 90% for color. In the tests without polarity inversion, the wear of the electrodes was lower due to the effect of passivation, but residual iron presented high levels of concentration reaching 57.5 mg/L. Treatment efficiency was lower, mainly due to the reduction of color. The treated effluent presented reddish-brown color in some tests.

Keywords: COD; Electrocoagulation; Electrode; Iron; Wear

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1. Introduction

The dairy industry is of utmost importance for the Brazilian food sector, but its contribution to pollution is also very significant. Water is the most employed natural resource in the sector and one normally links its use to ensuring sanitary and hygiene conditions. Depending on the type of installation of the industry, the cleaning system, and its management, each liter of processed milk can generate up to 10 L of effluent [1,2].

The residual waters of milk processing usually contain proteins, salt, greasy substances, lactose, as well as residues of chemical products used in cleaning processes. These effluents are associated with a high organic load and a high concentration of fermentable substrates with persistent unpleasant odors [3–5]. In industries in which there is a large production of cheese without utilization of serum, the value of the chemical

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oxygen demand (COD) can reach 50.000 mg/L. In these types of effluents, the lipid content may be greater than 1.500 mg/L [6,7].

According to Nogueira and Jardim [8], studies of new alternatives that are able to minimize the volume of water used in industrial processes or provide its reuse, as well as reduce its toxicity, are the main weapons used to combat environmental pollution caused by the dumping of effluents. An alternative that has been widely used is the electrocoagulation (EC). Currently, the tendency is to use electrochemical techniques such as EC, electroflotation, electrooxidation, and electrodisinfection which are considered promising methods for water/wastewater treatment [9,10].

A system of treatment by EC is composed of at least two electrodes (electrical conductors) immersed in an electrolyte that carries the ions. In this process, three successive operation stages occur: first, the formation of a coagulant agent through the electrolytic oxidation of the sacrifice electrode causes the neutralization of surface charges, the destabilization of colloidal particles, and the breakage of emulsions. Second, there is the agglutination of particles destabilized by hydroxides (e.g. iron and aluminum) which are natural coagulants that favor the formation and growth of flocs. Thirdly, the generation of oxygen (O_2) microbubbles occurs. These microbubbles form with the oxidation of water in the anode and of hydrogen (H_2) in the cathode, which rise to the surface where they collide with, and are adsorbed by the flocs, dragging the particles and impurities suspended in the middle, and thus promoting the clarification of the effluent. The most commonly used electrodes are made of iron and aluminum, due to their effectiveness, low cost, and due to the fact that they are materials that are readily available [11–13].

The main advantages of an EC system are related to the size of the electrolytic cell, requiring small areas for its implantation, with reduced electrolysis time, lower sludge production, ease of operation, the lack of need for dosage equipment, and high removal for various harmful substances [14]. Electrochemical technologies have reached such a state that, not only are they comparable to other techniques in terms of cost, but they are effective and easier to apply. For some situations, electrochemical technologies can be the vital step to treat effluents containing the most varied types of contaminants [12].

Another advantage of the EC is that it can also be used with other processes, such as for: EC/Electrooxidation [15], EC/Electro-Fenton [16], EC/Ultraviolet oxidation [17], EC/Ozone [18], EC/Ultrafiltration [19], EC/Nanofiltration [20], EC/Crossflow microfiltration [21], EC/Fluidized bed anaerobic reactor [9], EC/Activated carbon sorption filtration [22], and EC/Membrane filtration [23].

In relation to the use of the iron anode in EC, the oxidation of the metal generates ferrous (Fe^{2+}) and ferric (Fe^{3+}) ions. These ions cause some inconveniences in the EC process due to reddish-brown color that can occur at the end of the treatment. This phenomenon is directly related to the intensity of electric current applied and the duration of the electrolysis [24,25]. The generation of this waste is one of the biggest concerns in an EC system and effluents discharged into receiving bodies must respect the limits predicted in current legislation.

Another factor that affects the EC process in a negative way is the "passivation" phenomenon, which occurs in electrodes responsible for oxidation. This phenomenon involves the EC performance, because it interferes directly in the formation of coagulant ions. Electrode passivation is the formation of an inhibitory layer, usually an oxide, on the electrode surface preventing the dissolution of the metal and the transfer of electrons, thus limiting the generation of the coagulant agent. With time, the thickness of this layer increases, and reduces the effectiveness of the process as a whole, and a loss of efficiency due to the increase in resistivity. Passivation is seen as a serious limitation in the application of the EC system. Some simple initiatives, such as the use of new materials, of different types of electrodes, and operational strategies (such as the periodic polarity inversion), certainly lead to significant reductions in the impact of this phenomenon [26–28].

Facing this, the objective of this study was to evaluate the wear and tear experienced by electrode in the treatment of effluent from a dairy industry by the EC technique, and the residual concentration of iron generated by the process. It was also evaluated the influence of these factors on the quality of the treated effluent.

2. Material and methods

2.1. Experimental module

The electrolytic cell used in this experiment was composed of a 1,000 mL beaker and two iron electrodes. This electrode has six rectangular iron plates that are 3 mm thick, grouped in a parallel setting in a monopolar system. The plates were connected at the top with the aid of a screw, forming three anodes and three cathodes. Two rubber tubes were used as electrical insulation. The chosen source was an AC/DC converter type brand Instrutherm model FA-1030 that has maximum capacity of 10 A. The experiment mounted in bench scale can be seen in Fig. 1.

The experiments were carried out at State University of Maringá, and the effluent used in the study is from a local dairy plant. The effluent collections were made in periods of full production, in order to ensure very representative samples of industry dumping.

2.2. Experimental approach

Corrections of pH were made with the calcium hydroxide $Ca(OH)_2$, raising the crude pH of the effluent from 3.0 to 4.5. This value was chosen because it is close to the characteristic pH of the isoelectric point of casein (between 4.6 and 4.7), the main protein in milk. In accordance with [29], in this condition, the proteins of this effluent agglomerate, so there is formation of flocs, since the number of positive charges is equal to the number of negative charges, and this neutralization makes possible the coagulation process possible.

Preliminary tests with variations in time and electric current intensity were performed in order to determine the remaining variables. The tests were conducted with time (30, 60, 90 and 120 min) and electric current (0.5, 1.0, 1.5 and 2.0 A). The combination used was of the highest efficiency in reducing of turbidity, color and COD (pH 4.5, time 60 min, and electric current 1.5 A).

For evaluation of the wear, two electrodes were used, one with weight of 1,201.345 g and other with 1,188.530 g. They were disassembled and each plate was weighed individually on a Mars AY 220 precision scale. There were 24 trials, 12 for each electrode, replacing the effluent at the end of each batch. A sequence of tests was performed without the polarity inversion procedure, i.e. anodes and cathodes were the same throughout the experiment. The other sequence was performed with polarity inversion, which was made at the end of each batch, thus the anodes exercised the function of cathodes in the following experiment and vice versa. The objective of this test was to show how the phenomenon affects the treatment quality compromising the efficiency of the process.

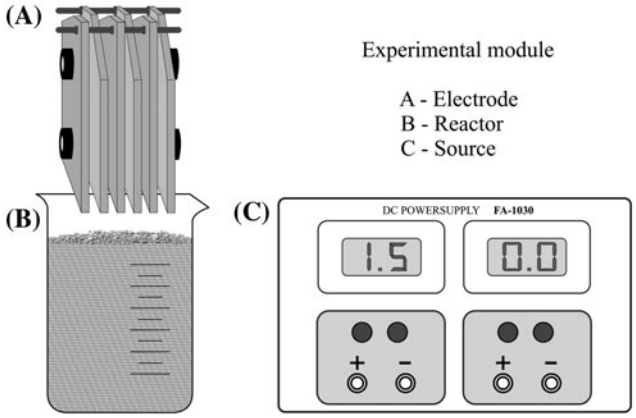

Fig. 1. System of EC mounted in bench scale.

At the end of the 24 tests, the electrodes were disassembled and cleaned by extracting all impregnated residue. After washing, the electrodes went into an oven to dry, and then they were weighed in order to determine iron mass consumed during the process.

In this experiment, it was not necessary to use NaCl as the supporting electrolyte, since the raw effluent presented conductivity levels favorable to application of the treatment.

2.3. Analysis

Analyses were performed on the treated effluent with the aim of assessing the efficiency of the process, turbidity, color, COD, and dissolved iron. After the end of each batch, the effluent remained at rest for approximately 2 h until the whole sedimentation process finalized, then a sample was collected for analysis. Analyses of the raw wastewater were also performed, in order to highlight its characteristics. All procedures followed the recommendations of the Standard Methods for the Examination of Water and Wastewater [30].

Turbidity was measured with the aid of the Max Labor (Apha 2130) brand turbidity meter. Analyses of color were carried out with colorimeter Hack 2100Q (Apha 2120). COD was performed using the colorimetric method of closed reflux, using a digester block Marconi MA 4004 as equipment. The readings were done on a Perkin Elmer Lambda 25 (Apha 5220 D) spectrophotometer. The BOD_5 was performed by the method of titration, with samples incubated for 5 d at 20°C using an Aqua Lytic AL606 (Apha 5210 B) incubator. The residual iron concentration was determined by the atomic absorption method using a Shimadzu AA-7000 (Apha 3111 A) spectrometer. Conductivity was performed with the aid of a Tecnopon CA 150 (Apha 2510) conductivity meter. Lastly, temperature was measures by the conventional method using an Icotherm (Apha 2550 B) mercury thermometer.

3. Results and discussion

3.1. Features of raw effluent

The physicochemical properties of raw effluent can be observed in Table 1.

Table 1
Results of physicochemical analysis of raw effluent

Parameter	Unit	Values
pH	pH unit	3.0 ± 0.16
Conductivity	μS/cm	1,050 ± 20.51
BOD_5	mg/L	6,500 ± 50.48
COD	mg/L	11,800 ± 680
Settleable solids	mg/L	650 ± 30
Turbidity	ntu	1,350 ± 199
Color	mg Pt/L	15,000 ± 479
Temperature	℃	24 ± 0.5
Dissolved iron	mg/L	0.50 ± 0.06

3.2. Trials of electrocoagulation (EC)

Table 2 presents the results of the 12 tests conducted to evaluate the electrode wear during the treatment of the effluent by EC.

Note that electrode 03 was the one that had the lowest consumed mass, with 0.1232 g/L of iron, and electrode 04 was the one that showed the greatest mass loss 0.3261 g/L. The other ones presented more balanced values ranging from 0.2194 to 0.272 g/L. The electrodes that presented higher wear were 01 and 06, which may have occurred because these are positioned at the extremities of the set, positioned close to the source connectors, receiving a greater load than the other ones. Electrodes 03 and 04, located at the center of the set, showed lower wear values. The other 12 tests were also carried out under the same conditions, but without the inversion of polarity, and the results can be observed in Table 3.

In the tests carried out without the inversion of polarity, one can observe very low wear values for electrodes 02, 04 and 06, since they acted just as cathodes. The highest wear was on the other electrodes, which were used as anodes.

If comparing the wear values of the anodes, one can observe that the wear was much smaller in the experiments without the polarity inversion. This occurred due to the phenomenon of passivation, which inhibited iron oxidation and therefore provided a smaller wear. The average consumed mass in anodes was 0.1705 g/L and in the cathodes it was 0.0327 g/L. Other authors also reported on the wear of electrode and reported very similar values to those found in this study.

Kobya et al. [31] used iron and aluminum electrodes in their study to treat effluents from textile industry. The variation of pH was from 3 to 11, the intensity of electrical current from 3 to 20 A/m2 and electrolysis time from 5 to 30 min. In the aforementioned study, the iron electrode showed mass wear between 0.30 and 0.65 g/L. Bazrafshan et al. [32] studied EC in the removal of chromium VI in a synthetic solution of chromium using iron and aluminum electrodes. The variables used in the experiment were: electrolysis time ranging from 20 to 60 min, current intensity from 20 to 40 V, and pH between 3.7 and 10. The value for the mass consumption of the electrode observed in this study was from 7.70 to 9.01 g/L. Zodi et al. and Kuokkanen et al. [33,34] developed a study on EC in the treatment of effluent from a paper industry with the objective of removing organic nonbiodegradable pollutants and arsenic. The optimization was achieved with the density of electric current being 150 A/m2, an electrolysis time of 90 min, and a pH of 7.70. The efficiency in the removal of parameters was 100% for turbidity and 68% for COD. In that study, the consumed iron mass varied from 0.207 to 0.310 g/L. Another study conducted by Bazrafshan et al. [35], about the removal of fluoride in aqueous environment using iron and aluminum electrodes, used the following variables: pH from 3 to 10 and intensity of electric current from 10 to 40 volts. The values found for the wear of the electrode were from 0.36 to 2.31 g/L of iron.

Dermentzis et al. [36] studied the removal of hexavalent chromium by EC using iron electrodes with a distance of 1.5 cm between the plates. The initial pH was between 4 and 8, the agitation applied was 500 rpm, the current intensity was 10, 20, and 40 A/m2, and electrolysis time ranged from 0 to 130 min. Under the given conditions of operation, mass loss of the electrode was 4.34 g/L. Chaturvedi [37] studied the removal of mercury from a synthetic effluent by EC using iron electrodes. The variables of the study were: electrolysis time from 5 to 60 min, intensity of electric current varying from 6 to 12 V, agitation speed from 200 to 600 rpm, and pH from 2.5 to 7.0. The electrode wear observed by the author was 1.33 g/L.

A more recent study conducted by Can [38] about COD removal in fruit juice production wastewater by EC. The author used a current density of 200 A/m2 and times of 5–25 min, with pH ranging from 6 to 7. The electrode consumption in this study using iron electrodes was 0.044–0.217 g. Alizadeh et al. [39] aimed to remove the reactive dye, orange 16, in an aqueous solution using an iron electrode with distance of 1 cm between the plates, density of electric current of 4, 10, and 20 A/m2, electrolysis time from 5 to 30 min, NaCl concentration from 1 to 3 mg/L, and pH from 2 to 11. The maximum removal was achieved with a current density of 20 A/m2, pH 5.50, reaction time of 30 min, and NaCl concentration of 1.5 g/L. The removal of COD was 66% and the efficiency in the removal of the dye was greater than 98%. The wear of the electrode observed in that study was from 0.005 to 0.035 g/L.

Table 2
Results of EC tests with polarity inversion

Electrode	Initial mass (g)	Final mass (g)	Wear (g/L)	Wear (%)
01	201.7467	198.4814	0.2721	0.1318
02	199.7210	196.7623	0.2465	0.1234
03	198.3535	196.8751	0.1232	0.0621
04	202.8364	200.2030	0.2194	0.1081
05	201.9145	198.7945	0.2600	0.1287
06	196.7729	192.8586	0.3261	0.1657

Table 3
Results of EC tests without polarity inversion. Plates 01, 03 and 05 (anode) and 02, 04 and 06 (cathode)

Electrode	Initial mass (g)	Final mass (g)	Wear (g/L)	Wear (%)
01	199.9647	197.6777	0.1905	0.0960
03	201.1808	199.0802	0.1750	0.0889
05	194.1636	192.4095	0.1461	0.0742
02	201.0377	200.7041	0.0278	0.0144
04	195.4463	194.6888	0.0631	0.0317
06	196.7369	196.6475	0.0074	0.0037

3.3. Efficiency of parameter reduction

In relation to treatment efficiency, Fig. 2 represents the results of the parameter analysis, in which one can observe the behavior of the EC for the experiments with and without the polarity inversion. Regarding efficiency in the reduction of turbidity, in trials conducted with polarity inversion, reduction values were higher than 99%. In the tests without the inversion of polarity, results were higher than the 97% only in the first five tests. In the following tests, there was a small loss of efficiency with results showing variations from 92 to 95%.

In COD reduction, efficiency ranged from 92 to 95% by applying the inversion of polarity. In the experiments without polarity inversion, one can observe results similar to that only in the first four tests, and then the efficiency began to fall due to the action of passivation. Lower efficiencies started becoming evident from the ninth test, reaching a minimum value of 60% of reduction.

For parameter color, the study found that the efficiency for tests with polarity inversion obtained values between 83 and 90%, and in the tests without polarity inversion only the first four showed more relevant results, ranging from 79 to 89%. From the fifth test, one can note a reduction of efficiency to approximately 42%, and from the ninth test, experiments began to show negative values.

Turbidity was the parameter that had less influence from passivation, being able to produce a certain quantity of flocs to the point of obtaining efficiency values greater than 90% in all tests. However, parameters of color and COD were the most harmed, which is clear upon observing Fig. 2, especially in the last tests. After some tests, the electrode started to suffer the effects of passivation, affecting the EC performance by cathode oxidation inhibition, which caused the fall in coagulant ion production, also affecting the intensity of electric current supplied to the electrodes. This inhibiting layer tends to increase with the passage of time, which justifies the gradual decline in treatment efficiency. In accordance with Hu et al. [40], the adoption of procedures for removal of this passive film is quite difficult, because it is a very thin layer, and any chemical or electrochemical measure could change its structure and properties, so that it may interfere in the effluent treatment process. Moreover, polarity inversion has been a simple technique and with a significant effect as a preventive measure.

3.4. Electrode wear, pH, and iron

In Fig. 3, one can observe the general appearance of two EC experiments, one performed with polarity inversion (A) and another without the inversion (B).

In the test with polarity inversion, the greater percentage of iron was retained in the floated/decanted material, presenting a limpid aspect effluent. One could observe a greenish coloration in both waste and effluent, in the experiment without polarity inversion.

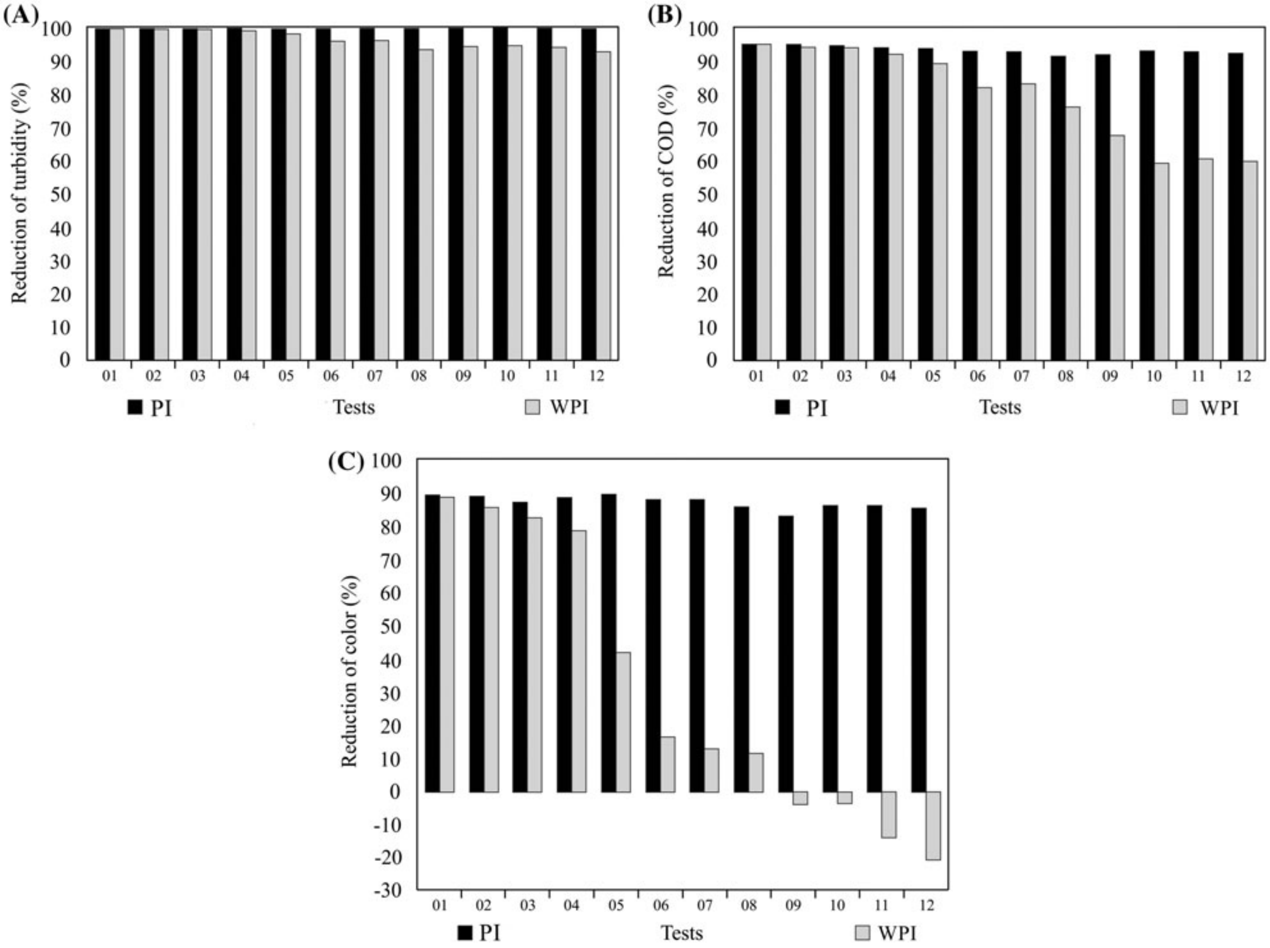

Fig. 2. Efficiency in reduction of turbidity, color and COD. Polarity inversion (PI); without polarity inversion (WPI).

In relation to the wear of the electrodes, it is possible to verify that the electrode that has undergone the passivation process presented less wear of the plates, which is an advantage for the treatment, because in this case, the durability of the electrode is greater. However, upon observing its performance in relation to the efficiency in the parameters reduction, one can note that it has not achieved satisfactory levels during the 12 trials (Fig. 4).

On the pH of the samples, the study observed that in all the experiments, the final value was higher than the original. In tests with polarity inversion, pH values presented a variation from 7.0 to 7.4 with a mean of 7.25. Without the inversion of polarity, the variation was from 6.6 to 7.1 with a mean value of 6.88. The pH increase in relation to initial concentration (4.5) was approximately 65%. According to Chen et al. and Daneshvar et al. [41,42] pH values may vary according to the initial pH of the sample and also according to the type of material used as electrode. This increase occurs when the value of the raw effluent is acidic, i.e. less than 7.0. In the case of basic pH, mainly with value greater than 9.0, the tendency is that there is a decrease during the treatment. Chen and Cañizares [12,43] relate the increase in pH in acidic conditions to cathodic reduction, where the hydrogen gas produced is fully released from the electrochemical cell, remaining in the solution hydroxides ions particles resulting from water hydrolysis, causing an increase in pH. This statement explains the similarity of pH values of the samples with and without inversion of polarity, since the cathode had equal behavior in both experiments.

The values for the concentration of residual iron of the treated effluent, in tests with polarity inversion, presented variations from 1.87 to 2.36 mg/L, and in tests without polarity inversion, results were very superior, presenting within the first tests a concentration of 4.0 mg/L. As a result, there was an increase in the concentration and until test 06 the iron content was high. However, the iron content was still below the

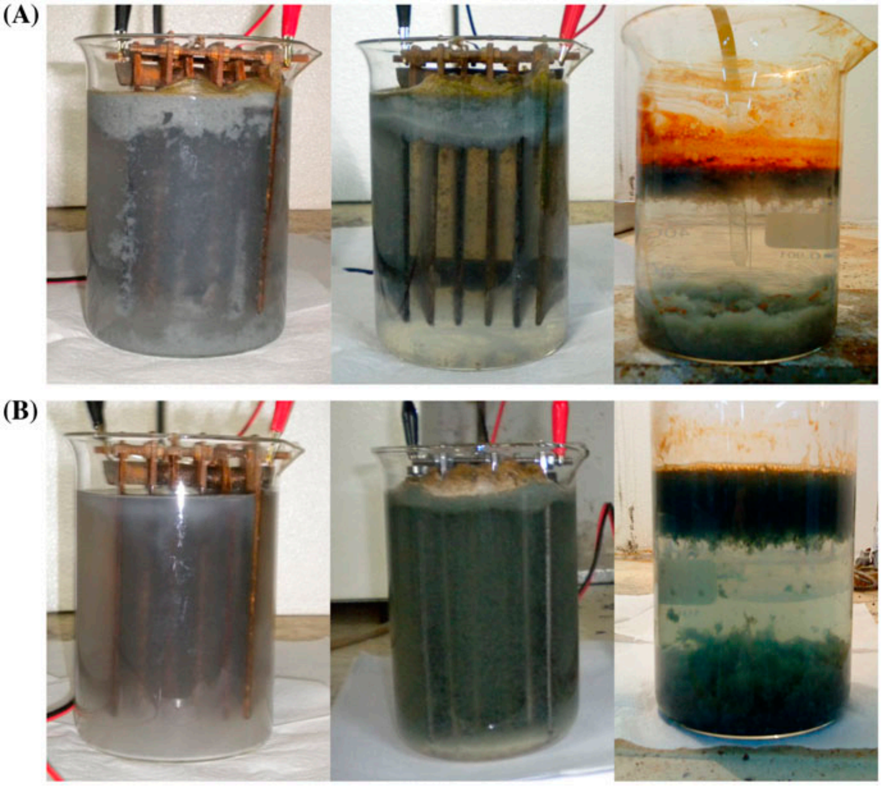

Fig. 3. General appearance of the effluent in tests conducted with PI (A) and WPI (B).

limit laid down in standard of the National Council for the Environment of Brazil (CONAMA) [44], which is 15 mg/L. From test 07 onwards, there was a gradual increase, with values reaching 57.5 mg/L. This high iron concentration left a characteristic green and yellow color in the effluent (Fig. 4). According to Feng et al. [45], this color is from the ions Fe^{2+} (green) and Fe^{3+} (yellow) and from the formation of $Fe_{(S)}$ precipitate, resulting from the reaction between the iron ions and species of sulfur, generated in electrolytic treatment.

3.5. Iron effect in the quality of effluent

In Fig. 5, one can observe effluent samples (B–E) that are related to recent trials conducted without polarity inversion. Sample (B) was collected immediately after the EC, presenting a greenish coloration, due to the presence of a large quantity of iron particles in the effluent. However, after the oxidation of the particles, Fe^{2+} went to Fe^{3+} changing the color of the samples to shades of yellow.

Other authors also reported the existence of colorations caused by the effect of the oxidation of iron in their studies. Sengil and Özacar [46] used for their experiment, an effluent of dairy product, collected in an industry in Turkey with daily production of 50 m^3. The effluent was COD around 18.300 mg/L, suspended solids 10.200 mg/L, pH between 6.0 and 7.5, and electrical conductivity of 1.200 μS/cm. The electrodes used were a bipolar type and connected in parallel, with a distance of 2.5 cm. The experiment was developed with an agitation of 100 rpm. The variables chosen were: sodium chloride concentration from 0.77 to 4.61 mg/L, pH from 3.5 to 10, and electric current density from 3 to 18 A/m2. The optimum conditions of removal was obtained using an electrical current of 6 A/m2, pH between 6 and 7 and electrolytic electrolysis time of only 1 min. The efficiency result was 98% of COD reduction, but the final effluent showed an orange color.

Gonzales and Torem [47] evaluated the effect of iron and aluminum electrodes in the treatment of oily synthetic effluents. The effluent had a pH between 8 and 9 and conductivity of 2.200 μS/cm. The variables used were: oil concentration of 3, 6, and 9 g/L and a number of electrodes ranging among 2, 4, and 6, 1 cm apart. The results showed that EC achieved a great reduction of COD (94.8%) and turbidity (98.5%) in the following experimental conditions: current density of 9.4 A/m2 and the operating time 30 min. In the

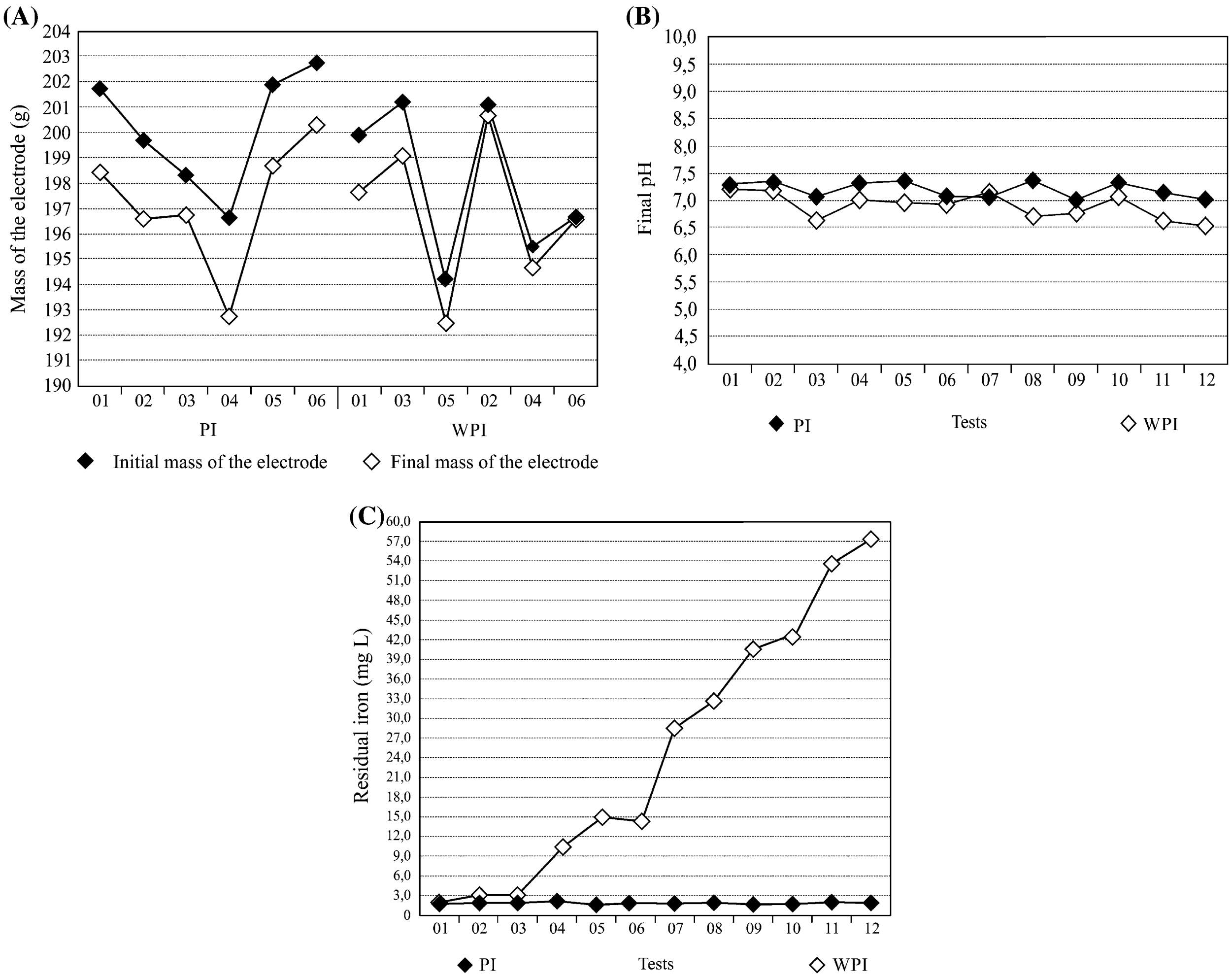

Fig. 4. Results of the electrode wear (A); final pH of the samples (B) and concentration of residual iron (C). PI; WPI.

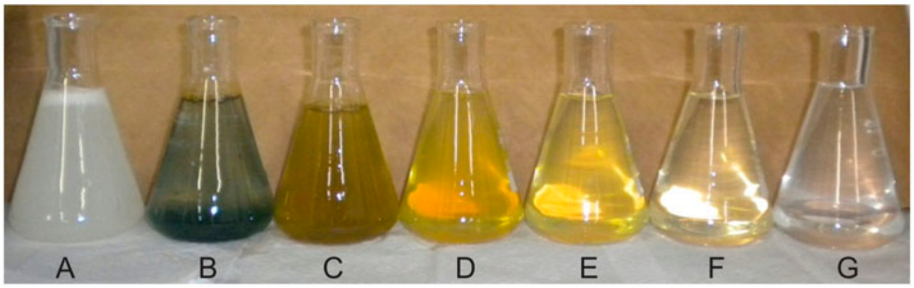

Fig. 5. Visual aspect of some samples effluent. Raw effluent (A); effluent after EC without polarity inversion (B–E); effluent after EC with polarity inversion (F and G).

experiments with iron electrode, the authors reported a yellow residual coloration in the treated effluent.

Another study of EC using dairy effluent and iron electrodes was performed by Valente et al. [48], using the following operational variables: electrolysis time from 5 to 25 min, electric current density from 37 to 61.6 A/m2, pH from 5 to 9, and distance between electrodes from 0.6 to 1.4 cm. The best operational condition was determined by variables time 15 min, pH 7, and electric current of 50 A/m2, resulting in

95% of reduction of the turbidity and 58% of COD. The final pH of the effluent was in the range of 9.5 and the appearance of orange color was observed in the final effluent.

One can notice that the presence of green and reddish-brown colorations have been frequent in studies of EC when iron electrodes are used, which may cause some inconveniences in the quality of the treated effluent. Hence, some factors must be taken into account in the choice of operating parameters. The levels of the electrical current must be adjusted well, as it will determine the amount of metal to be oxidized. The pH must be adjusted according to the characteristics of the effluent. Electrolysis time must vary according to the applied electrical load, whereas higher loads lead to a faster treatment. The distance between the plates is also an important factor, because the more distant from each other, the greater the load required for treatment will be. Another important factor that must be considered in order to improve EC efficiency is related to the conductivity of the effluent, because effluents with low conductivity do not respond well to the treatment, in which case it is necessary to adopt some corrective measures. The addition of sodium chloride (NaCl) is the most common procedure adopted by researchers.

4. Conclusion

The treatment proved to be effective in relation to the levels of residual iron, reaching values far below those required by standard. Trials with polarity inversion showed a maximum value of 2.36 mg/L. In the experiment performed without polarity inversion, acceptable levels were observed in trials 01–06, and only in the remaining ones were the values greater than 15 mg/L showing noncompliance to the standard.

Regarding the assessment of electrode wear, the results of this study showed that levels of iron mass loss were not very high, with an average value of 0.2412 g/L. This value is considered satisfactory, since results near that were found in other studies of EC. In the experiments without polarity inversion, electrode wear was lower, due to the passivation effect, but efficiency results in parameters reduction indicated the unfeasibility of the process.

The passivation effect showed no influence on the pH of the samples, with both experiments showing similar values. The green and yellow colorations resulting from iron oxidation were observed in some trials without polarity inversion, causing negative results regarding the efficiency of color reduction. Regarding treatment efficiency with polarity inversion, the results obtained were 99% for turbidity reduction, 95% for COD reduction, and 90% for color reduction.

In general, the results confirm the potential of EC technique in which through simple, compact and low operating cost equipment, it is possible to achieve significant results in the treatment of effluents. Another important factor is the proper choice of operating parameters, as well as the adjustment of their levels in accordance with the characteristics of the effluent and the type of electrode material, ensuring efficiency along the process.

Acknowledgments

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