Efficiency and operating cost of electrocoagulation system applied to the treatment of dairy industry wastewater

Henrique Cesar Lopes Geraldino¹*, Julliana Izabelle Simionato², Thábata Karoliny Formicoli de Souza Freitas³, Juliana Carla Garcia³, Orlando de Carvalho Júnior² and Caryna Januario Correr⁴

¹Universidade Estadual do Sudoeste da Bahia, Rod. Br-415, Km 3, 45700-000, Itapetinga, Bahia, Brazil. ²Universidade Tecnologica Federal do Paraná, Londrina, Paraná, Brazil. ³Universidade Estadual de Maringá, Maringá, Paraná, Brazil. ⁴Universidade Estadual de Londrina, Londrina, Paraná, Brazil. *Author for correspondence. E-mail: henriqueclg@hotmail.com

ABSTRACT. The objective of this study was to apply the technique of electrocoagulation (EC) in the treatment of dairy industry wastewater, using a system of bench. It was used a source of 10 amps and an electrode of iron containing six plates of 14 x 6 cm. Were made variation of pH (4.5 to 6), intensity of electric current (1 to 2A) and time of electrolysis (30 to 60 min.). The efficiency of the treatment was confirmed by analysis of turbidity, color and COD. The reduction of turbidity was greater than 98%, reaching a record 99.98%. The largest reduction in color was 94.90% and 11 of the 27 tests the efficiency was greater than 90%. The greatest reduction of COD was 96.36%. The treatment proved to be more efficient at pH 4.5, time 60 min. and current 1.5A. The estimated cost of operation was US$ 1.04 m³ which demonstrates the viability of treatment of dairy industry wastewater.

Keywords: conductivity, COD, electrolysis, iron electrode, oxidation.

Efficiência e custo operacional de um sistema de eletrofloculação aplicado ao tratamento de efluente da indústria de laticínio

RESUMO. O objetivo deste estudo foi aplicar a técnica da eletrocoagulação (EC) no tratamento de efluentes de indústria de laticínio, usando um sistema de bancada. Foi utilizada uma fonte de 10 amperes e um eletrodo de ferro contendo seis placas de 14 x 6 cm. Foram feitas variações de pH (4,5 a 6), intensidade de corrente elétrica (1 a 2A) e tempo de eletrolise (30 a 60 min.). A eficiência do tratamento foi confirmada pelas análises de turbidez, cor e DQO. A redução de turbidez foi maior que 98%, atingindo um total de 99,98%. A maior redução de cor foi 94,90% e em 11 dos 27 ensaios a eficiência foi superior a 90%. A maior redução de DQO foi 96,36%. O tratamento demostrou ser mais eficaz com pH 4,5, tempo de 60 min. e corrente 1,5A. O custo estimado de operação foi R$ 2,39 m³, o que demonstra a viabilidade do tratamento para efluente da indústria de laticínio.

Palavras-chave: condutividade, DQO, eletrolise, eletrodo de ferro, oxidação.

Introduction

The contamination of natural waters is currently a major problem that is leading environmental control agencies to establish stricter limits for the disposal of effluents into the environment. In addition, chemical treatments can induce a secondary pollution linked to chemical additives added, which may contaminate the treated water (DEMIREL et al., 2005).

Dairy industries represent an activity of great importance for the food sector Brazilian, though its contribution in terms of pollution is very significant. The water is the natural resource more employed in industry and their use is linked to the guarantee of hygiene and sanitary conditions. The sanitation of dairy products includes silos washes operations, pipes, tanks, pasteurizers and equipment that require large volumes of water (BRIÃO; TAVARES, 2012).

The effluent generated in dairy industries can vary greatly depending on the type of process used, the products produced, the quality of water required. Due to the high content of organic matter, such effluents can reach 150,000 mg L⁻¹ of COD, 2,900 mg L⁻¹ of fat and 5,100 mg L⁻³ of total solids (CARVALHO et al., 2013; VOURCH et al., 2008).

Several research has been done to improve the technologies for treating industrial effluents. The most common technique is the typical physical-chemical treatment combined with biological processes (WOODARD; CURRAN INC., 2006). Studies have been performed on systems employing electrochemical reactors for the decontamination of effluents containing high...
solid loadings. The electrocoagulation (EC) technique, which is also known as electroflocculation (EF) and electrocoagulation-flotation, is a promising alternative due to its effluent reduction efficiency (CRESPIILHO; REZENDE, 2004).

EC consists of passing the effluent through electrodes, which are arranged in parallel, in a horizontal or vertical flow fed by a continuous electrical current supplied by a self-regulated source. During treatment, the organic molecules are dissolved and inorganic salts undergo electrolytic dissociation, which perturbs the ionic balance of the medium. At the anode (positive), iron ions are generated, which form the coagulation agent, and at the cathode (negative), hydrogen microbubbles are formed, which carry the particles to the surface, constituting a flotation process (CRESPIILHO; REZENDE, 2004). This technique is of considerable interest due to its operational simplicity, in addition to its potential for treating a large variety of effluents (BORBA et al., 2010).

Given the above, the main objective of this study was to determine the optimal operation conditions and operating cost of treatment a bench scale EC system used effluent from a dairy industry.

Material and methods

The effluent used in this study was collected from the Vidativa Ltda. dairy industry, which is located in the municipality of Terra Boa, Paraná State in Brazil at coordinates 23°45’36.20” S and 52°26’43.53” W. The dairy treatment system consists of a primary treatment in a fat-separating box followed by a secondary treatment using stabilization ponds.

The EC technique was applied using a system of bench (Figure 1) mounted in the chemistry lab at the State University of Maringá (UEM). The reactor used was a glass beaker (borosilicate 3.3) with a maximum capacity of 1000 mL. The electrodes were placed in this beaker, which established the electrolytic cell. The electrode was composed of six iron plates (cells) measuring 14x6 cm with a thickness of 3 mm; these plates were grouped in parallel separated by a distance of 1.5 cm and secured by two rubber tubes for isolation. The source used in the experiment was an Instrutherm DC Power Supply FA-1030 with a maximum intensity of 10 amperes. After each assay, polarity inversion of the electrodes was performed to avoid excessive wear and the formation of passivation films. The passivation film attacks the anode responsible for the coagulant action, resulting in a reduction in the system efficiency. The tests were performed at room temperature, and before starting the assays, the source remained on for at least 30 min. to obtain better equipment performance.

Experimental planning and data analysis

To minimize the initial number of tests and to obtain the optimum operating conditions, a Full Experimental Planning 3^3 (FEP) with 3 variables (pH, electric current intensity and electrolytic reaction time) and 3 levels (-1, 0, +1) was applied (BARROS NETTO et al., 2010). Following the methodology applied in this experimental planning, a total of 27 trials were performed in duplicate and randomly, with duration of four months. To represent the experimental data, quadratic models were proposed based on the responses of the analyzed parameters and of the process variables (Equation 1).

$$\hat{y} = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_i x_i^2 + \sum_{i<j} \beta_{ij} x_i x_j + \varepsilon$$  (1)

Models were proposed for the turbidity, color and COD parameters based on the coefficients of the significant factors, and these models are described in Equations 2, 3 and 4, respectively.

$$\hat{y}_{turbidz} = a - x_1 + x_2 - x_3 - x_1^2 + x_2^2 + x_3^2 + x_1 x_2 x_3$$  (2)

$$\hat{y}_{cor} = a - x_1 + x_2 - x_3 - x_1^2 + x_2^2 + x_3^2 - x_1 x_2 x_3$$  (3)
\[ \hat{y} = DQO(\%) = a - x_1 + x_2 - x_3 + x_2^2 - x_3^2 - x_4 x_2 - x_1 x_3 - x_1 x_2 x_3 \]  

These models were validated through an analysis of variance (ANOVA), and they were statistically reliable at 95% (p < 0.05) according to the F ratio (\( F_{\text{calc.}} > F_{\text{tab.}} \)). The software Statistica 10 was used to develop the statistical analyses (STATSOFT INC, 2011). The choice of the initial pH (4.5) was made based on the isoelectric point of casein (4.6), the main protein in milk. Second Nespolo et al. (2015) in this condition of pH proteins of this effluent congregates, there is formation of flakes, since the number of positive charges is equal to the number of negative charges, and this inhibition allows the coagulation process. The other values (5 and 6) were adopted, a time that, preliminary tests with pH lower than 4.5 did not have good results. The other variables were determined from preliminary tests, and the values chosen were the electric current intensity (1, 1.5 and 2 amperes) and electrolytic reaction time (30, 45 and 60 min.). The efficiency of the process was determined based on the percentage reduction in the turbidity, color and COD parameters. The pH and final conductivity of the treated effluent were also monitored.

The effluent samples were collected and shipped in 30 liter polyethylene containers. The analyses were performed in the in natura effluent for physicochemical characterization and in treated effluent (triplicate) for the purpose of evaluating the efficiency of the technique. In each experiment, the pH was adjusted using a solution of alkalizing calcium hydroxide in suspension Ca(OH)\(_2\). After each assay, the treated effluent was allowed to rest on a bench for 24 hours to decant, subsequently samples were collected for analysis. To collect the samples, a 60 mL syringe attached to a tube was used. All analyzes were performed according to the recommendations of the Standard Methods for the Examination of Water and Wastewater, the pH (4500 H\(^+\)), turbidity (2130 B), color (2120), COD (5220 D), BOD\(_5\) (5210 B), the OD (2500-O C), sedimentary solids (2540 F), total solids, fixed and volatile (2540 G), residual iron (3111 A), the conductivity (2510 B) and temperature (2550 B) (APHA, AWWA, WPCF, 2005).

The calculation of electrode wear, electrode wear time, electric energy consumption and operating cost, are represented in (Table 1). The calculation of electrode wear followed Crespilho and Rezende (2004), the calculation of electrode wear time followed Wiendl (1998), the electric energy consumption was performed according to Kobya et al. (2006) and system operating cost was based on (DONINI et al., 1994; KOBYA et al., 2006).

**Table 1.** Equations used to determine the operating cost of system.

| Calculation | Equation | Legend |
|-------------|----------|--------|
| (5) Electrode wear | \[ m_{el} = \frac{i \cdot t \cdot M}{F \cdot \alpha \cdot n} \] | \( i=\) current (A) \( t=\) time (s) \( M=\) molar mass (g) \( F=\) Faraday constant \( \alpha=\) number of electrons |
| (6) Electrode wear time | \[ t = \frac{M_{\text{el}}}{\alpha \cdot i} \] | \( M_{\text{el}}=\) initial mass \( \alpha=\) electrochemical equivalent (g/Ah) \( i=\) current (A) |
| (7) Electric energy consumption | \[ C_{en} = \frac{U \cdot L \cdot t}{V} \] | \( U=\) electric tension (V) \( L=\) current (A) \( t=\) time (h) \( V=\) volume (m\(^3\)) |

\* Operating cost \[ C_{op} = a \cdot C_{en} + b \cdot C_{el} \]

\* Energy cost based in (Copel-tariff for industrial class. Iron cost based in (Instituto Aço Brasil).

**Results and discussion**

The characteristics of the in natura effluent are presented in (Table 2). The analysis revealed an effluent with high organic load content, showing high level of COD, total solids and turbidity.

**Table 2.** Physico-chemical parameters of the in natura dairy industry wastewater.

| Parameter | Unit   | Values  |
|-----------|--------|---------|
| pH        | pH unit| 3.30±0.11 |
| Conductivity | \( \mu \text{S cm}^{-1} \) | 1.06±0.43 |
| BOD\(_5\) | mg L\(^{-1}\) | 4.67±150.44 |
| COD       | mg L\(^{-1}\) | 11.81±1.228 |
| OD        | mg L\(^{-1}\) | 4.08±0.46 |
| Settliable solids | mg L\(^{-1}\) | 715±21 |
| Total fixed solids | mg L\(^{-1}\) | 458±29 |
| Total volatile solids | mg L\(^{-1}\) | 5.71±1.54 |
| Turbidity | ntu    | 1.347±0.194 |
| Color     | mg Pt L\(^{-1}\) | 14.90±2.638 |
| Temperature | °C     | 24.3±0.6 |
| Dissolved iron | mg L\(^{-1}\) | 0.64±0.09 |

The Table 3 presents the results of the 27 assays conducted during the experimental period. It is observed that the proposed system exhibited better efficiencies for reducing turbidity, showing values above 90% in all assays.
The Figure 2 shows the efficiencies for reducing the turbidity, color and COD parameters. With respect to the turbidity, the conducted assays showed variations in efficiency between 90.20 and 99.98%. It is also noted that there was a decrease in efficiency, especially in the last assays after the 19th assay, with values lower than 95%. This result may be related to the pH of 6, which was used in the last assays.

According to Bruice (2006) in the coagulation process occurs a phenomenon called isoelectric point. That corresponds to the value of pH of the medium to which the molecule is electrically neutral, i.e., when the number of positive charges is equal to the number of negative charges, then occurring, coagulation. In relation to dairy effluent, the pH characteristic of isoelectric point is approximately 4.6. In this condition the protein congregates resulting in formation of floc. This statement may justify the decrease in the efficiency of reducing the parameters as the pH increases.

The color reduction efficiencies ranged from 19.26 to 94.90%. Of the 27 conducted assays, 13 showed reduction efficiencies lower than 50%. We note that higher color reduction efficiencies were observed in the first assays, in which the pH was 4.5. As with the turbidity reduction, the color reduction efficiency was also reduced in the last assays. The COD reduction efficiencies were very similar to the color reduction efficiencies. In assays 01 to 06, it is possible to observe efficiencies greater than 85%, and from this point, these values begin to significantly decrease.

In EC studies using dairy effluent, it was noted that the correct choice of pH is essential to guarantee the efficiency of the treatment and that there is also a determinant factor considering the type of material used as electrodes because iron and aluminum, which are the most used materials, have their greatest efficiencies under different pH conditions. In some samples that used pH 6 and 2A, it was possible to observe shades of green and yellow in the final effluent. These samples showed a low color reduction efficiency, which demonstrates that the process has low efficacy when performed outside of the ideal range.
The iron electrode exhibits a very strong residual green or yellow coloring during and after treatment due to the generation of Fe$^{2+}$ (green color) and Fe$^{3+}$ (yellow color) in the electrolytic treatment (FENG et al., 2007). This means that the iron generation is closely related to the charge, which, in turn, can be controlled by the obtained current. Thus, the measured current in an electrocoagulation process is, in principle, proportional to the amount of ionized metal generated in solution. From this information, by knowing the amount of metal ions required to promote the coagulation of impurities, it is possible to establish the current to be applied for a range of predetermined time and the area of the electrodes that will participate in electrolysis for better treatment efficiency (CRESPILOH; REZENDE, 2004).

Values of pH and conductivity of the effluent

As shown in Figure 3, the final pH values of the samples exhibited many variations and were not lower than the initial pH value in any experiment. The maximum pH was 8.55 in assay 19, where the initial pH was 6, with a time of 60 min. and a current of 2A. The lowest pH recorded was 6.03 in assay 09, which had an initial pH value of 4.5. The experiment in which the best values in the reduction of parameters was obtained presented a final pH of 7.35 (assay 2). Throughout the entire experimental period, the pH of the final effluent met the values established by Brazilian legislation for the discharged of effluent which establishes values between 5 and 9 for discharge standards into receiving bodies (BRASIL, 2011).

In studies of EC some changes in value of pH depend mainly on the electrode material and the initial pH value (KOPARAL et al., 2008). According to Chen (2004) and Daneshvar et al. (2004) there is increase in pH when its initial value is low, i.e., less than 7. In the case of pH greater than 9, there is tendency for the pH to decline during treatment.

With respect to the conductivity of the effluent, in the 27 experiments, the final conductivity presented values that were both higher and lower than the initial one. The highest conductivity observed in the treated effluent was 1.212 $\mu$S cm$^{-1}$ in assay 16, which used a 2A current, and the lowest conductivity observed was 0.955 $\mu$S cm$^{-1}$ in assay 03, which used a 1A current. It is also observed that the experiments that showed higher final conductivity values were those that employed a current between 1.5 and 2A and a time from 30 to 45 min. Additionally, the largest decreases in conductivity occurred mainly in experiments with a longer duration and lower applied current.

The analysis of dissolved iron showed a minimum value of 1.32 mg L$^{-1}$ and a maximum value of 5.07 mg L$^{-1}$. On average, the amount of dissolved iron in the assays was 2.44 mg L$^{-1}$. These results meet the values predicted in the norm which sets a limit of 15 mg L$^{-1}$ for dissolved iron (BRASIL, 2011).

Regarding temperature, the initial temperature values of the effluent were approximately 24°C, and the final temperature values were 27°C on average. This value meets that predicted in current norm, which places a limit of up to 40°C for the discharge of effluents into water bodies.

The correlation models between the parameters and experimental variables of the process were validated by analysis of variance, which was performed to confirm the validity of the quadratic models and to show that the regression is statistically significant (Table 4).
Table 4. Results of ANOVA for models of turbidity, color and COD: SV-Source of variation; QS-Quadratic sum; DF-Degrees of freedom; MS-Mean square.

| Parameter | SV | QS  | DF  | MS  | Fcalc. | Ftab. | p value |
|-----------|----|-----|-----|-----|--------|-------|---------|
| Turbidity | Regression | 179.58 | 3   | 89.79 | 36.44  | 3.03   | 0.0000  |
|           | Residual    | 20.22  | 23  | 10.11 | 4.10   | -      | 0.0321  |
|           | Lack of fit | 0.32   | 2   | 0.16  | 0.06   | -      | 0.9371  |
|           | Pure error  | 49.28  | 20  | 2.46  | -      | -      | -       |
|           | Total       | 249.40 | 26  | -     | -      | -      | -       |
| Color     | Regression  | 11845.17 | 3   | 5922.58 | 52.35  | 3.03   | 0.0000  |
|           | Residual    | 3463.64 | 23  | 1732.92 | 15.32  | -      | 0.0000  |
|           | Lack of fit | 371.51  | 2   | 185.76 | 1.64   | -      | 0.2186  |
|           | Pure error  | 2262.48 | 20  | 113.12 | -      | -      | -       |
|           | Total       | 17944.80 | 26  | -     | -      | -      | -       |
| COD       | Regression  | 9765.51 | 3   | 4882.76 | 49.02  | 3.03   | 0.0000  |
|           | Residual    | 2351.25 | 23  | 1175.62 | 11.80  | -      | 0.0000  |
|           | Lack of fit | 46.94   | 2   | 23.47  | 0.23   | -      | 0.7922  |
|           | Pure error  | 1992.03 | 20  | 99.60  | -      | -      | -       |
|           | Total       | 14155.73 | 26  | -     | -      | -      | -       |

For the turbidity parameter, the calculated determination coefficient was $R^2=0.89022$; this value indicated that approximately 89% of the experimental variance can be explained by the proposed second-order model. For the color parameter, the coefficient showed a value of $R^2=0.90725$, demonstrating an experimental variation of approximately 91% in the reduction of this parameter. For COD, the determination coefficient was $R^2=0.91895$, which was capable of explaining approximately 92% of the variance. In this study, all quadratic models tested were adequate for explaining the behavior of the variables regarding the analyzed parameters at satisfactory levels of approximately 90%.

It was observed that the pH was the parameter that showed the greatest significant effect on the efficiencies of color and COD reduction. Using the F test, it was verified that all models are valid ($F_{cal} > F_{tab}$). Furthermore, according to the p value of the test, the validity of the model was confirmed because the ($p < 0.05$).

To evaluate the optimum test conditions, response surface graphs (Figures 4, 5 and 6) were constructed considering the following parameters: turbidity, color and COD. For each graph, the following variables were considered: time and pH (A), current and pH (B) and current and time (C). For the turbidity parameter (Figure 4), the best results for time and pH were obtained at 60 min. and 4.5, respectively; for current and pH, the best results were obtained at 2 and 4.5, respectively; and for current and time, the best results were obtained at 1A and 60 min. The turbidity, it can be stated that the pH parameter was the one that most influenced the results, and from graphs (A and B), we can observe a very wide range of maximum reduction of pH (4.5 to 5).

For the color parameter (Figure 5), the best result was obtained for a time and pH of 60 min. and 4.5, respectively; for current and pH, it was 1A and 4.5, respectively; and for current and time, it was 1A and 60 min., respectively. Evaluating graphs (A and B), we note that the range of maximum reduction is small, with grouping more concentrated in the pH range of 4.5, and in graph (C), the range of maximum reduction has become broader with values concentrated in the ranges from 45 to 60 min. and current 1 to 2A.

Regarding COD (Figure 6), the best result for time and pH was 4.5 and 60 min., respectively; for current and pH, it was 1.5 to 4.5, respectively; and for current and time, it was 60 min. and 1A, respectively. As shown in graphs (A and B), narrower ranges of maximum reduction in relation to pH (4.5 to 5) are observed, and as shown in graph (C), there was a greater extent in relation to the efficiency, with values between 45 and 60 min. and all ranges of amperage.
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Figure 5. Response surface of experimental data for color reduction. Optimization equation: \[ \hat{Y} = 61.84 - 25.58x_1 + 11.77x_2 - 5.97x_1^2 + 11.20x_1x_2 + 5.53x_2^2 - 1.97x_3 + 1.54x_1x_3 - 1.91x_2x_3 - 4.53x_1^2x_3 - 1.54x_2^2x_3 - 5.10x_1x_3^2 - 1.94x_2x_3^2 + 11.77x_2^2 - 0.99x_1^3 - 0.54x_2^3 - 7.99x_3^3 - 5.10x_1^2x_3^2 - 1.91x_2^2x_3^2 - 4.53x_1x_2x_3^2. \]

Figure 6. Response surface of experimental data for COD reduction. Optimization equation: \[ \hat{Y} = 53.90 - 23.29x_1 + 9.81x_2 + 0.99x_3 - 6.95x_1^2 + 7.22x_2^2 + 4.53x_1x_2 + 11.77x_1x_3 + 5.53x_2x_3 + 1.54x_1x_3 - 1.54x_2x_3 - 7.99x_3^2 - 0.99x_1^3 - 0.54x_2^3 - 5.10x_3^3 - 1.94x_2^2x_3^2 - 0.54x_1^2x_3^2 - 1.94x_2^2x_3^2 + 7.99x_3^3. \]

Considering the results found for the three parameters, it is possible to conclude that the optimized conditions for EC with dairy effluent was (pH of 4.5, time of 60 min. and current of 1.5 A).

Calculation of electrode wear

As described in Equation 5, we can calculate the maximum amount of electrode material that was consumed during the EC process, in which the molar mass of iron is 56 g mol\(^{-1}\) and the oxidation number of the element is 2.

\[ t_e = \frac{i.t.M}{F.x} = \frac{1.5 \times 3.600 \times 56}{96.500 \times 2} - \frac{302.400}{193.000} = 1.566 \text{gFe L} \]  

Electrode wear time

The calculation of electrode wear time was performed according to Equation 6. To avoid interference in the results of this experiment, polarity inversions were performed for each assay to avoid the formation of a passivation film on the electrode. The electrolytic equivalent of Fe was 1.040 g A h, and the initial mass of the electrode plates was 1.201345 g.

\[ t = \frac{M_o}{i.t} = \frac{1.201,345}{1.040 \times 1.5} = \frac{1.201,345}{1.560} = 770.09 \text{h} \]  

According to Mollah et al. (2004) polarity reversal increases the electrode life by up to two times; thus, the electrode that is responsible for the oxidation shall perform the function of reduction and vice versa. Considering this factor, the time required for electrode wear would be approximately 2.310.27 h.

Calculation of energy consumption

According to Equation 7, one can calculate the electric energy consumption during the EC process. Was considered the value of industrial electrical energy (A4 conventional/september 2013) US$ 0.1028 kWh.

\[ C_{en} = \frac{U.i.t}{V} = \frac{3.0 \times 1.5 \times 1}{0.001} = 4.5 \text{ W h m}^{-3} \]

\[ 4.5 \times 4.500 \text{ W h m}^{-3} = 4.5 \text{ kW h m}^{-3} \]

Calculation of the operating cost of the system

One can calculate the operating cost of the batch reactor in the EC system using Equation 8. The value of a kg of iron is US$ 0.99, but the mass estimated cost of a plate (200 g) is US$ 0.20.

\[ C_{op} = a.C_{en} + b.C_{el} \]

\[ C_{op} = 0.1028 \times 4.5 + 0.20 \times 1.566 \]

\[ C_{op} = \text{US$ 1.04 m}^{3} \]

Whereas the daily generation of effluent at the dairy industry is approximately 40 m\(^3\), the monthly value estimated is US$ 1.248.00.

Conclusion

The application of the EC technique to dairy industry wastewater was demonstrated to be efficient in reducing the studied parameters,
obtaining a 99.98% efficiency for reducing turbidity, a 94.90% efficiency for reducing color and a 96.36% efficiency for reducing COD in its best configuration. Through statistical analysis, it was possible to determine the optimized conditions for the EC of dairy wastewater (pH 4.5, time 60 min. and electric current 1.5A). The result was considered satisfactory because the EC exhibited efficient treatment with a low operating cost. The operating cost for the EC system was estimated US$ 1.04 m$^3$ day and US$ 1.248.00 month using its best configuration variables, reaching an efficiency of almost 100% for turbidity reduction and approximately 95% for color and COD reduction.

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