v. 3, n. 24, 2023

Journal of
Engineering
Research

OPTIMIZATION AND VALIDATION OF AN ELECTROLYTIC SYSTEM IN THE TREATMENT OF COFFEE WASTEWATER WITHOUT RECIRCULATION VIA RESPONSE SURFACE METHODOLOGY (RSM)

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DOI 10.22533/at.ed.3173242314072

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Abstract: Residual water from coffee without recirculation can cause serious impacts if released into the environment without treatment. The electrolytic technique appears as a promising alternative to minimize this damage. However, high effluent times in the reactor can make the treatment unfeasible, consuming a lot of electrical energy and increasing the wear of the electrodes. For this reason, the objective of this study was to optimize the Hydraulic Detention Time (Time) based on the removal of turbidity, Total Solids (TS) and Chemical Oxygen Demand (COD), monitoring the behavior of pH and electrical conductivity. In the experiments, a glass electrolytic reactor with a volume of 1000 cm^3 was used. Batch experiments were performed following a completely randomized design in a subscript scheme, varying the factors: plate distance (PD: 10, 20 and 30 mm) current density (CD: 25, 50, 75 and 100 A m^{-2}) and hydraulic detention time of the reactor effluent (Time: 0, 60, 121, 183, 247, 312, 378, 446, 516 and 586 s). After using optimization techniques, it was found that the averages of the optimal operating conditions of the monitored variables are in Time intervals of 376 seconds, 70 A m^{-2} of CD and 26 mm of PD, reflecting the greater removal of pollutants. The values predicted by the models generated overestimated turbidity by 59% and underestimated TS and COD by 14% and 2%, respectively. The validation in the optimized conditions, showed removal of 68.35%, 25.55% and 3.68%, for turbidity, total solids and COD, respectively. The overall electrical consumption was 1.75 kW.h m^{-3}, while the operational treatment cost was US$ 0.13 m^{-3}. Electricity represents the highest cost in electrolytic treatment, about 78.8% of the total operating cost under the conditions of the studied system, which can be reduced with the capture of photovoltaic electricity.

Keywords – Coffee Wastewater;

Electroflotation; Systems optimization; Response Surface.

INTRODUCTION

Coffee has great economic importance in the Brazilian trade balance. The value stipulated by the market depends on the quality of the grains [1], which in turn are directly related to the way they were processed [2].

In order to maintain the natural quality, the fruits are generally peeled and demucilated in a wet way, generating the Coffee Residual Water (CWW) [3]. This effluent has a high concentration of potassium, sugars, proteins, starch, pectin, phenolic compounds, total nitrogen, total phosphorus [1] and, mainly, organic matter. Such characteristics of CWW, justify the need for treatment before it is released into water bodies, or recirculated in the processing unit (peeler) and later use as fertilizer in the field [4].

In this context, the electrolytic system appears as a promising alternative in the treatment of effluents. This technique, also known as electrocoagulation, electroflocculation and electroflotation [5], has some advantages when compared to conventional effluent treatment methods, such as high efficiency in the removal of solids, compact system, relatively low costs and possibility of automation [6]. However, long Hydraulic Detention Times (Times) provide an increase in pH [7], increases in turbidity caused by the generation of aluminum hydroxides [8] and higher energy consumption, requiring optimization of the factors involved in the process.

Most studies focus only on removal efficiency, few on removal rate and minimal on the specific cost of each variable. For Kuokkanen et al. [9], many authors do not present economic values related to current densities in optimal operating conditions. Thus, it is also essential to evaluate energy consumption according to the removal rate of each pollutant. [10].

Thus, the objective of this study was to optimize an electrolytic system operating in the treatment of Coffee Wastewater without recirculation (CWW), improving the performance of the system in order to obtain the lowest possible final concentration of pollutants.

MATERIAL AND METHODS

The experiment was conducted at the Research Laboratory of the Federal Institute of Science and Technology of Espírito Santo (Ifes), *Campus* Ibatiba, Espírito Santo, Brazil. The coffee residual water without recirculation came from the debarking and demucilating of Arabica coffee beans (*Coffea arabica*) from a processing unit located in the city of Ibatiba at 900 meters altitude, with UTM coordinates: 7,753,073 S and 240,188 E (Zone 24 K).

The CWW samples were packaged following methodologies adopted by ABNT [11] and MATOS et al. [12, 13].

For the CWW treatment, an electrolytic reactor in commercial glass was built, following the suggestions of distance from plates, areas of the electrodes, electrical conductivity of the effluent and applied current established by Mollah et al. [14], presenting internal dimensions: width of 8.15 cm, length of 13.8 cm and depth of 8.9 cm and volume of 1000 cm^3 (Figure 1).

The electrodes were made of 99.50% pure aluminum plate (ABNT/ASTM 1050) measuring 0.8 mm thick, 7.9 cm wide by 13.0 cm long, with a surface area of 0.02054 m^2, being completely immersed in the effluent. The configuration used was monopolar in parallel, where the electrodes are polarized interchangeably through external connections and the anodes being the electrodes that will be oxidized (consumed) [14].

The first stage of the experiment was performed in batch, in a completely randomized design, in a subdivided plot scheme, with two repetitions, in which plots the factor Plate Distances in three levels (PD = 10, 20 and 30 mm), in the subplots the Current Density factor in four levels (CD = 25, 50, 75 and 100 A m^2) and in the sub-plots, the Hydraulic Detention Time factor in ten levels (Time: 0, 60, 121, 183, 247, 312, 378, 446, 516 and 586 s). The non-equidistant intervals of the Time factor aimed at compensating for the treatment time, once, after removing the aliquots from the interior of the reactor, there was a decrease in the volume of effluent in it.

The characterization of CWW's in batch experiments, as well as in continuous flow, was performed by the variables COD, phosphorus, nitrogen and total solids determined following the Standard Methods [15]. The total sugar content was determined according to Albarici et al., [16], potassium by NBR 13810 [17], total phenols by the method of Folin Ciocalteau and the others by specific and properly calibrated equipment: conductivity meter, Hanna photometer (colorimeter) and pH meter.

The response variables of the experiments were turbidity, total solids, COD, pH and electrical conductivity, based on their environmental representativeness [18] and intrinsic to the effluent. The effluent temperature was maintained at 20°C ±2, thus avoiding the influence of this variable.

Analysis of variance was performed for each response variable by the F test, at a significance level of 5%, continuing with regression analysis after finding interaction between the factors (PD, CD and Time). The verification of the adequacy of the terms of the mathematical models adjusted was performed by means of the significance (*p*) of each of the regression coefficients via sequential. For residues, normal distribution, homogeneity and homoscedasticity were evaluated, as also performed by Combatt et al. [19].

To find the levels of each factor, PD, CD and Time, which optimize the response predicted by the system, through the raw data of the experiments, the midpoints of the factors were found where it presented the lowest concentration of the studied responses variables. These points were used to fix one of the factors by varying the others in the generation of the equation that describes the response surface (RSM).

The equations generated in the RSM were partially derived as a function of each factor (PD, CD or Time), finding the minimum points of the function. The verification of the minimum point occurred by checking the signs of the quadratic terms of the generated equation and by elaborating the Hessian matrix (2x2), containing the second derivatives of each equation obtained previously.

The validation of the adjusted model was performed using three statistical indices: graphical comparison of the actual results with the theoretical results predicted by the best model, the accuracy factor and the bias factor, represented by Equations 1 and 2, respectively [19].

$$\text{Accuracy} = 10^{\sum \frac{\left|\log \frac{p}{o}\right|}{N}} \tag{1}$$

$$\text{Bias} = 10^{\sum \frac{\log \frac{p}{o}}{N}} \tag{2}$$

where: N is the number of observations, "p" is the predicted value and "o" is the observed value.

The optimal intervals exposed by the model were adjusted to simulate values of the response variable. Before activating the simulation's continuous flow, the reactor was connected in batch with the CWW sample in the optimized conditions (PD and CD) and the effluent flow was activated a few seconds before reaching the optimal time (hydraulic detention time - HDT) optimized for the CWW.

The validation experiments (in continuous flow) were performed in triplicate with at least 3 (three) times the HDT established in the optimization, using the data from the central third of the samples collected at the reactor outlet in the posterior steps and discarding the initial and final third. The strategies used aim to avoid external influences, maintain hydrodynamics and stabilize the reactor flow [20].

For the purpose of graphical comparison, the results of the collections performed during the simulation of the reactor in continuous flow were compared to the values predicted by the models generated in the RSM for each of the modeled variables (Turbidity, TS and COD) of the CWW.

The CWW samples used in the validation had physicochemical characteristics similar to those used in the optimization experiments and initial temperature of 20°C ±1. The preservation of the attributes of the samples occurred under refrigeration at 4°C [11-13].

The average removal rate (ARR) was calculated based on the average removal or change of variables in the collection intervals, according to Equation (3):

$$ARR = \bar{X}_{t_0}^{t_f} \frac{(C_{t_0} \square C_{t_1})}{(t_1 \square t_0)} \qquad (3)$$

where:

ARR – Average Removal Rate in NTU min^{-1} or mg L^{-1} $min^{-}1$ or dS m^{-1} min^{-1} or pH units min-1;

$\bar{x}$ is the average of the removal or alteration rates of the variables analyzed in each (t_0 to t_f), in minutes, in the collection intervals;

C is the concentration of the variable studied over time, in NTU, mg L^{-1} or dS m^{-1};

t is the time interval in which the variable was collected, with t_0 being the initial collection of the experiment, t_1 the first collection after an interval of t minutes, and so on until t_f, the end.

The general electrical energy consumption (GEEC), represents the operating energy consumption in the HDT established the treatment of CWW [7, 14, 21], was obtained according to Equation 4:

$$GEEC = \frac{V \times I \times t}{60 \times Sv} \qquad (4)$$

where:

CEG, General Electric Energy Consumption in kW.h m^{-3} or Wh dm^{-3};

V is the applied voltage, in *Volts*;

I is the applied current, in *Ampère*;

t is the hydraulic detention time (HDT), in minutes, used for each CWW; and,

Sv is the volume of effluent treated in liters.

The specific electrical energy consumption (SEEC) was calculated according to Asha and Kumar [10] considering the initial and final concentrations of the compounds removed from each CWW studied in the optimized HDT's (Equation 5):

$$SEEC = \frac{V \times I \times t \times 1000}{60 \times (C_0 - C_t) \times Sv} \qquad (5)$$

where:

CEE, Specific Electricity Consumption in kW.h 1000^{-1} units of the compounds removed (kW.h kg^{-1} for ST's and COD; and, kW.h $kNTU^{-1}$ for turbidity);

C_o and C_t, the initial and final concentration, respectively of each variable analyzed over time.

The mass of electrode consumed (Mel) is defined mathematically by equation 6 (9, 14, 21]:

$$\mathrm{Mel} = \frac{\mathrm{I} \times \mathrm{t} \times \mathrm{M}}{\mathrm{z} \times \mathrm{F}} \quad (6)$$

where:

Mel = maximum amount of electrode consumed, in g;

t = time of application of the current, in s;

M = molar mass of the electrode material, which for aluminum is 26.982 g mol^{-1};

z = number of electrons involved in the oxidation reaction of the anode element (for aluminum, z = 3);

F = Faraday constant, 96,485 C mol^{-1}.

The cost of operating the system operating under optimized conditions in continuous flow was calculated according to equation 7 [7, 9]:

$$C_{\text{Operation}} = (a \times Mel) + (b \times CEG) \quad (7)$$

where:

$C_{\text{Operation}}$ = Operating cost, US$ m^{-3} treated effluent

a = mass cost of aluminum used in the plate (US$ 0.54 kg^{-1}; Cempre [22]

b = Energy cost (US$ 0.056 $kW.h^{-1}$; Escelsa [23]

RESULTS AND DISCUSSION

The physical-chemical characterization of coffee wastewaters without recirculation (CWW) used in the treatments is shown in Table 1. The values are close to those found by Eustáquio et al. [24], Matos et al. [12], Selvamurugan et al. [25] and Ulavi and Kumar [26] when they studied this effluent without recirculation.

The analyzed variables expose values above the limits established by the legislation for launching without treatment in a water course [27], highlighting the importance of treatment. The high values of electrical conductivity (EC) are due to the high concentration of potassium in the solution, peculiar to fruit peels [28], justifying the importance given by KUOKKANEN et al. [9] to this variable in electrochemical treatments.

Figure 2 illustrates the visual aspect of CWW samples collected in the reactor in the condition: PD 20 mm, CD 100 A m^{-2} and Time levels (0 to 1070 s), with samples 10 to 15 being collected in longer treatment times than used in the designs and used in this case only for example. It is noteworthy that the lowest turbidity was that of sample 4 (253 NTU), after that moment a whiter but unclear color of the effluent was observed, which contributed to increase turbidity.

The analysis of the raw data showed that the lowest average turbidity occurs at the Time levels of 418 s, PD of 20 mm and CD of 62.5 A m^{-2}. Thus, after the application of the RSM techniques, at these points the adjusted models that describe the response surfaces for the turbidity behavior as a function of Time, CD and PD (Figure 3), are represented by Equations 8, 9 and 10:

$$\text{Turbidity}_{\text{Time:418 s}} = 445.81 + 11.374{*}CD - 0.0237{*}CD^2 - 1.722{*}PD - 0.325{*}CD{*}PD \quad (8)$$

$$\text{Turbidity}_{\text{PD:20mm}} = 910.51 + 2.548{*}CD - 0.0237{*}CD^2 - 3.074{*}Time + 0.00438{*}Time^2 + 0.00552{*}CD{*}Time \quad (9)$$

$$\text{Turbidity}_{\text{CD:62,5A m}^{-2}} = 806.20 + 9.547{*}PD - 1.216{*}Time + 0.00438{*}Time^2 - 0.0756{*}PD{*}Time \quad (10)$$

where: Final turbidity (NTU) of CWW after treatment; Time is the hydraulic detention time (seconds); CD is the Current Density (A m^{-2}) and PD is the Plate Distance (mm), under the evaluated conditions.

It is noteworthy that the factors Time and CD presented significant quadratic terms at 5%, while the PD linear relation. Mollah et al. [14] explain that the distance between the electrodes (plates) is a linear relation of the Electrical Conductivity (EC), electrical resistance of the effluent and CD, when applied in parallel plate systems subjected to a direct current source and immersed in an electrolyte, representing the present study.

The quadratic behavior of the terms Time and CD are related to the change in turbidity in the system. In all the experiments performed, it was observed in the initial minutes that turbidity decreased and, after a few minutes, it increased, in many cases, to levels greater than that in the untreated sample. Such oscillation may be related to the appearance of a whitish color, especially when applying higher levels of Time and CD. The formation of molecules that complex with Al^{3+} [9], the formation of clots with the solids present and the precipitation at specific pH ranges, by changing the concentration of the formed species and their proportional relation [29, 30], and the subsequent flotation by the generation of gases *in situ* at the cathode [21], may have contributed to this achievement.

Time squared with positive sign (Equation 4 and 5) allowed us to suppose that it represents a minimum point. After applying the mathematical procedures under the Equations that represent this factor, the optimal response of Time is detected in 345 seconds and, CD and PD, 95.12 A m^{-2} and 27.27 mm, respectively. The values found represent the points from which the response variable (Turbidity) has the lowest average levels, a behavior of interest in this study.

The greater the distance between the electrodes (plates), the greater the difference in potential applied [14], a fact that can be seen in Figure 3 (a). Smaller CD's can be applied in order to minimize energy consumption, however, the experimental region of the other factors may not be displaced due to physical or instrumental reasons, therefore, it is suggested to work the best conditions within the experimental space studied [31].

Through Figure 3 (bc) it can be seen that Times between 300 and 400 seconds provided lower levels of turbidity and, consequently, greater removal. The largest CD and PD provided treatments with less turbidity (Figure 3a), probably due to the higher generation of aluminum hydroxides and gases (H_2) that acted, respectively, on the flocculation and flotation of solids [21], reducing treatment time.

Lower average levels of Total Solids (TS) were detected in the raw data in Time of 285 s, PD of 20 mm and CD of 62.5 A m^{-2}. After applying the RSM at these intervals, the adjusted models that describe the response surfaces for the behavior of this variable in the treated effluent as a function of Time, PD and CD (Figure 4), are represented by Equation 11, 12 and 13:

$$ST_{Time:285s} = 16{,}237.7 - 8.622*CD + 1.341*CD^2 - 204.72*PD + 10.51*PD^2 - 7.327*CD*PD \quad (11)$$

$$ST_{PD:20mm} = 17{,}443.8 - 148.55*CD + 1.341*CD^2 - 7.092*Time + 0.0113*Time^2 - 0.0232*CD*Time \quad (12)$$

$$ST_{CD:62.5\ A\ m^{-2}} = 23{,}061.8 - 693.32*PD + 10.51*PD^2 - 10.698*Time + 0.0113*Time^2 + 0.1076*PD*Time \quad (13)$$

where: TS is the concentration of total solids from the treated CWW ($mg.L^{-1}$).

The optimization of the equations by

mathematical procedures showed minimum levels of TS at 321 to 374 seconds of time (Figures 4bc). As for the CD and PD factors, these provide lower concentrations of the variable in question at 58.66 A m^{-2} and 31 mm, respectively (see Figure 4 a b c).

The quadratic terms of the Time factor in the RSM equation for turbidity and TS can be explained, since, with the action of the Time factor at constant PD and CD, the fixed dissolved solids of the effluent itself agglutinate with the hydroxides generated in the reactor forming small flakes, increasing turbidity. Consistent with this statement are Valente et al. [32] and Casillas et al. [33], which even observed an increase in dissolved solids in the dairy effluent treated by the electrolytic medium.

The concavity observed in the combination of the factors Time x CD (Figure 4 b) and to a lesser extent in Figures 4 (a) and 4 (c) corroborate the quadratic adjustment of the terms.

The conduct shown by the COD variable is consistent with that presented by the TS, with the exception of the Time x CD ratio, which was not significant at 5%, which demonstrates that the applied current density may not favor the removal of COD over time.

Equations 14 and 15 and Figure 5 describe and illustrate the phenomenon as explained earlier, in which the performance of factors on the behavior of the COD variable is observed, resulting in a concave surface, assuming an adjustment of quadratic terms.

$$COD_{Time:488s} = 32{,}373.16 - 195.137*CD + 1.898*CD^2 - 659.637*PD + 16.885*PD^2 - 1.250*CD*PD \quad (14)$$

$$COD_{CD:57{,}7A\ m^{-2}} = 28{,}731.17 - 617.76*PD + 16.88*PD^2 - 11.204*Time + 0.0175*Time^2 - 0.233*PD*Time \quad (15)$$

where: COD is the concentration of chemical oxygen demand of the treated CWW ($mg.L^{-1}$).

The lowest average concentrations of COD were detected in the raw data in PD of 20 mm, CD of 57.7 A m^{-2} and Time of 488 s, intervals used in the generation and adjustment of the response surface equation.

By the RSM techniques employed, the minimum levels of COD occurred at 470 seconds of time, CD of 58.6 A m^{-2} and PD of 21 mm (Figure 5), optimizing the system's response. The performance of the quadratic terms for the factors in this variable demonstrates that the treatment time can act as a limiter in the removal efficiency, contradicting some studies that point out that the longer the treatment time, the greater the removal of the COD [10, 34-37].

The lower levels of COD were evidenced in average levels of CD's and PD's, not requiring a high current applied to smaller PD's, as evidenced by Tchamango et.al. [38] and Chen et. al. [44] in the electrochemical treatment by batch of synthetic milk and restaurant effluent, respectively. In both works, the authors attributed this behavior to the presence of dissolved organic compounds, which are difficult to remove by processes that use coagulation as a form of separation.

The observation reinforces the evidence that the mechanism for removing COD by electrolytic treatment is not yet fully understood, especially when ionic species and compounds soluble in the effluent are present [33, 34]. It is also reported that effluents with high concentrations of dissolved organic compounds, sugars and carbohydrates with high water solubility, such as CWW [1], have low efficiencies for removing COD by electrofloculation [33, 39].

Asha and Kumar [10], using aluminum electrodes to treat CWW in India, report that they obtained a low removal efficiency

(30%) of COD in the first 15 minutes of treatment. The authors attribute the fact that the evolution of hydrogen at the cathode has not yet started. They pointed out, however, that after 60 minutes of electrolysis the COD removal rate was close to 90% and up to 93%, in 75 minutes. However, it is worth noting that the authors did not evaluate the behavior of turbidity or the formation of hydroxides that could make the treatment unfeasible.

In the meantime, it is noteworthy that the moment when the Times showed a sudden increase in turbidity, apparently made the efficiency limit for removing the monitored variables.

As for the behavior of the other assisted variables, the pH showed a progressive and linear increase ($R^2 = 0.60$), starting from an initial average of 4.78 to a final average of 5.40 (Equation 16). The observed fact is common in electrolytic treatment and has already been evidenced by other authors [40 - 43). Chen [44], attribute the increase in pH to the reduction of hydrogen in the water molecule at the cathode.

$$Ph_{CWW} = 5.2505 + 0.0015*Time - 0.0497*PD + 0.0074*CD \quad (16)$$

According to Equation 16, greater increments, in terms of pH min^{-1} units, occur in time levels and, above all, higher CD's. This increase is due, certainly, to the greater generation of hydroxyls, products of hydrolysis, which occurs at the cathode [9] when greater CD and time are applied. The negative contribution of the PD factor, on the other hand, is due to the lower production of hydroxides and gases as the plates move away [9]

As for the electrical conductivity (EC), it did not show a significant linear adjustment at 5% and outlined a low determination coefficient ($R^2 = 0.43$), indicating that it is not well explained by the variation in the levels of the factors. The removal of solids may have contributed to lower EC, but, on the other hand, long Hydraulic Detention Times and high CD's can contribute to raise the levels of EC by the incorporation of metal ions in the solution [45], with no direct proportional relation.

The relation observed between the variables monitored in this study strengthens the hypothesis of having a maximum treatment time, since long times can compromise the quality of the effluent, the consumption of electricity and wear of electrodes. Thus, it is assumed that times higher than those presented in this research (average of 320, 348 and 462 s for Turbidity, TS and COD, respectively) could make the treatment via electrolyte unviable, which contradicts some studies that evaluate only the final efficiency of the treatment and not the performance of the treatment in intervals of time.

Table 2 shows the ranges of factors optimized by the RSM equations and methods previously described. The optimization intervals for the factors presented in Table 2, considering the same variable, are due to the interaction between the factors, which do not happen in the same way for the same factor when varying the others.

Figure 6 shows the behavior of the variables turbidity, total solids and COD in values observed (O) in experiments in conditions close to the modeled and predicted (P) by the equations in the intervals of the optimized factors (Table 2) of CWW. As can be seen in Figure 6, the predicted values for turbidity are above those observed, while total solids below and reasonable COD (Figure 6). The turbidity peaks may have influenced the factors used in the modeling, overestimating future values of this variable.

In this sense, it is detected in Table 3 that the accuracy factor showed a variation in the

P/O ratio (Predicted/Observed) of 59, 16 and 13% in the turbidity, solids and COD data, respectively. The registered variations express the agreement (adjustment) of the model between the result of a test (observed) and the reference value (predicted) accepted as conventionally true [46], thus, the coefficients found show the average deviation between the data (P/O).

For the bias factor, values close to 1 express little overestimation of the model over the observed data [19]. It is noteworthy that the predictive data of the model adjustment overestimated turbidity by 59%, underestimated total solids and COD by 14% and 2%, respectively, on the observed data (Table 3). The oscillation of turbidity over time by the formation of hydroxides and solids by the generation of gases *in situ* at the cathode [21] may have contributed to this, since the results are modeled on the observed data.

For the sake of explanation, the deviations found show that the turbidity predicted by the model ranges from 301 to 766 NTU, total solids from 6,537 to 8,777 mg L^{-1} and COD from 17,486 to 22,401 mg L^{-1}, values close to those observed, corroborating with the results of the Bias factor. The agreement (adjustment) between the result of an experiment (observed) and the reference value (predicted) expresses that the model behaved conventionally within the accepted as true [46].

The COD is the one that expresses the least deviation and estimation, being closer to the real values by the model, followed by total solids. Turbidity, on the other hand, showed an overestimation and high deviations by the model generated, since the values significantly differed from the unit (1), which demonstrates a great over/underestimation and deviations from the model with respect to the observed data [19]. The observed suggests that the model, even adjusted to the data, is used considering the previous observations in the turbidity estimate.

The simulation of the reactor in continuous flow in the optimized conditions provides more information about the above statements. Figure 7 illustrates the behavior of the average levels of the turbidity variables, total solids and COD in the 8 (eight) sample collections at the reactor outlet during simulations (S) in continuous flow and the values predicted (P) by the models, under conditions optimized for the CWW.

The combination of optimum points of the factors (Time, CD and PD), resulting from the optimization, may have favored a lower final concentration of turbidity and total solids in the treatment. Higher levels of simulated COD above those observed, corroborating what was observed by Valente [34], show that the mechanism for removing COD by electrolytic processes is not yet fully elucidated.

The removal efficiencies for the variables turbidity, total solids and COD, in addition to changes in pH, electrical conductivity and temperature, under the optimized conditions, can be seen in Table 4. The optimized condition favored the generation of gases (mainly H_2) and coagulating agents ($Al(OH)_3$) helping to remove suspended solids, which are the precursors of turbidity [21], but not being efficient in removing the dissolved fraction (± 75%) of ST's from CWW's [14].

As for COD, sufferable removals occurred due to the compounds that make up that fraction in the CWW are, for example, sucrose, glucose, fructose and other dissolved solids, which depend on the electro-oxidation mechanism for removal, the last process involved in this treatment system [14, 33, 47], which depend on high times for all electrochemical reactions to happen.

The pH, electrical conductivity and temperature showed greater changes in absolute terms with the increase in HDT in the reactor. In the case of pH, this increase is certainly due to the greater generation of hydroxyls, products of hydrolysis, which occurs at the cathode [9]. Long HDT's may have contributed to increase the incorporation of metal ions in the solution [45] and increase the Joule effect by increasing conductivity and temperature, respectively [47].

The removal rate is an important indicator in the evaluation of wastewater treatment. Higher removal rates were evidenced for the variables turbidity (133.01 NTU min^{-1}) and total solids (143.56 mg L^{-1} min^{-1}), possibly related to the flotation mechanism of suspended solids in the initial minutes of treatment [9, 14, 21, 47], while lower COD removal rates (20.66 mg L^{-1} min^{-1}) are related to low HDT.

The general and specific electrical energy consumption of each variable studied in the CWW under the optimized conditions is shown in Table 5. It is observed that the highest general consumption is related to the highest HDT of the treatment [7, 14, 21] and the lowest removal rate in the time of COD. This analysis becomes relevant, as it relates to energy consumption efficiency for removing the compound [10].

The general consumption is slightly higher than the values found by Phalakornkule et al. [48] for effluents containing dyes using iron electrodes (1.42 kW.h m^{-3}), by Phalakornkule et al. [49] for textile effluents using iron and aluminum electrodes (1.62 kW.h m^{-3}), probably due to the higher commercial value of aluminum.

COD showed higher energy consumption in the treatment of CWW when compared to other studies. Asha and Kumar [10], using aluminum electrodes to treat CWW that had already undergone a pretreatment by an anaerobic reactor, had a cost of 1.102 kW.h kg^{-1} using a CD of 93.87 A m^{-2}.

In another study, Asha and Kumar [50] found costs of 3.61 kW.h kg^{-1} of COD using aluminum electrodes and 40.0 kW.h kg^{-1} using iron electrodes, in addition to superior efficiencies for aluminum electrodes when compared to iron electrodes. For the authors, the superior flocculation power of aluminum ions, related to the greater specific surface area and rapid dissolution in the solution, favored the quick adsorption capacity to the organic compounds present in the effluent to aluminum instead of iron, also reflecting in lower aluminum consumption.

Valente et al. [34] observed that the operating cost of removing COD from dairy effluent went from US$ 0.19 m^{-3} to US$ 0.10 m^{-3} when removal increased from 50 to 70%, at an applied current of 12.3 A m^{-2} and initial COD ranging between 951 to 3,170 mg L^{-1}.

The cost of US$ 0.13 m^{-3} is below the US$ 0.54 m^{-3} obtained by Kobya et al. [42] evaluating an EF system with iron electrodes for the treatment of effluents containing dyes (*Red* 3B). For dairy effluent using iron electrodes, Kushwaha et al. [51] observed a cost of US$ 0.96 m^{-3} of treated effluent for a 70% reduction in COD.

The results obtained corroborate with Bayramoglu et al. [52] and Bayramoglu et al. [53] who report that the highest cost of treatment by electroflotation is due to the consumption of electricity. Gardiman Junior et al. [54] observed that, even with the lower currents (0.5 A, which in this study is equivalent to 25 A m^{-2}), there were higher costs in the consumption of electricity than in the material used.

CONCLUSIONS

Based on the results of the experiments conducted and methodologies employed, it is concluded that:

CWW has high levels of organic matter expressed in COD, sugars and phenolic compounds, in addition to potassium, electrical conductivity, total nitrogen and total solids.

The experiments conducted showed an average removal of 75% for turbidity, 11% for total solids and 13% for COD. The pH increased in average from 0.11 to 0.78 depending on the levels of Time, PD and CD applied. The EC outlined changes from -0.24 to 0.13 dS m^{-1} in the treatments, with no pattern of behavior.

The turbidity fluctuated over time in the treatments, showing even greater increments than the initial ones, suggesting that it is the main limiting variable of the treatment.

Adjusting the pH to values below 5.0 could assist in the oxidation of the metal used in the anode, avoid passivation, provide stabilization of turbidity and greater removal of total solids and COD.

The optimization outlined different values for the monitored variables, expressing the particularities of each one. In effect, it is understood that the average of the factors studied (Time, PD, CD) can be used in future experiments or treatments in continuous flow. Therefore, values of 376 seconds of treatment time, 70.0 A m^{-2} of current density and 26.5 mm of plates distance, are presented as the average of the optimized variables.

The values predicted by the model were recorded by overestimating turbidity by 59% and underestimating total solids and COD by 14 and 2%, respectively.

In the treatment of CWW in continuous flow in the optimized conditions, there were lower levels of turbidity and total solids and higher for COD in relation to the values predicted by the model.

The removal of variables in the reactor in continuous flow was 68.35%, 25.55% and 3.68%, for turbidity, total solids and COD, respectively. The pH, electrical conductivity and temperature increased after treatment.

Higher specific electrical consumption was observed for COD, possibly due to the high concentration and low removal in the evaluated time.

Electricity represented 78.8% of the total operating cost under the conditions of the studied system.

Even though the simulation has demonstrated more satisfactory removals than the model itself, the analyzed variables still show values above the limits established by the legislation for launching untreated water in course. The improvement in the efficiency and costs of removing the monitored variables could be achieved by combining biological or tertiary processes with the electrolytic treatment, assisting in the degradation of organic matter and dissolved solids from the effluent.

The study made it possible to assess, based on the elements of the total operational cost of treating coffee wastewater without recirculation, the potential use of the technique when compared to other forms of treatment and the possibility of using this technology on larger scales.

ACKNOWLEDGMENT

The authors would like to thank the Federal Institute of Espírito Santo for the financial contribution in translating and publication this manuscript.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest in the research and that the research results can be published by this journal.

ETHICAL CONDUCT

The authors declare that the manuscript was not submitted to another publication simultaneously. The submitted work is original and has not been published elsewhere in any form or language.

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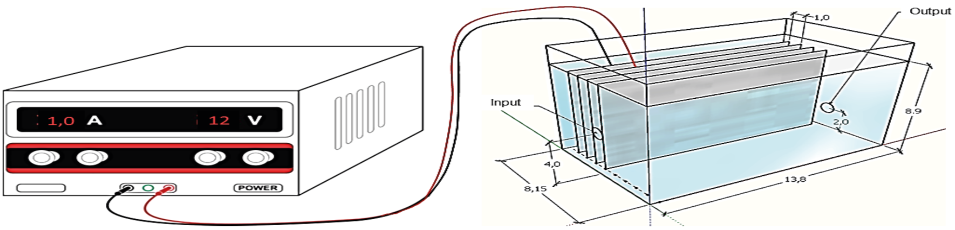

Figure 1 – Schematic representation of the electrolytic reactor with the electrical source. Dimensions in centimeters.

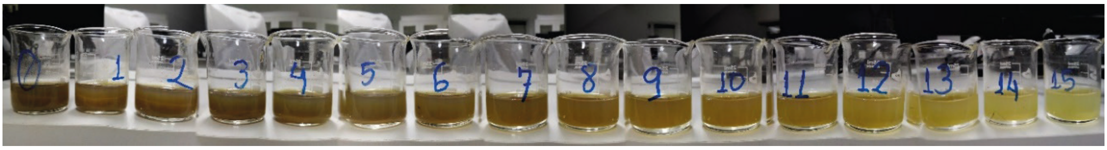

Figure 2 – Aspect of coffee wastewater without recirculation treated in the condition: Plate distance of 20 mm, Current Density of 100 A m^{-2} and collection interval in the reactor from 0 to 1070 s.

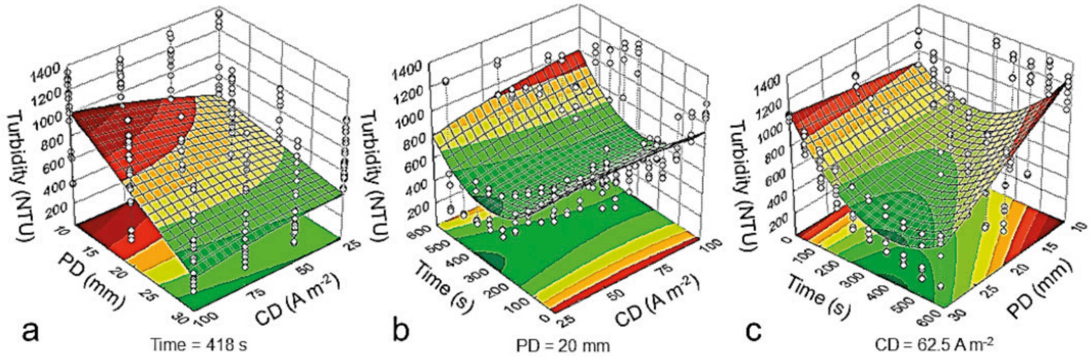

Figure 3 – Response surface for the behavior of the variable Turbidity as a function of (a) Plate Distance (PD) and Current Density (CD) for the Time of 418 s, (b) Time and CD for PD of 20 mm and, (c) Time and PD for CD of 62.5 A m^{-2} in the electrolytic treatment of coffee wastewater without recirculation.

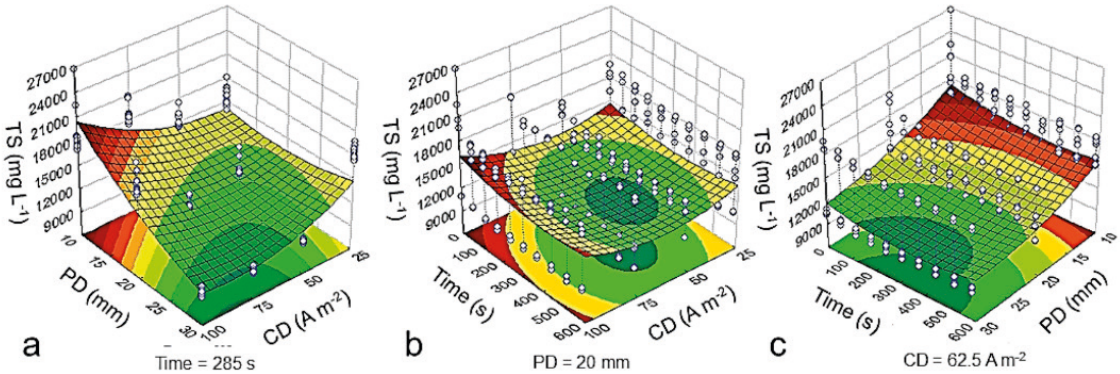

Figure 4 – Response surface for the behavior of the variable total solids (TS) as a function of (a) Plate Distance (PD) and Current Density (CD) for the Time of 285 s, (b) Time and CD for PD of 20 mm and, (c) Time and PD for CD of 62.5 A m^{-2} in the electrolytic treatment of coffee wastewater without recirculation.

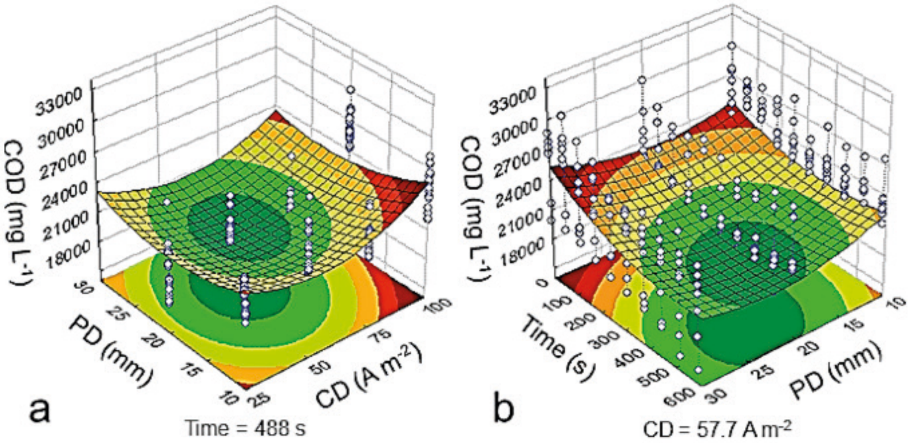

Figure 5 – Response surface for the behavior of the variable Chemical Oxygen Demand (COD) as a function of (a) Plate Distance (PD) and Current Density (CD) for 488 s Time, (b) Time and PD for CD of 57.7 A m^{-2} in the electrolytic treatment of coffee wastewater without recirculation.

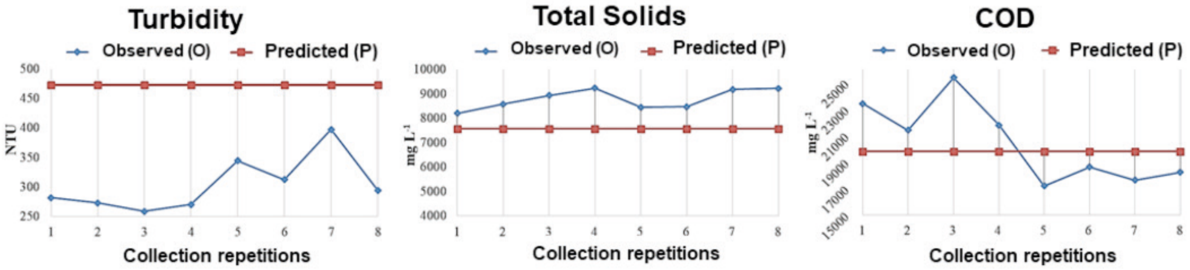

Figure 6 – Predicted (P) and observed (O) values of the variables turbidity, total solids and chemical oxygen demand – COD for coffee wastewater without recirculation.

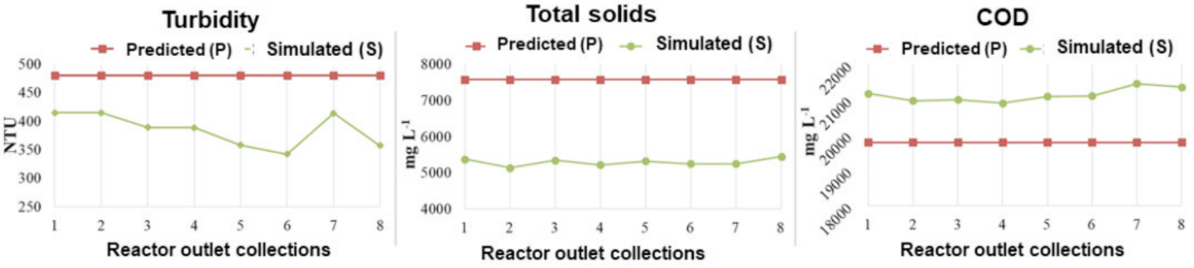

Figure 7 – Average behavior of the variables turbidity, total solids and chemical oxygen demand (COD) in simulations (S) (collections at the reactor outlet) in continuous flow and predicted values (P) by the models, under conditions optimized for coffee wastewater without recirculation.

Variable	Unity	Average Values
Sugars	mg L^{-1}	6,950 ± 36.5
EC	EC	1.43 ± 0.45
Cor	PCU	3,610 ± 193
COD	mg L^{-1}	26,800 ± 8,725
PC	mg L^{-1}	212.0 ± 8.1
K	mg L^{-1}	471.4 ± 55
tN	mg L^{-1}	12.2 ± 0.8
DO	mg L^{-1}	5.1 ± 0.3
pH	----	4.7 ± 0.4
tP	mg L^{-1}	16 ± 1.2
TS	mg L^{-1}	15,818 ± 7,184
Turbidity	NTU	1,008 ± 141

Table 1 – Physical-chemical characterization of coffee wastewater without recirculation (CWW)

EC: Electrical Conductivity; COD: Chemical Oxygen Demand; PC: Phenolic Compounds; K: Potassium; tN: Total nitrogen; DO: Dissolved Oxygen; tP: Total Phosphorus; TS: Total Solids; NTU: Nephelometric Turbidity Units; PCU: Platinum Cobalt colored units.

Variable	Time (seconds)	CD (A m^{-2})	PD (mm)
Turbidity	295 to 345	88.1 to 95.1	27 to 29
Total Solids	321 to 374	58.5 to 61.9	28 to 31
COD	453 to 470	57.6 to 58.4	21.4 to 21.7

Table 2. Intervals of the independent factors studied to optimize an electrolytic system in the treatment of coffee wastewater without recirculation (CWW).

COD: Chemical Oxygen Demand; CD: Current Density; PD: Plate Distance.

	Turbidity		**Total Solids**		**COD**	
	Accuracy	Bias	Accuracy	Bias	Exatidão	Bias
P/O (Adjustment)	1.59	1.59	1.16	0.86	1.13	0.98

Table 3 – Adjustment factors Accuracy and Bias of the predicted over observed values (P/O) for each variable monitored in the validation in the optimized conditions of coffee wastewater without recirculation

COD: Chemical Oxygen Demand.

Effluent	**Removal (%)**			**Alteration**		
	Turbidity (NTU)	TS (mg L^{-1})	COD	pH	EC (mS cm^{-1})	T (°C)
CWW	68,35	25,55	3,68	0,36	0,05	0,25

Table 4 – Removal efficiencies (%) for the variables turbidity, total solids and chemical oxygen demand (COD) and change in absolute terms for pH, electrical conductivity and temperature in the treatment of coffee wastewater without (CWW) under optimized conditions

TS: Total Solids; COD: Chemical Oxygen Demand; EC: Electrical Conductivity; T: Temperature.

Effluent	**General**	**Turbidity**	**TS**	**COD**	**Material**	**Energy**	**Total**
	(kW.h m^{-3})	(kW.h $kNTU^{-1}$)	(kW.h kg^{-1})			US$ m^{-3}	
CWW	1.75	2.09	1.21	8.70	0.027	0.10	0.13

Table 5 – Consumption of general and specific electricity per variable, material cost (aluminum), electricity consumed and total operational wastewater treatment for coffee without recirculation (CWW)

TS: Total Solids; COD: Chemical Oxygen Demand.