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Application of the statistical experimental design to optimize the electrocoagulation technology in the treatment of cosmetic industry wastewater

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ABSTRACT

This research presents an experimental design study to statistically optimize electrocoagulation (EC) technology with the use of aluminum (Al) and iron (Fe) electrodes applied in the treatment of cosmetic industry wastewater. The experimental design was conducted according to the rotational central compound design, composed of 12 trials to evaluate the joint influence of two independent variables, current density and operation time. Considering the efficiency of the variable responses obtained through the analysis of variance (ANOVA) in relation to the removal of chemical oxygen demand (COD), turbidity, apparent color and absorbance at λ – 350 nm, the experimental results demonstrated with 95% significance of the independent variables. The operating conditions optimized for the EC technology corresponded to: 1,672 A m^{-2} with operation time of 18.98 min for the Al electrode and 996 A m^{-2} with operation time of 29.54 min for the Fe electrode. These conditions were considered promising alternatives for the maximization of COD (66.12% Al and 61.35% Fe), turbidity (90.96% Al and 92.31% Fe), apparent color (97.07% Al and 93.69% Fe) and absorbance at λ – 350 nm (97.83% Al and 92.69% Fe).

Keywords: Cosmetic industry wastewater; Electrocoagulation; Aluminum or iron electrodes; Statistical optimization; Rotational central compound design

1. Introduction

From the 20th century, cosmetic products began to be produced on an industrial scale, providing a greater variability of products. At the end of this century, this advance later resulted in a significant portion of the cosmetic industry's economic contribution to the global economy [1].

Although cosmetic production is relatively simple, it does not prevent the generation of a variety of contaminants that can be found in its effluents, especially high concentrations of surfactants, dyes, fragrances, oils and greases, surfactants, suspended solids, phosphates and polyphosphates, resulting in a high COD and low biodegradability [1–5].

The use of the abovementioned compounds by the cosmetic industry wastewater can lead to significant impacts on aquatic systems as they are easily absorbed by plants and animals. Consequently, they can accumulate in the human body and are responsible for causing different degrees of

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toxicity, presenting mutagenic and carcinogenic characteristics to living beings [6,7].

Due to the characteristics of cosmetic effluents, the development and enhancement of efficient treatment technologies should be stimulated. This is because, despite the progress of studies reported in the literature on the treatment of cosmetic effluent [4,5], the removal of most of the pollutants has not been fully elucidated.

For the treatment of these effluents, EC technology can be considered an effective option in the removal of persistent organic pollutants, which are difficult to biologically degrade [2,8,9].

Nowadays, the treatment of pollutants specific to the cosmetic industry, such as triclosan [6,10,11], is reported in the literature. However, the principal treatment technologies applied to the cosmetic industry wastewater correspond to the Fenton process [12,13], peroxide oxidation [14], anaerobic sludge blanket reactor [5] and membrane bioreactor [3]. Published research using EC with aluminum and/or iron electrodes for the treatment of cosmetic industry wastewater [2] is limited.

EC technology uses a reactor composed of electrodes made of different metals, such as aluminum and iron, commonly used due to the low cost and easy obtainability [7,8]. The EC results in the dissolution of Al or Fe electrodes in Al^{n+} or Fe^{n+} ions, respectively, giving rise to in situ coagulants, through electrochemical reactions [8,15,16]. Thus, it reduces the probability of by-product generation and minimizes sludge production [8,17–19].

The EC, at the anode of the sacrificial electrode, causes the release of Al or Fe and generates microbubbles of oxygen (O_2), through Reactions (1) and (2). At the cathode, H_2 gas and OH^- are produced from Reaction (3), allowing the coagulation, flocculation and flotation of the compounds to be removed, which favors the formation of insoluble metallic hydroxides from Reaction (4) [17,19]:

$$Me_{(s)} \rightarrow Me^{n+}_{(aq)} + ne^- \tag{1}$$

$$2H_2O_{(l)} \rightarrow 4H^+_{(aq)} + O_2 + 4e^- \tag{2}$$

$$2H_2O_{(l)} + 2e^- \rightarrow 2H_{2(g)} + 2OH^- \tag{3}$$

$$Me^{n+} + ne^- \rightarrow Me_{(s)} \tag{4}$$

It is also considered that the operation time and the current density are some of the main operational parameters of the EC, since they are important in controlling the electrochemical reactions in the reactor [8,20–22].

In this context, rotational central compound design (RCCD) is presented as a strategy to optimize the independent variables, current density and the operation time. The RCCD allows variables that influence EC, which are analyzed simultaneously at different levels and determines the most significant variables for the desired response, hence, minimizing the number of treatments that need to be performed. To this end, an outline consisting of a 2k factor, an axial portion and central points that improve the estimates of the quadratic model and allow additional degrees of freedom [23,24].

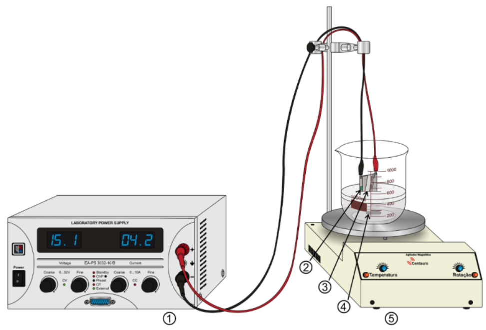

Fig. 1. Electrochemical reactor (batch). (1) DC power supply; (2) insulation material (synthetic rubber); (3) electrodes (Al or Fe); (4) magnetic bar; (5) magnetic stirrer.

Table 1
Values studied for the independent variables

Independent variable	j (A m^{-2})	t (min)
Code	X_1	X_2
–1.41	156.00	4.90
–1	400.00	10.00
0	1,000.00	22.50
1	1,600.00	35.00
1.41	1,840.00	40.10

X_1, current density; X_2, operation time.

Table 2
Characterization of cosmetic industry wastewater

Parameters	Gross effluent
Temperature	25.90°C ± 0.08°C
Apparent color	8,989.25 ± 7.83 uH
Turbidity	818.67 ± 0.94 NTU
pH	5.38 ± 0.02
Conductivity	2,917.67 ± 9.67 μS cm^{-1}
DO	1.78 ± 0.22 mg L^{-1}
COD	1,042.19 ± 43.70 mg L^{-1}
Sedimentable solids	0.09 ± 0.01 mg L^{-1}
TSS	21.67 ± 2.36 mg L^{-1}
FSS	13.33 ± 2.36 mg L^{-1}
VSS	8.33 ± 2.36 mg L^{-1}
OG	57.00 ± 3.00 mg L^{-1}
Abs at λ – 350 nm	0.568 ± 0.003 uA
Abs at λ – 254 nm	0.238 ± 0.005 uA

pH, hydrogenation potential; DO, dissolved oxygen; COD, chemical oxygen demand; TSS, total suspended solids; FSS, fixed suspended solids; VSS, volatile suspended solids; OG, oils and grease; Abs, absorbance.

Thus, the objective of this research was to apply the RCCD with two independent variables, the current density and the operating time, for the treatment of cosmetic industry wastewater by means of EC with Al and Fe electrodes.

Table 3
Efficiency of EC technology using Al and Fe electrodes

T	Independent variables		Response variables (% removal)							
			Al electrode				Fe electrode			
	X_1	X_2	COD	Turbidity	Apparent color	Absorbance at λ – 350 nm	COD	Turbidity	Apparent color	Absorbance at λ – 350 nm
1	1,600.00	35.0	57.66	90.27	97.37	94.54	49.74	88.23	0.00	20.77
2	1,600.00	10.0	68.43	92.51	96.37	93.37	57.48	89.74	82.45	82.39
3	400.00	35.0	61.49	63.84	81.71	76.83	60.89	86.28	84.43	86.97
4	400.00	10.0	40.48	0.00	0.00	0.00	22.16	68.72	0.00	0.00
5	1,000.00	22.5	61.14	94.71	97.80	96.77	58.86	89.04	92.41	78.87
6	1,000.00	22.5	62.86	94.46	97.20	95.89	59.46	86.89	90.29	85.91
7	1,000.00	22.5	61.78	95.36	97.51	96.83	60.52	95.97	96.53	95.42
8	1,000.00	22.5	62.93	95.19	97.76	96.19	58.66	94.46	96.66	94.72
9	1,840.00	22.5	65.83	87.41	97.17	93.37	33.50	86.77	80.18	84.15
10	156.00	22.5	36.47	0.00	0.00	0.00	34.77	42.70	0.00	0.00
11	1,000.00	40.1	69.63	85.91	90.87	95.25	66.44	82.33	79.87	59.51
12	1,000.00	4.9	49.62	0.00	0.00	0.00	46.24	83.22	0.00	0.00

T, Test; X_1, current density A m^{-2}; X_2, operation time (min); COD, chemical oxygen demand.

2. Materials and methods

This research was developed in the Laboratory of Water and Ecotoxicology, located at the Federal University of Southern Frontier (UFFS), Cerro Largo/RS campus, Brazil.

2.1. Cosmetic industry wastewater

The raw effluent without pre-treatment was obtained from a cosmetic industry plant located in the state of Rio Grande do Sul, Brazil. This effluent is a result of the cleaning of production equipment for shampoos, conditioners, hair and body creams, deodorants, sunscreens and makeup.

2.1.1. Characterization of cosmetic industry wastewater

The physical–chemical analyzes of the characterization of cosmetic industry wastewater were performed according to the methodology of the Standard Methods [25], which were: temperature (°C), apparent color (uH), turbidity (NTU) (mg L^{-1}), hydrogen ionic potential (pH), conductivity (µS cm^{-1}), dissolved oxygen (DO) (mg L^{-1}), COD (mg L^{-1}), sedimentable solids (SS) (mg L^{-1}), total suspended solids (TSS) (mg L^{-1}), fixed suspended solids (FSS) (mg L^{-1}), volatile suspended solids (VSS) (mg L^{-1}), absorbance (Abs) (uA), and the content of oils and greases (OG) (mg L^{-1}).

2.2. Experimental module

The electrochemical reactor (batch) consisted of a DC power supply (EA-PS ® 3016-20 B), a beaker with a capacity of 1 L of effluent, a pair of Al electrodes or a pair of Fe electrodes, with 5 × 10 cm dimensions, and a magnetic stirrer (Centaur®, CAMA-15) (Fig. 1).

2.3. Experimental procedure

In order to investigate the efficiency of the treatment of cosmetic industry wastewater by means of the EC, two experimental procedures were performed: (1) use of Al sacrifice electrodes; (2) use of Fe sacrifice electrodes. In the application of EC technology with the use of Al-sacrifice electrodes, 25 cm^2 of these electrodes were immersed in the cosmetic industry wastewater. The electrode pair was electrically isolated by synthetic rubber at a distance of 1 cm. The volume for treatment was set at 500 mL, a stirrer at 600 RPM and a room temperature of 25°C ± 3°C. The conductivity of the cosmetic effluent was adjusted by addition 6.20 g L^{-1} of NaCl.

The time of stabilization of the electrocoagulated effluent was fixed in 30 min. Later, to identify the best operating conditions of the electrochemical reactor, analyzes of the physical–chemical parameters COD, turbidity, apparent color and absorbance at λ – 350 nm were performed. The same experimental procedure performed with Al sacrifice electrodes was adopted in the application of EC with Fe sacrifice electrodes.

2.4. Experimental planning and statistical analysis

Preliminary screening tests were performed, considering current density and operation time, according to the values

Table 4
Analysis of variance for the Al and Fe removal variable for the 22 factorial design

	Variation source	SS	d*f*	MS	*F*		*p*
					cal	tab	
Aluminum electrodes	COD removal						
	Regression	1,229.8	4	307.4	23.3	4.1	0.0004
	Sediments	92.2	7	13.2			
	Total	1,322.0	11				
	Turbidity removal						
	Regression	18,563.1	5	3,712.6	19.1	4.4	0.001
	Sediments	1,163.9	6	194.0			
	Total	19,727.0	11				
	Apparent color removal						
	Regression	20,479.8	5	20,479.8	18.4	4.4	0.001
	Sediments	1,343.5	6	1,343.5			
	Total	21,823.3	11	21,823.3			
	Absorbance at λ – 350 nm						
	Regression	19,872.3	4	4,968.0	18.4	4.1	0.0002
	Sediments	1,223.8	7	174.8			
	Total	21,096.2	11				
Iron electrodes	COD removal						
	Regression	2,085.0	5	417.0	24.8	4.4	0.0006
	Sediments	101.2	6	16.9			
	Total	2,186.2	11				
	Turbidity removal						
	Regression	2,277.0	5	455.4	5.78	4.4	0.03
	Sediments	473.6	6	78.9			
	Total	2,750.6	11				
	Apparent color removal						
	Regression	20,924.9	5	4,185.0	7.7	4.4	0.01
	Sediments	3,242.9	6	540.5			
	Total	24,167.8	11				
	Absorbance at λ – 350 nm						
	Regression	17,557.8	5	3,511.5	9.4	4.4	0.008
	Sediments	2,237.0	6	372.8			
	Total	19,794.8	11				

SS, sum of square; d*f*, degree of freedom; MS, mean of square; *F*, Fisher's ratio; *p*, probability; COD, chemical oxygen demand; Abs., absorbance at λ – 350 nm.

described in the study by Boroski et al. [2]. Based on the preliminary test results, the current density values were set according to the minimum and maximum capacity of the power supply. The RCCD considered two independent variables: current density (X_1) and operation time (X_2). For this purpose, a complete factorial design 2^2 was performed, including four axial points and four repetitions at the central point, totaling 12 tests [26] for each pair of electrodes (Al or Fe) (Table 1).

The values of the response variables are shown in Table 1, factor scores (–1 and +1) indicate the minimum and maximum level of each variable, the center point (0) in quadruplicate provides a measure of the pure error, and the points, and the axial points (–1.41 and +1.41) determine the quadratic terms [23,27,28].

To optimize the EC, as for the response variables X_1 and X_2, the physico-chemical parameters of COD, turbidity, apparent color and absorbance at $\lambda - 350$ nm were considered. The experimental results were submitted to statistical analysis by the software STATISTICA® 11 and ANOVA, which considers the terms of the model statistically significant for a confidence level of 95% (p-value < 0.05). For the results of the physico-chemical parameters, the global desirability method was applied and the EC technology was optimized in the best adjustment condition of variables X_1 and X_2 (Table 1).

Subsequently, the results of the analytical parameters for COD, turbidity, apparent color and absorbance at $\lambda - 350$ nm were analyzed by the statistical test called Test-t. Thus, it was possible to identify, with a significance level of 95% ($p < 0.05$), the similarity or discrepancy between the averages of the results obtained after the treatment of the cosmetic industry wastewater with Al and Fe sacrifice electrodes by EC. Thus, the following hypotheses was considered, H_0: the means are statistically equal ($p > 0.05 - H_0$ is accepted); and H_1: the means are statistically different ($p < 0.05 - H_0$ is rejected).

3. Results and discussion

Table 2 shows the results obtained regarding the characterization of cosmetic industry wastewater. The high

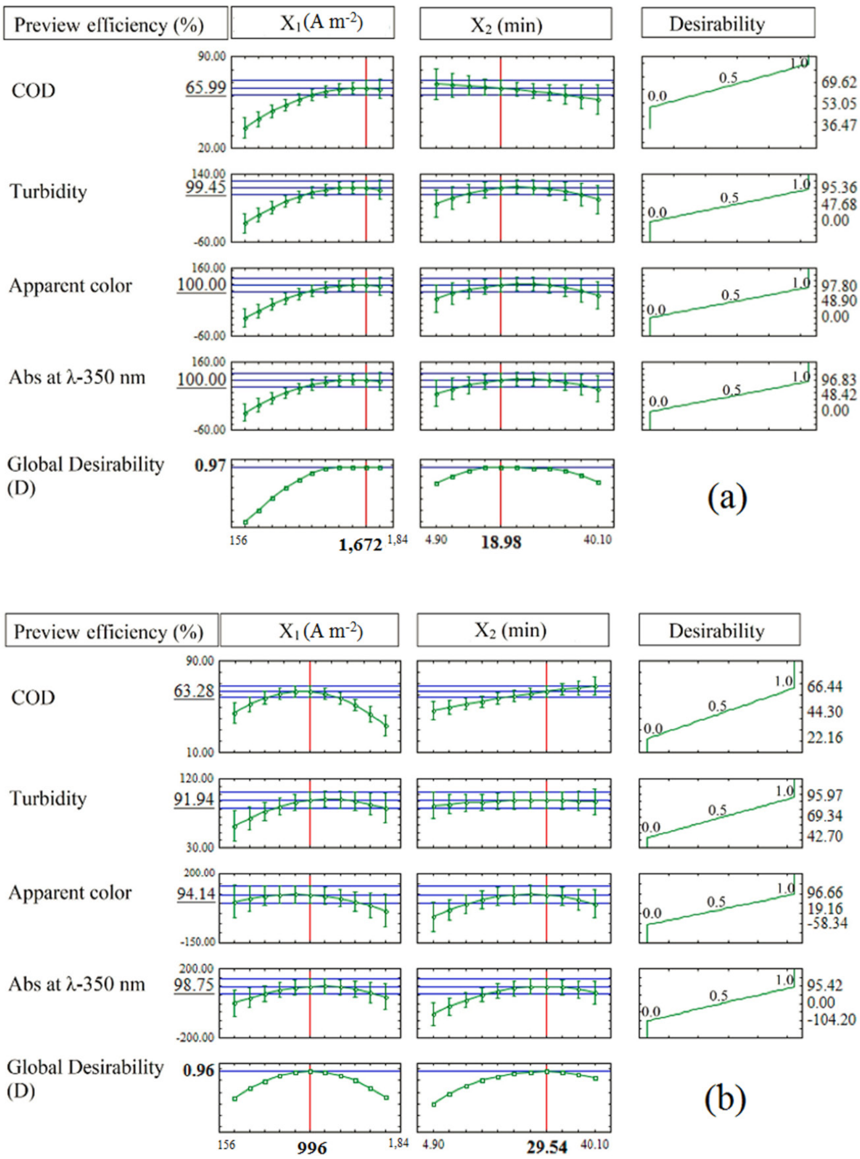

Fig. 2. Simultaneous optimization of the response variables by means of the global desirability function for (a) Al and (b) Fe. COD, chemical oxygen demand; Abs, absorbance.

COD content, turbidity and apparent color in the effluent, which is an indication of a high content of suspended matter and dissolved solids [29] are observed. The maximum absorbance detected at λ – 350 nm corresponds to conjugated aromatic rings. Additionally, it is necessary to emphasize that the original effluent from the factory presents a high ion content.

3.1. Results of experimental planning and statistical analysis

Table 3 shows the results of the EC efficiency in the treatment of cosmetic industry wastewater with the use of Al and Fe electrodes.

As shown in Table 3, the best COD removal efficiency results are found in Test 11, providing removals of 69.63 % using Al sacrifice electrodes and 66.44 % using Fe sacrifice electrodes used in EC. However, in the simultaneous analysis of the results presented in Table 3, it is verified that the higher removal efficiency of the physico-chemical parameters occurred in tests 7 and 8, in the conditions of operation time and current density of 22.5 min and 1,000 A m^{-2}.

Boroski et al. [2] studied combined electrocoagulation and TiO_2 photo-assisted treatment applied to wastewater effluents from pharmaceutical and cosmetic industries. A removal efficiency of about 91%, 86% and 94% of the turbidity, COD and an absorbance at λ – 350 nm, respectively, was obtained, with a current density of 763 A m^{-2}, however, with a high treatment operation time of 90 min and an initial pH of 6.0.

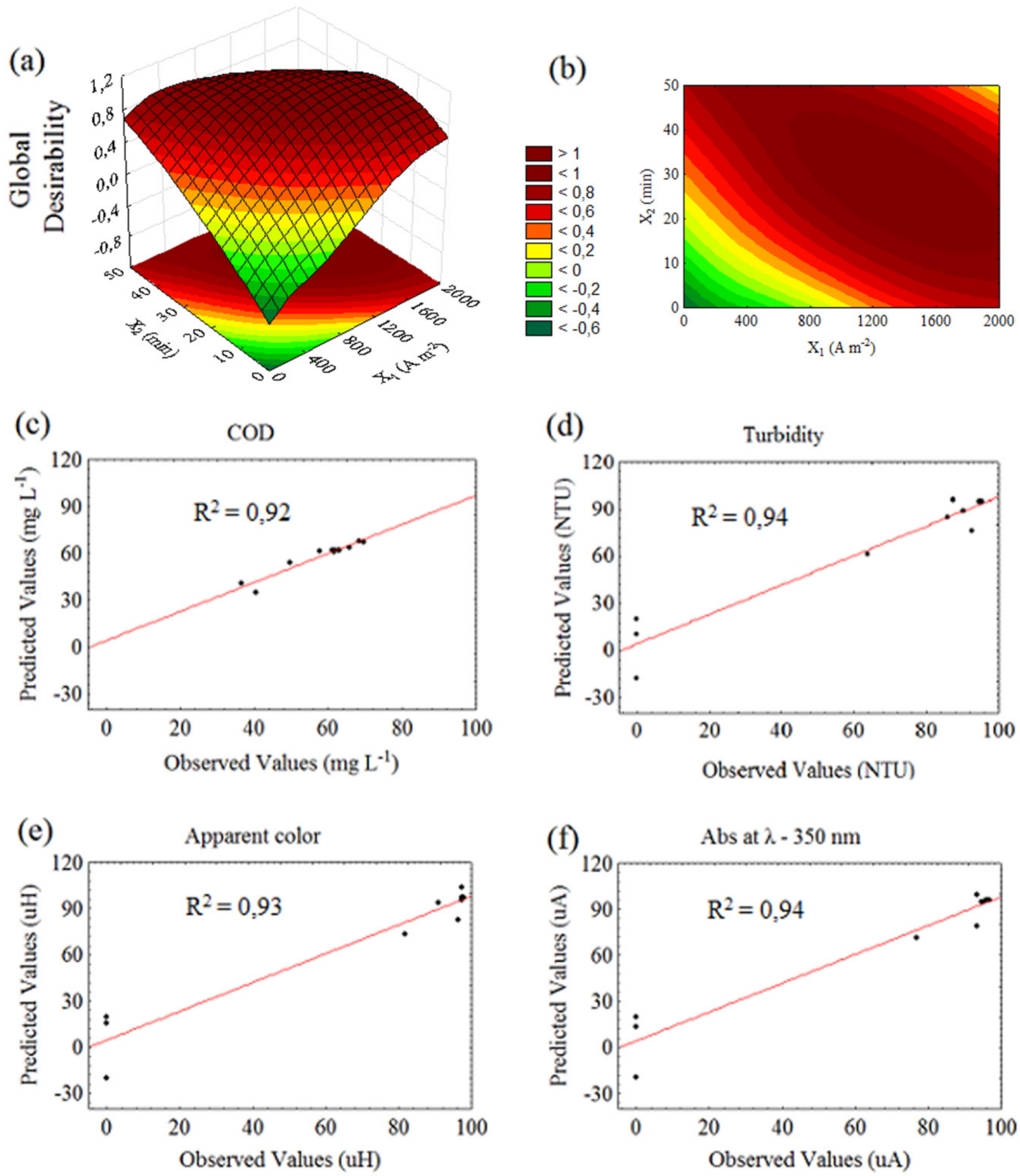

Fig. 3. EC using Al electrode. Surface response (a), contour profile (b), correlation between the values observed and predicted for the response variables: chemical oxygen demand (COD) (c), turbidity (d), apparent color (e), absorbance (Abs) at λ – 350 nm (f).

By means of ANOVA, the percentage of removal of the response variables for both sacrifice electrodes was obtained (Table 4).

It is observed (Table 4) that the *p*-value of the variables X_1 and X_2 were statistically significant at 95% ($p < 0.05$) for the four variable responses (removal of COD, turbidity, apparent color, absorbance at λ – 350 nm). The Fcal > Ftab for the saccharide electrodes of Al and Fe admitted that the proposed model is valid at said level of significance.

Thus, the global desirability method was used to determine the best conditions for adjusting the cosmetic industry wastewater treatment by EC, in order to obtain the optimal operational values of the independent variables (X_1 and X_2). The simultaneous optimization of multiple responses (removal of COD, turbidity, apparent color, absorbance at λ – 350 nm), as shown in Figs. 2(a) and (b). Where the vertical red lines shown indicate the maximum individual desirability for each of the response variables relative to X_1 and X_2 and the horizontal blue lines refer to the efficiency predicted by the model.

The optimized values of the variables X_1 and X_2 corresponded to: 1,672 A m^{-2} and 18.98 min (Al) and 996 A m^{-2} and 29.54 min (Fe), respectively. In these conditions, the coefficient of global desirability (*D*) obtained was 0.97 (Al) and 0.96 (Fe), demonstrating that the process obtained an excellent optimization response, since these indices are very close to the optimal condition (1.0).

From the global desirability, it was possible to obtain the response surface graph as well as the contour profile as a function of X_1 and X_2 for both electrodes (Figs. 3 and 4).

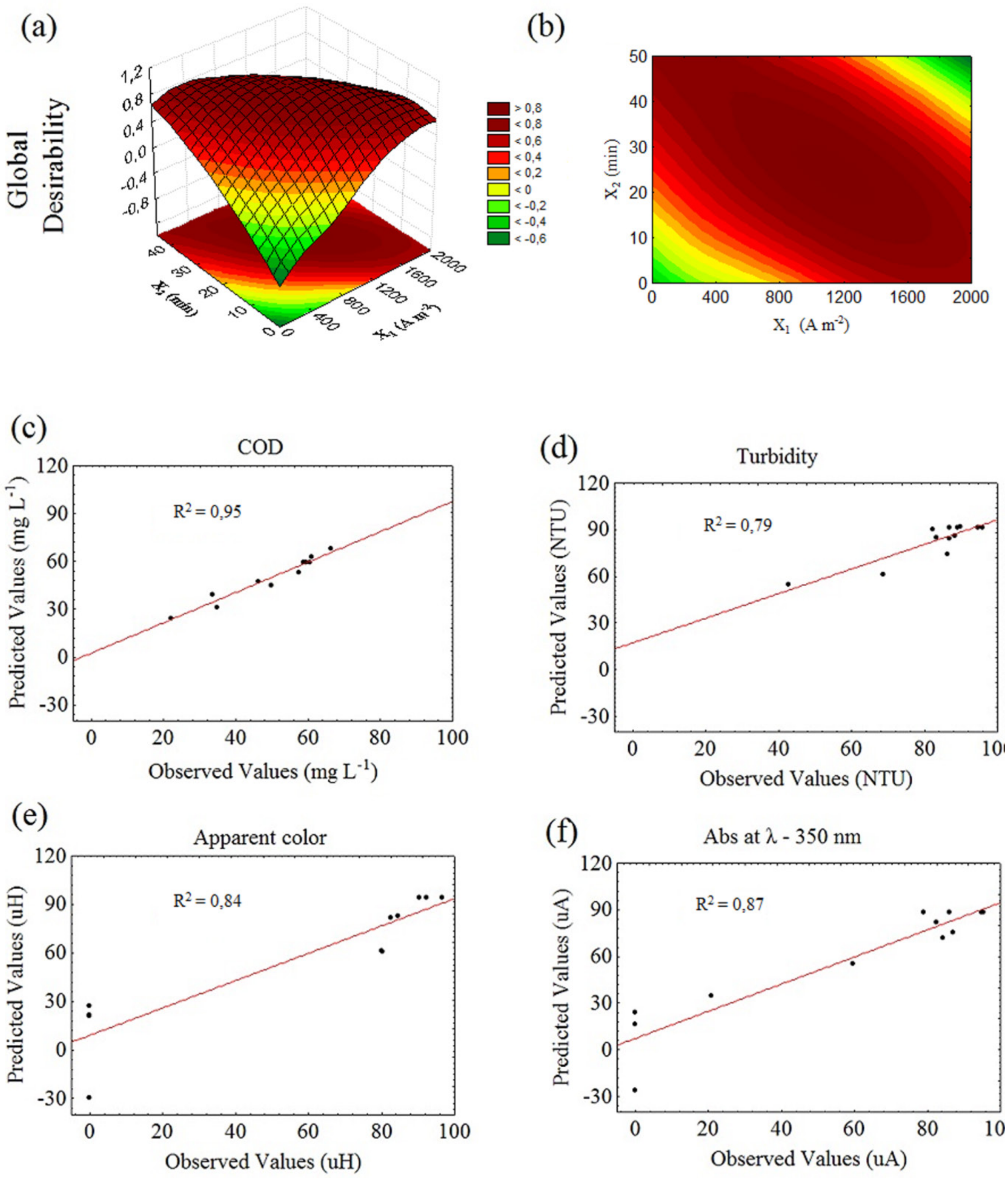

Fig. 4. EC with the use of Fe electrode. Response surface (a), contour profile (b), correlation between the values observed and predicted for the response variables: chemical oxygen demand (COD) (c), turbidity (d), apparent color (e), absorbance (Abs) at λ – 350 nm (f).

Table 5
Validation of the global desirability model

Parameters	Efficiencies (Al)		Efficiencies (Fe)	
	Preview (%)	Real (%)	Preview (%)	Real (%)
COD (mg L^{-1})	65.99	66.12	63.28	61.35
Turbidity (NTU)	99.45	90.96	99.45	92.31
Apparent color (uH)	100.00	97.07	94.14	93.12
Absorbance at λ – 350 nm (uA)	100.00	97.83	98.75	92.69

COD, chemical oxygen demand.

The values obtained by the mathematical model and those observed in the experiments showed little dispersion (Figs. 3 and 4), which proves to be a reliable fit for the model and the experimental data because the dispersion is directly related to a beneficial correlation between the data provided and the experimental data. The coefficient of determination (R^2) provides a variance ratio that is explained by the regression equation in relation to the response variations. In EC, with the use of Al electrode, R^2 was above 0.92 for all response variables. EC with the use of Fe electrode, identified the lowest adjustment in the turbidity analysis. For the other variables, the coefficient of determination was above 0.84.

3.2. Validation of the global desirability model

After obtaining the results of the global desirability function (Fig. 2), a new EC test was performed, considering the optimization values predicted for Al sacrifice electrodes (X_1: 1,672 A m^{-2} and X_2: 18.98 min) and sacrifice electrodes of Fe (X_1: 996 A m^{-2} and X_2: 29.54 min). The results are described in Table 5.

The theoretical results for the removal of COD, turbidity, apparent color and absorbance at λ – 350 nm, considering the Al and Fe sacrifice electrodes in the EC, corroborated with the experimental values, as presented in Table 5.

Considering a significance level of 95% for the experimental results (Table 5), the response variables presented for Test-t, a value equal to 0.13, thus, accepting the null hypothesis (Section 2.4). That is, there is no significant difference between the averages of the results of the cosmetic industry wastewater treated by Al and Fe sacrifice electrodes in EC.

4. Conclusion

The influence of the independent variables X_1 and X_2 on the removal of each of the variables monitored (COD, turbidity, apparent color, absorbance at λ – 350 nm) and their relationships could be identified by means of: ANOVA, global desirability and Test-t.

The RCCD (2^2) experimental design with four axial points and four repetitions at the central point and the ANOVA, ensured with 95% significance that the variable current density (X_1) and operation time (X_2) of EC were responsible for maximum efficiency in removal of physico-chemical parameters of COD, turbidity, apparent color and absorbance at λ – 350 nm. In the analysis of the global desirability and the response surface, the optimal values of the independent variables were identified: X_1 (1,672 A m^{-2}) and X_2 (18.98 min) for the sacrifice electrode of Al and X_1 (996 A m^{-2}) and X_2 (29.54 min) for the Fe sacrifice electrode.

From the optimized conditions mentioned above the treatment of EC reached a removal of COD (66.12% Al and 61.35% Fe), turbidity (90.96% Al and 92.31% Fe), apparent color (97.07% Al and 93.69% Fe) and absorbance at λ – 350 nm (97.83% Al and 92.69% Fe).

Finally, the use of RCCD and the analyses of: ANOVA, global desirability, surface response and Test-t, allowed a better understanding of EC technology applied in the treatment of cosmetic industry wastewater with Al and Fe sacrifice electrodes. The results encourage the applicability of EC technology as a promising alternative for the treatment of cosmetic industry wastewater.

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