Electrocoagulation Applied for Textile Dye Oxidation Using Iron Slag as Electrodes

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Abstract

The Indigo Blue dye is widely used in the textile industry, specifically in jeans dyeing, the effluents of which, rich in organic pollutants with recalcitrant characteristics, end up causing several environmental impacts, requiring efficient treatments. Several pieces of research have been conducted in search of effective treatment methods, among which is electrocoagulation. This treatment consists of an electrochemical process that generates its own coagulant by applying electric current on metallic electrodes, bypassing the use of other chemical products. The objective of this work was to evaluate the potential use of iron slag in the electrocoagulation of a synthetic effluent containing commercial dye Indigo Blue and the effluent from a textile factory. The quantified parameters were color, turbidity, pH, electrical conductivity, sludge generation, phenol removal, chemical oxygen demand (COD), and total organic carbon (TOC). The electrocoagulation treatment presented a good efficiency in removing the analyzed parameters, obtaining average removal in the synthetic effluent of 85 % of color and 100 % of phenol after 25 min of electrolysis. For the effluent from the textile factory, average reductions of 80 % of color, 91 % of turbidity, 100 % of phenol, 55 % of COD, and 73 % of TOC were measured after 60 min of electrolysis. The results obtained demonstrate the potential of using iron slag as an electrode in the electrocoagulation process in order to reuse industrial waste and reduce costs in the treatment and disposal of solid waste.

1. Introduction

The textile industry stands out as one of the most polluting sectors in terms of the volume and complexity of the effluents produced. The dyeing and finishing processes contribute significantly to the generation of wastewater through the use of dyes during the production process, promoting an effluent with specific characteristics, such as intense color, high chemical oxygen demand (COD), a large amount of dissolved solids, and pH variation (Zaroual et al., 2006; Cerqueira et al., 2009; Mook et al., 2017).

Due to the presence of dyes, several environmental impacts may be caused when this effluent is discharged without meeting the regulatory requirements. One of these impacts occurs by partially reducing the penetration of sunlight into the aquatic environment, hindering the photosynthesis process and the production of oxygen by aquatic plants, thus directly affecting the amount of dissolved oxygen present in the environment, causing serious effects on the life cycle of fish (Chakraborty, 2014). Also, high concentrations of nitrogen, phenols, iron, and chromium, which end up harming aquatic life, and high COD and biochemical oxygen demand (BOD) values may result in the reduction of oxygen in the affected ecosystem (Chakraborty, 2014; Buscio et al., 2015; Liu et al., 2019).

Indigo Blue dye is widely used in the coloring of jeans (Abdelileh et al. 2020). The structural and chemical properties of indigo blue make it poorly soluble in water (Volkov et al., 2020; Yin et al., 2020). Thus, the fiber dyeing process occurs through a series of oxidation-reduction processes: immersion in a sodium dithionite bath entails the reduction of the dye, which takes the leuco-indigo form, presenting high solubility in water and affinity with the fiber for dyeing; after immersion, the fiber is exposed to air to

return to the original insoluble form (Albuquerque et al., 2013; Abdelileh et al., 2020; Hendaoui et al., 2021).

There are numerous physicochemical, chemical, and biological techniques for the treatment of these textile effluents. Due to the environmental implications, in addition to conventional treatments, research has emerged using new treatment technologies for the degradation of these compounds, among which electrochemical processes have stood out (Holkar et al., 2016; Bener et al., 2019).

Electrocoagulation is a widely applied technique for pollutant removal widely used in the treatment of textile effluents due to its environmental friendliness, versatility, energy efficiency, safety, and cost-effectiveness. It uses simple equipment of easy operation, which facilitates its maintenance (Khandegar and Soroha, 2013). The electrochemical reactor is composed of an electrolyte and two electrodes (cathode and anode). Oxidation-reduction reactions occur at the electrodes through the application of a continuous electric current, and a coagulant is released into the effluent by the anode through the oxidation of the material. At the cathode, the electrode reduction process occurs, releasing hydroxyl ions to the medium (Gendel and Lahav, 2010). After this combination of reactions, water electrolysis occurs, producing oxygen at the anode and hydrogen at the cathode, forming microbubbles and causing the flocculation of the particles present in the effluent (Mollah et al., 2004; Verma, 2017).

The most commonly used materials in electrochemical treatments are aluminum and iron; however, other materials have been used as electrodes in various studies, such as zinc, stainless steel, galvanized steel, steel, graphite, platinum, and diamond (Lee and Gagnon, 2015; Silva et al., 2016; Zazou et al., 2019), and industrial waste such as scrap metal may also be used. The foundry industry in Brazil in the first half of 2018 was responsible for producing approximately 800,000 tons of castings, of which 600,000 tons represent the production of cast iron, a volume 6 % higher than in 2017 (Carmelio et al., 2018). In the process of casting metal parts, a solid residue called slag is produced. This residue is rich in iron and, because it has no other application, ends up in industrial landfills.

Aiming at the sustainability of the environment and cost reduction for the industry through using the waste generated and reducing landfill disposal, the present work shows the evaluation of the application of iron slag from a foundry as electrodes in an electrochemical process employed as a secondary treatment for color removal in synthetic and real effluents containing textile dye.

2. Materials And Methods

2.1 Electrodes

The iron slag was obtained at a foundry located in Erechim, RS, Brazil. The collected raw material needed to be broken manually for obtaining smaller parts to be used as electrodes (Fig. 1). After breaking, the raw scoria pieces were weighed, and cathode and anode pairs with similar masses were formed. The characterization of the raw iron slag has already been reported in previous work by Bragagnolo et al. (2018), and the main constituents of the material are presented in Table 1.

Table 1

Properties and constituents of the raw iron slag used as electrodes for textile dye oxidation

Constituents			Properties	Reference
Oxides	(%)	Others (%)	Loss on ignition (%)	Bragagnolo et al. (2018)
SiO_2	70.4	2.38	1.32	
AlO_3	13.6	-	-	
K_2O	4.98	-	-	
Fe_2O_3	3.29	-	-	
Na_2O	2.79	-	-	
CaO	1.20	-	-	

2.2 Electrochemical reactor

The reactor used for the treatment was made of glass with the dimensions 15 cm x 30 cm, totalizing a volume of 3 L of effluent per treatment (Fig. 2). The wires used were of a 6 mm gauge, stripped 10 cm at one end to wrap the slag and 5 cm at the other end to connect to the power source, being changed at each experiment. The electric current applied in the treatments was based on the literature, ranging from 0.3 A to 0.9 A (Ghanbari et al., 2014; Matias et al., 2015). For better electron conductivity in the effluent, 1 g L^{-1} of NaCl was added to each treatment. All trials were performed at room temperature of 20 °C to 25 °C (± 1.0 °C), and unadjusted pH from 7.5 to 8.7 (± 1.2) was used.

2.3 Synthetic effluent

The synthetic effluent consisted of commercial Indigo Blue dye, requiring grinding and sieving before use in order to achieve its homogenization. The concentration limits used were based on the literature, ranging from 50 mg L^{-1} to 80 mg L^{-1} (Ghalwa et al., 2016; Hendaoui et al., 2021).

The electrolysis time in the synthetic effluent treatment was set at 25 min after analyzing the color removal kinetics performed (Fig. S1) using the dye concentration of 32.2 mg L^{-1} and removing samples every 10 min of the treatment until the color removal stabilized.

The experiments were performed following a 2^2 factorial experiment design (Table 2), analyzing the variables of electric current (0.3 A to 0.9 A) and dye concentration (50 mg L^{-1} to 80 mg L^{-1}). The combination of these variables using experimental planning resulted in a total of eleven trials, as shown in Table 3.

Table 2

Levels and variables used in the experimental design of the electrocoagulation process for the oxidation of textile dye from synthetic effluent.

Variables	Level				
	α = -1.41	-1	0	+1	α = +1.41
Current (A)	0.3	0.45	0.6	0.75	0.9
Dye Concentration (mg L^{-1})	50	58	65	73	80

Table 3

Matrix of the 2^2 factorial experiment design with coded values for the electrocoagulation process for the oxidation of textile dye from synthetic effluent.

Exp. run No.	Current	Dye concentration
1	-1	+1
2	-1	-1
3	+1	+1
4	+1	-1
5	0	0
6	0	0
7	0	0
8	-1.41	0
9	+1.41	0
10	0	-1.41
11	0	+1.41

2.4 Real textile effluent

The industrial textile effluent was obtained from a Jeans company located in Erechim, RS, Brazil. It was collected in an equalization tank after a sieve system. The experiments using the real effluent totalized fifteen runs, and only the electric current was analyzed as a variable since the initial color concentration was determined in the effluent characterization stage. Thus, each current used in the real effluent (0.3 A to 0.9 A) was analyzed in triplicate to understand the influence of the electric current on the electrochemical

treatment. The electrolysis time was set at 1 h; after analyzing the color removal kinetics using the actual effluent, samples were collected every 10 min until the color removal stabilized (Fig. S2).

2.5 Analytical Methodology

The electric current applied to the treatments was based on the literature, ranging from 0.3 A to 0.9 A (Ghanbari et al., 2014; Matias et al., 2015). For better electron conductivity in the effluent, 1 g L^{-1} of NaCl was added to each treatment. All assays were performed at room temperature (20 °C to 25 °C), and unadjusted pH was used in the range of 7.5 to 8.7. At the end of each run, the initial and final treatment samples were analyzed for Color (method 2120 B), Turbidity (method 2130 B), pH, conductivity, Phenol (method 5530 D), Chemical Oxygen Demand (method 5220 D), and Total Organic Carbon (method 5310 B), with all parameters being evaluated according to the methodology proposed by the Standard Methods for the Examination of Water and Wastewater (APHA, 2005). The pieces of equipment used in this study are available in the electronic supplementary material 1 (Text S1). In addition to these parameters, sludge production and electrode wear were also analyzed using the Gravimetric Method. The statistical analysis was performed using software Minitab 15, and graphs were made using GraphPad Prism 8.

3. Results And Discussion

3.1 Electrocoagulation of the synthetic textile effluent

The results of applying the electrocoagulation process with iron slag electrodes to the synthetic effluent are presented in Table 4, showing the values of the initial and final concentrations for color removal, according to the experimental design. One may observe an efficient color removal in the effluent, varying from 68 % to 95 %, presenting a removal average of 85 %. This variation in color removal may occur due to the composition of the iron slag electrodes because the concentrations of iron and other constituents of the electrodes are not known with accuracy since they are made of solid foundry residues and replaced at each new treatment; therefore, there may be variability in their composition.

Table 4
Color removal from synthetic wastewater containing textile dye by the electrocoagulation process using iron slag electrodes.

Exp. run No.	Initial concentration (mg L^{-1})	Final concentration (mg L^{-1})	Color removal (%)
1	91.4	23.85	73.90
2	76.51	5.08	93.35
3	97.12	10.38	89.30
4	69.77	3.04	95.64
5	69.77	15.49*	77.80
6	66.51	4.06*	93.89
7	67.32	4.67*	93.05
8	71.40	22.83	68.01
9	69.16	4.46	93.53
10	46.30	14.46	68.75
11	105.08	10.18	90.30
* Standard deviation of the center point = 6.4 mg L^{-1}			

In the present study, the color removal efficiency using the proposed treatment was verified. Even with the complex composition of the iron slag electrodes, a high color removal efficiency reaching 95 % was obtained. In work developed by Verma (2017), electrocoagulation was applied to a synthetic effluent containing 200 mg L^{-1} of dye using one iron and one aluminum electrode, resulting in 86 % color removal after 1 h of treatment.

One of the main parameters for the efficiency of the electrocoagulation treatment is the amount of coagulant generated, which is related to the applied electric current density and the electrolysis time (Kabdasli et al., 2010). According to studies by Azarian et al. (2018) and Nariyan et al. (2017), the pollutant removal efficiency of an electrocoagulation treatment is directly proportional to the applied electric current density, explained by Faraday's Law. However, after a statistical analysis of the experimental results obtained in this study, it was observed that the two variables analyzed (i.e., electric current intensity and dye concentration) did not show significant effects on the efficiency of color removal from the synthetic effluent. This statement may be confirmed through the Pareto chart with 95 % confidence (Fig. 3).

The pH and electrical conductivity values obtained in the eleven tests performed are shown in Fig. 4a. One may observe that the pH values after the electrocoagulation treatment are above the value allowed for discharging liquid effluents into water bodies (pH between 5 and 9) according to Resolution No. 430 of the Brazilian National Environment Council (CONAMA) (Conama, 2011).

In electrochemical processes, the pH increases during the treatment due to the generation of OH^- ions during the water reduction step (Cerqueira et al., 2009). In electrocoagulation, the final pH is high compared to the initial pH, corroborating the values obtained in this study. Cerqueira et al. (2009) evaluated the initial pH as an influencing parameter in electrocoagulation using iron electrodes and observed that the initial pH of 7 led to better values of color and turbidity removal and COD. Ramazan et al. (2019) studied the effect of pH applying different electrolytes using iron electrodes, reporting that, by adding NaCl to the solution to be treated, the pH increased from 7.2 to 11.35.

Brazilian regulations do not establish guideline values of electrical conductivity (Fig. 4b) for discarding effluents into water bodies. Bande et al. (2008) observed that introducing a higher concentration of NaCl into the effluent to be treated results in an increase in conductivity and a decrease in the voltage offered by the effluent, thus reducing the amount of energy required to perform the electrocoagulation process. However, Wang et al. (2009) showed that a large increase in the NaCl concentration should not occur as it affects the COD reduction efficiency, with an excess of Cl^- ions in the medium being unfavorable to the coagulation of pollutants.

The wear of the iron slag electrodes (Fig. 5) was determined by weighing them before and after the treatment. For there not to occur mass loss with the washing of the electrodes, the use of abrasive materials was avoided and, after each test, the electrodes only received distilled water to remove the generated sludge that could have adhered to the iron slag for subsequent weighing and disposal.

In the study by Zazouli and Taghavi (2012), the wear of the electrodes was remarkable at higher current densities. In this study using iron slag, the current density showed no effect on the electrode wear; however, it should be taken into account that the maximum limit of the current applied in this study was 9 A, unlike the maximum current of 25 A applied by Zazouli and Taghavi (2012).

The sludge production in the electrocoagulation process using iron slag electrodes in a synthetic effluent is represented in Fig. 6. One may observe the increase in sludge generated as the intensity of the electric current is raised; this behavior was expected since there is a direct relationship between the amount of sludge formed and the current applied in the electrochemical process. Studies by Zodi et al. (2009) and Chen (2004) confirm the increase observed in this study. The authors reported that, when using iron and aluminum electrodes in electrocoagulation, they obtained results where the sludge formation is directly related to the density of the current applied. The amount of sludge formed is also related to the volume of coagulant material produced with the removal of total suspended solids and other compounds present in the effluent.

A small phenol presence was observed with an initial concentration ranging from 0.3 mg L^{-1} to 9.1 mg L^{-1}; these concentrations were 100 % removed. Despite the complexity of the composition of the iron slag used, the removal was very efficient for this type of pollutant. The study conducted by Silva et al. (2016) corroborates the results obtained in this study: the authors evaluated the removal of contaminants

in a textile effluent through electrochemical degradation, obtaining approximately 100 % of phenol removal.

The TOC concentration was also evaluated initially and at the end of the trials. In the treatment, there occurred a TOC removal of approximately 18 %. The other assays did not present TOC removal. The initial TOC concentrations found ranged from 6 mg L^{-1} to 14 mg L^{-1}. For evaluating this parameter, it is necessary to consider the chromophore group breakdown; such chemical groups are responsible for the coloration of the dye, being the structure responsible for its fixation to the textile fiber (Paschoal and Temiliosi-Filho, 2005). The results obtained in the TOC analyses suggest that the electrocoagulation process may only be breaking this chromophore group bond and, therefore, only removing the color and not the organic matter present in the effluent considering the low TOC removal obtained.

3.2 Electrocoagulation in a real industrial textile effluent

The effluent collected from the textile industry was characterized in order to know its properties (Table 5).

Table 5
Characterization of the raw textile effluent collected in an equalization tank after a screening system.

Characterization of the raw effluent	
Color	887.7 Pt Co L^{-1}
Turbidity	1042.60 NTU
pH	8.06
Conductivity	673.7 µS cm^{-1}
Temperature	22 ºC
COD	664.0 mg O_2 L^{-1}
BOD_5	1076.2 mg O_2 L^{-1}
Total Solids	68.40 mg L^{-1}
Phenol	1.17 mg L^{-1}
TOC	208.20 mg L^{-1}

Through the application of the electrocoagulation process using iron slag electrodes in the industrial effluent, it was possible to observe the reduction of color, turbidity, TOC, and COD under each current applied. Figure 7 presents the mean values of the removals in triplicate.

The best results obtained for the removal of color and turbidity were observed with the smallest electric current applied (0.3 A): 80 % of color and 91 % of turbidity were removed. These values show that the

applied electric current did not present significance in the electrocoagulation treatment under the evaluated conditions, with it being possible to use low electric currents and obtain high treatment efficiencies, saving costs with applied electric energy.

According to the CONAMA resolution No. 430, which provides for the liquid effluent discharge standards (Conama, 2011), the color parameter has no concentration limit, with the only condition being that it should not change the color of the water body. Thus, CONAMA resolution No. 357, which provides for the classification of water bodies and effluent discharge standards (Conama 2005), was used as a reference for discharge. For Brazilian Class I rivers, the natural true color of the water body is required, while for Class II the true color has a limit of 75 mg Pt L^{-1}. The final concentration values after the electrocoagulation treatment are all below the limit allowed for Class II rivers, which would allow the release of the treated effluent into the water body without the need for secondary treatment.

Núñez et al. (2019) evaluated the application of electrocoagulation for removing color and turbidity from textile factory wastewater using iron electrodes. The authors used 200 mL of the industrial effluent and an electrolysis time of 10 min, finding removal efficiencies of 70 % for color and 45 % for turbidity, values lower than for the treatment applying iron slag as electrodes in this study.

In the evaluation of the reduction in TOC, the values were close for each current applied, not demonstrating a significant variation, finding with the current of 0.9 A an efficiency of 73 %. For the COD, a maximum reduction of 55 % was obtained with the application of a current of 0.6 A; the other current intensities led to average reductions of 30 %. Phenol removal was also evaluated, and the treatment presented an efficiency of 100 %, with total removal occurring in all the tests.

Sirma et al. (2019) evaluated TOC and COD reduction in a textile effluent by the electrocoagulation process using 800 mL of the effluent and aluminum and iron electrodes. The authors reported better removals of TOC for the aluminum electrode (29 %), while the iron electrode only achieved a 5 % removal. As for the COD reduction, the authors found a low efficiency, with a maximum reduction of 19 %. These values show that iron slag is efficient in reducing TOC and COD, presenting reductions in the order of 73 % and 55 %, respectively.

The sludge production in the electrocoagulation process using iron slag electrodes in industrial effluents showed values directly related to the increase in electric current (Fig. 8).

A large standard deviation of the sludge production is noticeable. This is attributed to the intrinsic characteristics of iron slag since it is a solid waste of varied composition, making it difficult to predict sludge production. However, even with the high deviation observed, the amount of sludge generated is reduced, which would justify its application in the process.

When applying a high current density in the treatment, an increase in the anodic metal dissolution is observed and, consequently, an increase in the amount of sludge produced (Zodi et al., 2009). This reaction of increased sludge production is evident in the tests performed using iron slag: the higher the

electric current density, the greater the amount of sludge produced. When studying the electrocoagulation process for the decolorization of textile wastewater, Bener et al. (2019) showed that the wear of the electrodes is directly related to the applied electric current density. However, when using iron slag, the observed behavior was not similar: the most considerable wear presented was at the intermediate current of 0.6 A (Fig. 9), possibly due to the characteristics of the slag itself.

Conclusions

The electrocoagulation process using iron slag as electrodes showed good efficiency in treating a synthetic effluent and an industrial effluent containing textile dye. This process presents a great advantage related to cost reduction due to the non-addition of inputs since it is a residue produced in the foundry industry, besides being environmentally and financially interesting given that its application in effluent treatment would also reduce its disposal in industrial landfills, thus contributing to the foundry industry by reducing the cost of the disposal of this material.

Some points still have to be investigated in order to reach the release standards, such as the pH that was above the allowed by the Brazilian regulations in both analyzed effluents and the TOC removal, which did not show significant results in the evaluated tests with the synthetic effluent, requiring greater attention to the chromophore group. From the statistical analysis of the data obtained, it is observed that none of the variables evaluated (i.e., electric current and dye concentration) presented statistical significance in the treatment ($p < 0.05$).

The use of iron slag as an electrode was limited because it presents uncertainties in the concentration of iron in each fraction used in the treatment, which leads to varying results for each test performed. However, electrocoagulation is a method that has many strengths, thus requiring a better evaluation of the use of iron slag in the process for understanding its behavior in the electrochemical treatment.

Declarations

Ethics approval and consent to participate:

Not applicable

Consent for publication:

Not applicable

Availability of data and materials:

All data generated or analysed during this study are included in this published article [and its supplementary information files].

Competing interests:

The authors declare that they have no competing interests

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Authors' contributions:

All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Rafaela De Maman, Gean Delise Leal Pasquali, Vilson Conrado da Luz, Laura Behling, Clarissa Dalla Rosa and Adriana Dervanoski. The first draft of the manuscript was written by Rafaela De Maman, Laura Behling, Vilson Conrado da Luz and Gean Delise Leal Pasquali and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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Figures

Figure 1

Iron slag used as electrodes for the electrocoagulation system of textile dye and industrial effluent

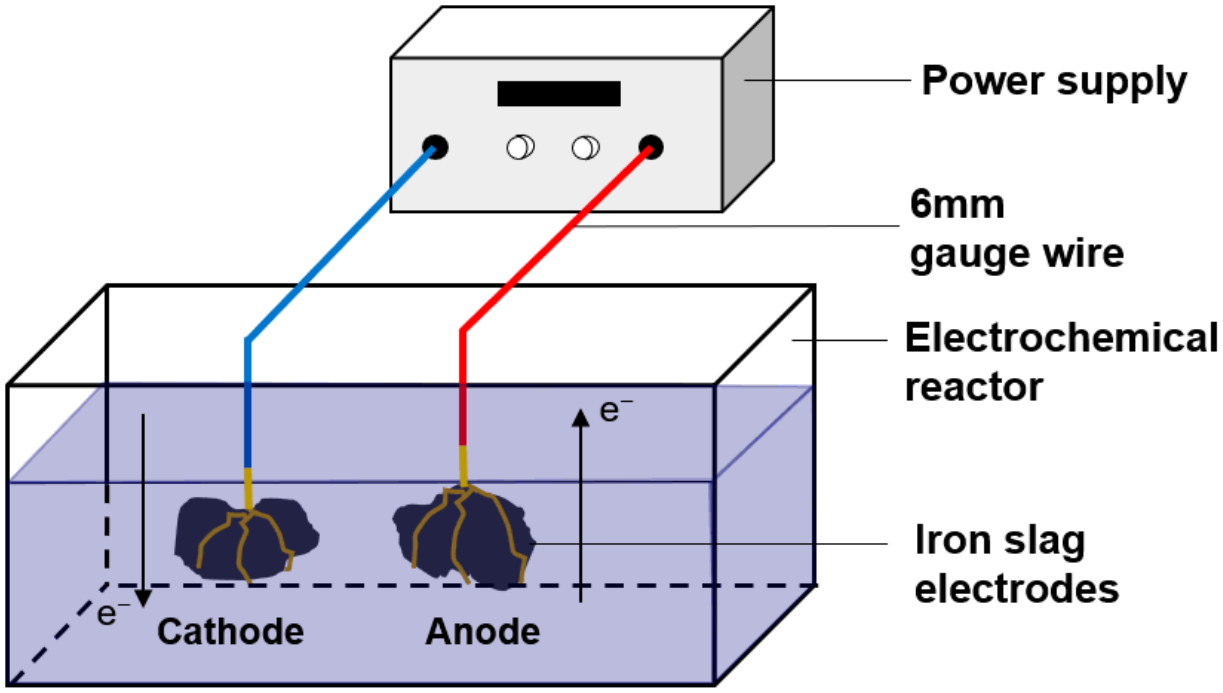

Figure 2

Schematic representation of the electrocoagulation system using iron slag electrodes applied for the oxidation of textile dye in synthetic and industrial effluents

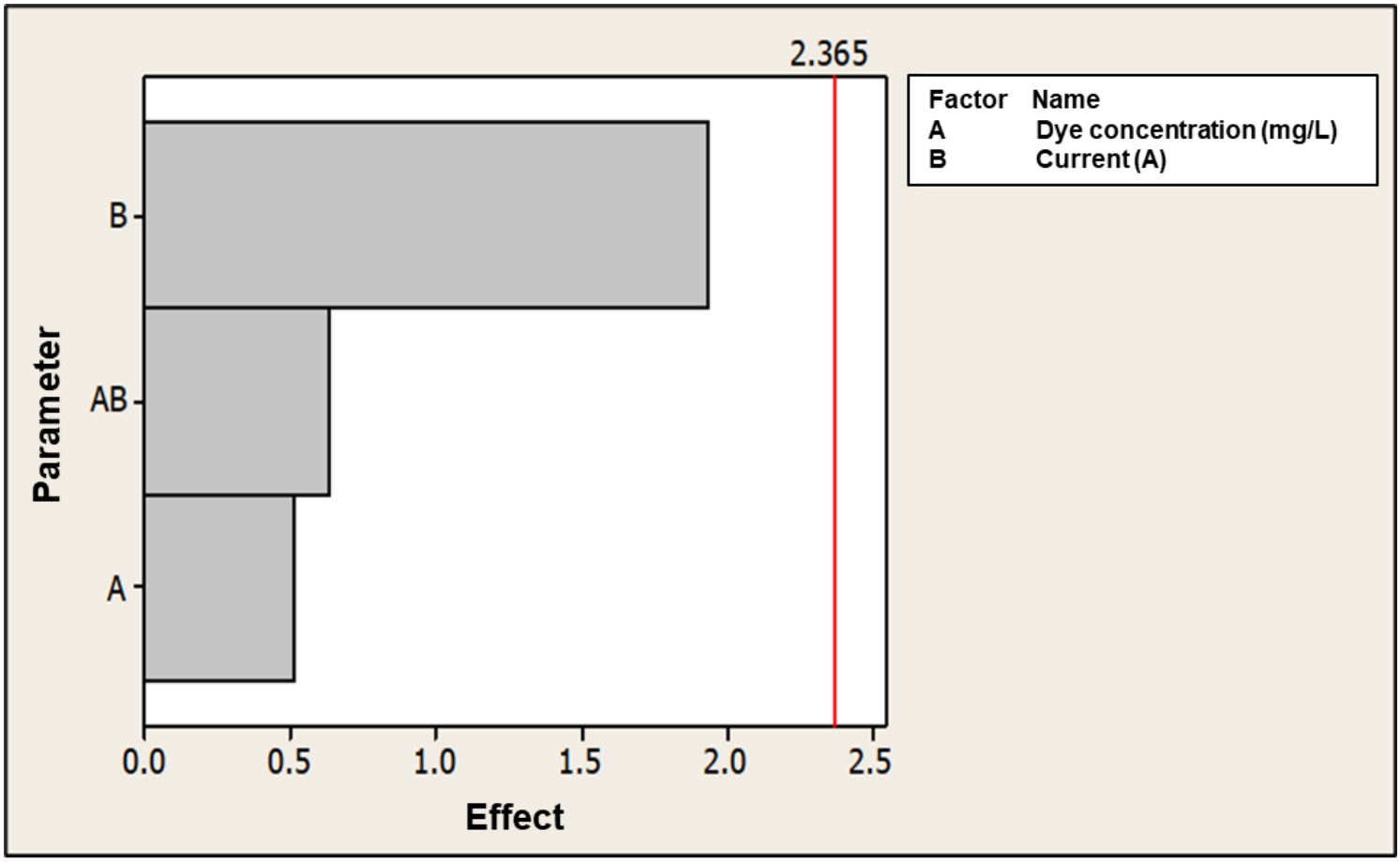

Figure 3

Pareto chart for the analysis of the experimental data obtained for the removal of color from the synthetic textile effluent by the electrocoagulation process using iron slag electrodes with 95 % confidence.

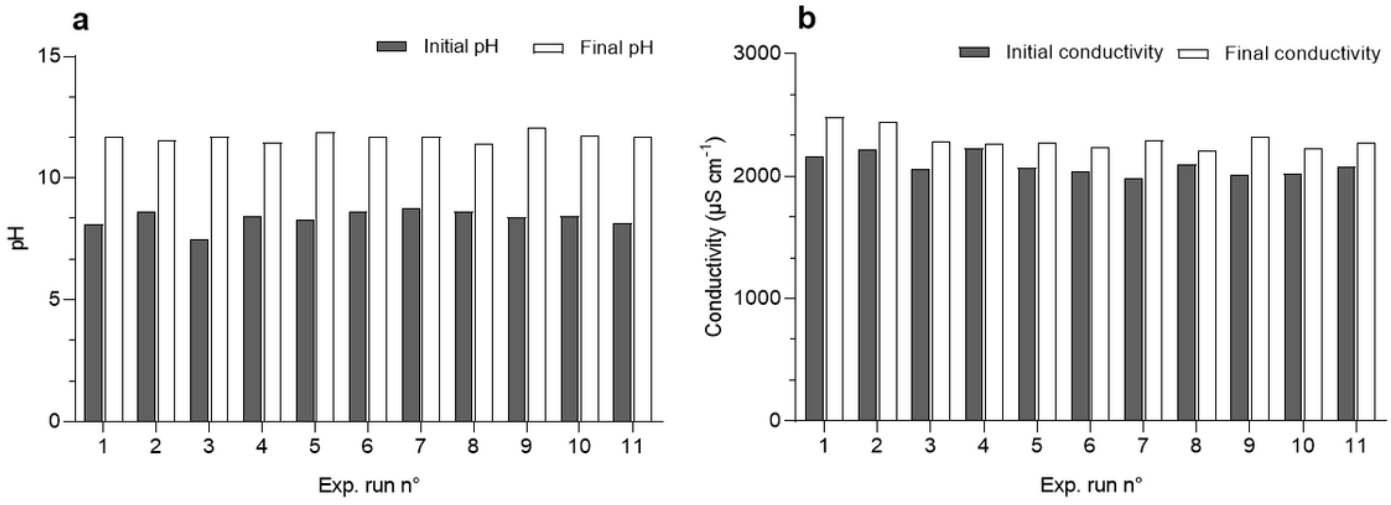

Figure 4

Initial and final pH (a) and electrical conductivity (b) during the electrocoagulation process for the oxidation of textile dye in a synthetic effluent using iron slag electrodes.

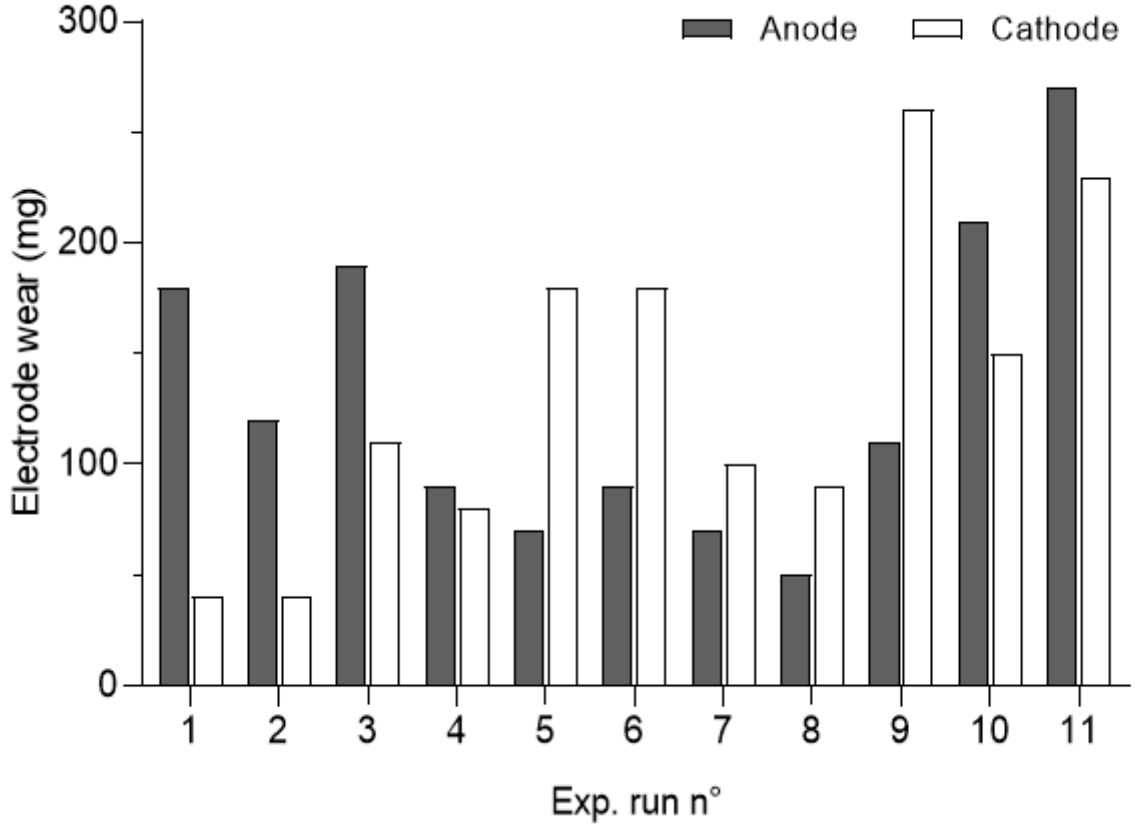

Figure 5

Wear of iron slag electrodes in the electrocoagulation process for the oxidation of textile dye in a synthetic effluent.

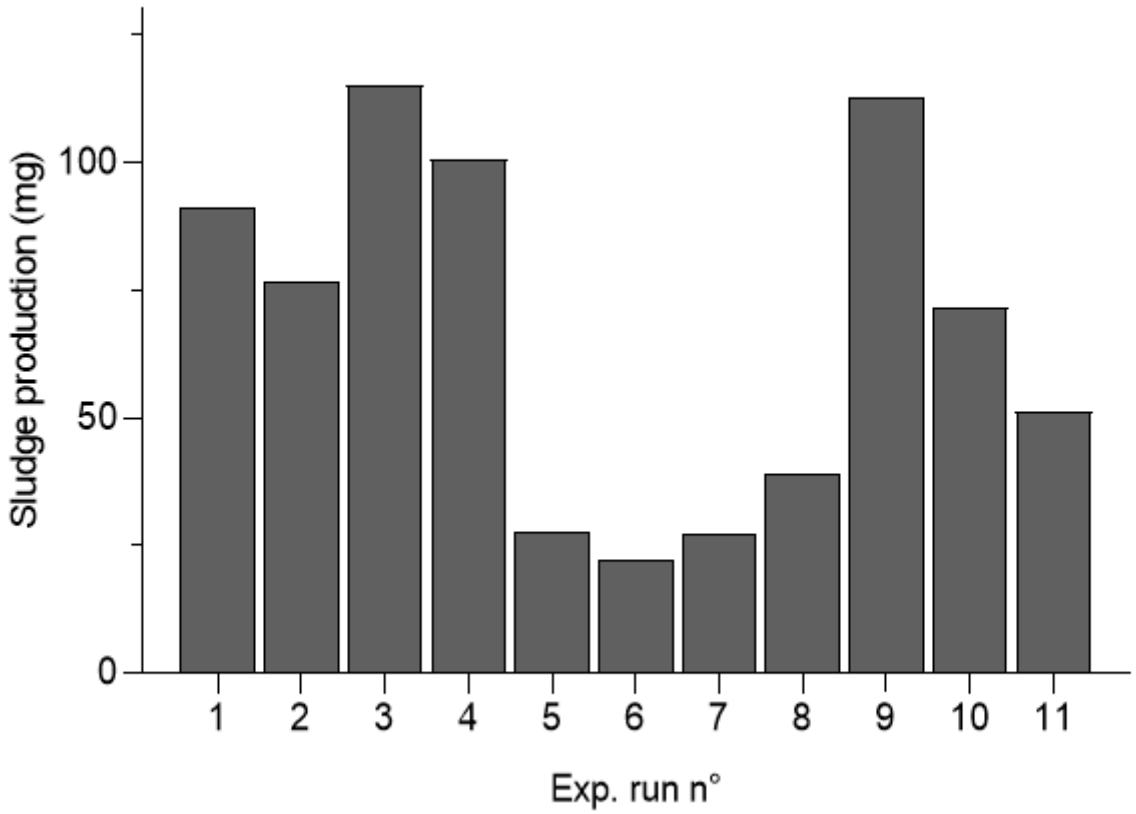

Figure 6

Sludge production by the electrocoagulation process for the oxidation of textile dye in a synthetic effluent using iron slag electrodes.

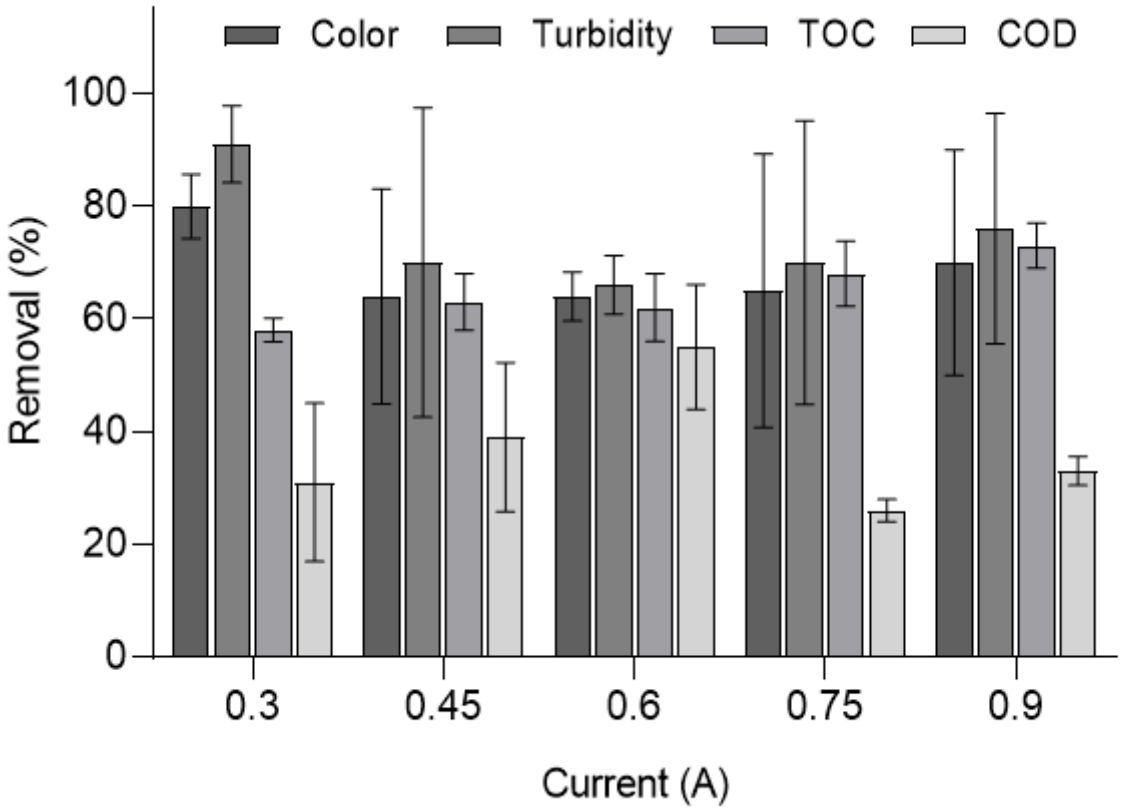

Figure 7

Removal color, turbidity, TOC, and COD by the electrocoagulation process of an industrial textile dye using iron slag electrodes.

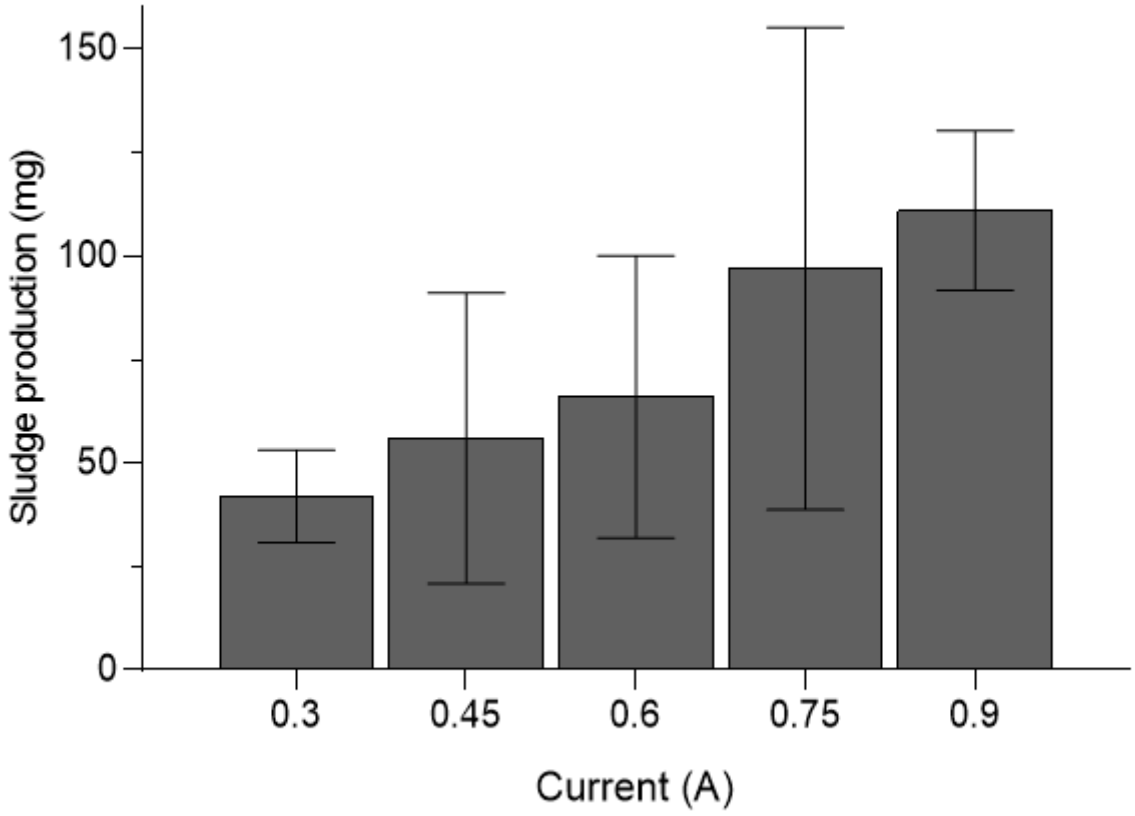

Figure 8

Sludge production by the electrocoagulation process for the oxidation of textile dye in a real effluent using iron slag electrodes.

Supplementary Files

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- ElectronicSupplementaryMaterialEscoria.docx