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Oxidation of Textile Dye Through Electrocoagulation Process Using Scrap Iron Electrodes

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Abstract The textile industry stands out as one of the largest consumers of water among the industrial sectors. Additionally, its effluent presents characteristics such as high load of chemical oxygen demand (COD), total organic carbon (TOC), suspended solids, color, turbidity, phenol, and salts, which require an efficient treatment of the wastewater produced. Among the several researches that have arisen focused on the treatment of textile effluents, electrocoagulation stands out. This method consists of an electrochemical process that generates its own coagulant by applying electric current to metal electrodes immersed in the solution. The electrodes used in the present study are metallic plates made of scrap iron. The objective of this work is to evaluate their application in an electrocoagulation process for the decolorization of real and synthetic effluents. The efficiency of the treatment was evaluated by applying it to a synthetic effluent containing commercial indigo blue dye and to a real effluent from the textile industry, assessing parameters such as color, turbidity, pH, electrical conductivity, COD, TOC, phenol, soluble iron, sludge generation, and electrode wear. The synthetic effluent obtained average color removal of 95%, 96% phenol, and low sludge production in 120 min of electrolysis. In the real effluent from the textile industry, an average color removal of 92%, 97% turbidity, 100% phenol, 65% TOC, and 49% COD in 90 min of electrolysis was obtained. The electrocoagulation process using scrap iron as electrodes proved to be efficient in removing the dye present in the real textile industry effluent, as well as in the synthetic effluent.

Keywords Dyes · Electrochemical · Foundry industry · Scrap iron · Textile industry · Wastewater

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1 Introduction

The textile industry adds high amounts of chemicals to the production process, which makes wastewater treatment difficult. The most frequently found chemicals in textile wastewater are dyes, surfactants, solvents, and other organic compounds (Akhtar et al., 2020; Torres et al., 2019). These pollutants mainly come from the dyeing and finishing processes, attributing to the effluent a high chemical oxygen demand (COD) (1100–4600 mg L^{-1}), suspended solids

concentration (72–956 mg L^{-1}), salt content, color content (1450–4750 ADMI), BOD (10–1800 mg L^{-1}), turbidity (1665–2484 NTU), total organic carbon (TOC) (54–530 mg L^{-1}), and low biodegradability (Savin & Butnaru, 2008; Syafalni et al., 2012; Carmen & Daniel, 2012; Albuquerque et al., 2013; Kobya et al., 2016; GilPavas et al., 2017; Yaseen & Scholz, 2019; Sirirerkratana et al., 2019). Electrical conductivity (1000–10,245 μS cm^{-1}), phenol (14.6–35.8 mg L^{-1}), pH (5–10), and iron (< 10 mg L^{-1}) values are also found (Ghaly et al., 2014; Patel and Vashi, 2015; Yaseen & Scholz, 2019).

Indigo blue is a dye with the chemical formula $C_{16}H_{10}O_2N_2$. It was formerly extracted from the leaves of the true indigo bush (*Indigofera tinctoria*) and is now produced synthetically and used mainly for dyeing jeans. Indigo belongs to the vat dye class: the presence of the ketonic group makes it insoluble in water, but it is reduced in the presence of sodium dithionite in an alkaline solution, making it soluble in water in the form of leuco-indigo and having high affinity with the fiber, thus being able to start the dyeing process. Once in the fibers, the leuco-indigo is oxidized back to its insoluble form (Albuquerque et al., 2013; Hendaoui et al., 2021).

Many methods are used for treating textile effluents, such as adsorption, precipitation, membrane filtration, chemical degradation, and chemical coagulation. However, these processes have several operational problems and high cost for system installation and operation, with additional expenses for inputs and other issues (Singh & Arora, 2011; Verma, 2017). As a result, studies are under developed using the electrocoagulation method, which has been prominent for the treatment of textile wastewater (Akhtar et al., 2020; Brillas & Martínez-Huitle, 2015; Torres et al., 2019).

Electrocoagulation presents a wide use in the removal of various pollutants due to its versatility, safety, ease of application and maintenance, ease of control, low sludge production, and environmental compatibility (Núñez et al., 2019; Thakur et al., 2009). The electrocoagulation process involves generating coagulants in situ by applying electric current to metal electrodes. The metal ions generated by electrochemical dissolution of a consumable anode spontaneously undergo hydrolysis in water, depending on pH, forming several coagulant species, including hydroxide precipitates, which are capable of removing pollutants by adsorption/sedimentation, and other metal ion species. In addition, the simultaneous cathodic reaction allows the removal of pollutants by deposition or by flotation. The most commonly used metals for electrodes are iron and aluminum, due to their wide availability and low cost (Emamjomeh & Sivakumar, 2009; Ya et al., 2018).

In the first 8 months of 2019, the foundry industry in Brazil accumulated a production of 1.57 million tons of castings, 2.6% higher than in the same period of 2018 (Carmelio, 2018). Cast iron is the leader in production in the country (1.26 million tons), followed by steel (117,865 tons) and non-ferrous metals (130,227 tons). In 2018, Brazil ranked tenth in the world for castings production, in a ranking led by China, India, and the USA. Together, these three countries account for 70% of global production, totaling 71.13 million tons (Carmelio, 2018). Due to the high availability of waste coming from the foundry industry, the present work sought to approach the application and evaluation of the use of a by-product of the cutting of metallic pieces, which would be used in the foundry of new iron pieces. Scrap iron from the local foundry industry was used for the electrodes in the electrochemical process for removal of color pigments from synthetic and textile industry effluents.

In this work, an electrocoagulation system using scrap iron electrodes was developed and evaluated for implementation as a secondary treatment for dye removal from synthetic and real effluent containing textile dyes.

2 Materials and Methods

2.1 Electrodes

Scrap iron was collected in a foundry industry located in the city of Erechim-RS, Brazil. This is the same material used in the manufacture of parts produced by the company. The material needed adjustments to gain the shape of plates with smaller dimensions for its use as electrodes (Fig. 1). The plates were cut into pieces measuring 12 × 9 cm. After cutting, they were weighed and stored in pairs for later use.

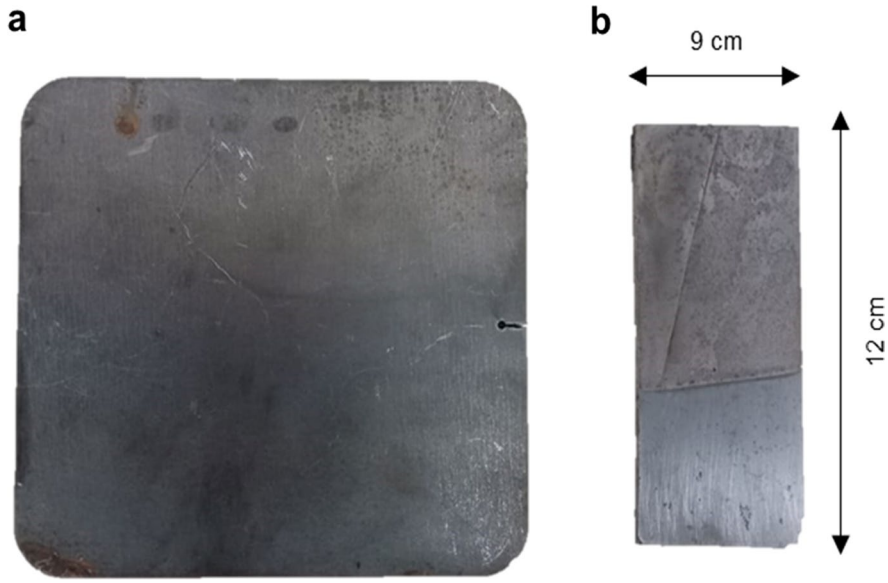

Fig. 1 Collected (**a**) and cut scrap iron (**b**) for use as electrode in the electrocoagulation process for textile dye oxidation

2.2 Electrochemical Reactor

The electrochemical reactor used in the treatments was made of glass, measuring 15 × 30 cm, totaling a volume of 3 L of effluent per run (Fig. 2). The cables used were 6 mm gauge wire, soldered in alligator claws on one end to hold the scrap iron plates and with 5 cm stripped on the other end, to connect them to the power source. The electric current density applied to the treatments was ranging from 300 to 900 mA, and the distance between the plates was fixed at 10 cm. For better electron conductivity in the effluent, 1 g L^{-1} of NaCl was added to each run. All tests were performed at room temperature (20–25 °C), using the natural, pH in the range of 7.5–8.7.

2.3 Synthetic Effluent

The synthetic effluent containing the commercial dye Indigo Blue was prepared in different concentrations, ranging from 50 to 80 mg L^{-1}, based on values reported in the literature (Abu Ghalwa et al., 2016; Hendaoui et al., 2021). Once the limits of dye concentrations were defined, a color scan was performed in a spectrophotometer to identify the dye absorption peak, which was defined at 580 nm. After that,

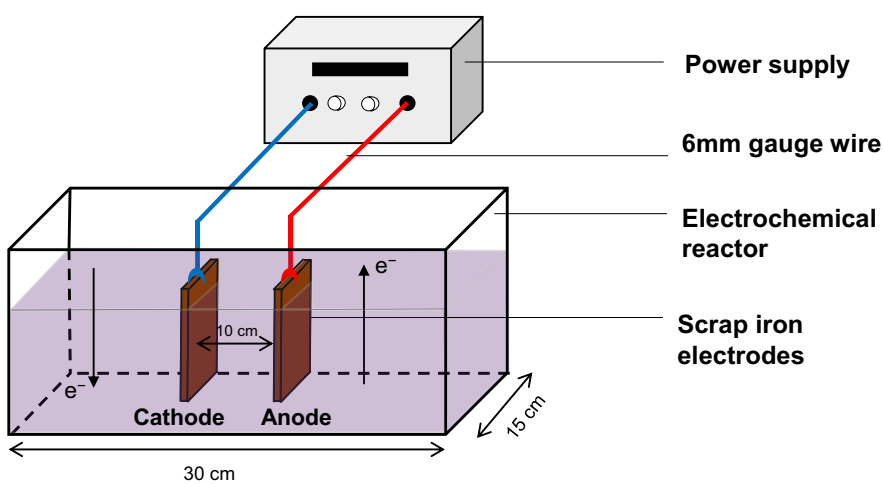

Fig. 2 Representative diagram of the electrocoagulation system

Table 1 Levels and variables used in the experimental design of the electrocoagulation process for oxidation of textile dye from synthetic effluent

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

Table 2 Matrix of the experimental design 2^2 with coded values for electrocoagulation process for 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

a standard color curve was built to determine the dye concentrations in the synthetic effluent samples.

The electrolysis time was set at 2 h, after preliminary kinetic tests of the electrochemical treatment were performed, using 50 mg L^{-1} of Indigo Blue dye, collecting an effluent sample every 10 min of treatment and monitoring the color removal until its stabilization. The experiments were performed following a 2^2 factorial experimental design (Table 1), establishing the variables as current density (0.3–0.9 A) and Indigo Blue dye concentration (50–80 mg L^{-1}), totaling 11 experimental runs, as shown in Table 2.

2.4 Real Textile Effluent

The real textile effluent was provided by a jeans industry located in the city of Erechim-RS, Brazil. The effluent was collected in an equalization tank after a sieve system. The samples were stored in 2 L bottles, totaling 50 L, and then frozen to preserve its characteristics until the time of use.

Because color concentration was already determined in the characterization of the textile industry effluent, the variable studied to observe its influence on the treatment was only the density of the electric current. Triplicates of each assay were carried out with different currents applied to the textile effluent (0.3, 0.45, 0.60, 0.75, and 0.90 A), totaling 15 assays. Electrolysis time was set at 90 min. After analyzing the color removal kinetics using the real effluent, samples were collected at every 10 min until the stabilization of color removal. Details are shown in the supplementary material (Fig. S1).

2.5 Analytical Methodology

Initial and final samples were analyzed for each test performed, assessing the following response variables: color (Method 2120 B), turbidity (Method 2130 B), pH, conductivity, phenol concentration (Method 5530 D), chemical oxygen demand (Method 5220 D), total organic carbon (Method 5310 B), and iron (Method 3500 C). All parameters were assessed according to methodologies proposed in the Standard Methods for the Examination of Water and Wastewater (APHA et al., 2005). All the equipment used is shown in the supplementary material (Text S1).

Additionally, sludge production and electrode wear were also analyzed, both using the gravimetric method. From the results obtained, a statistical analysis was performed with Minitab 15 software, to determine the factors that significantly ($p < 0.05$) influence the electrocoagulation treatment process, considering the response variables under study. Graphs were made in GraphPad Prism 8.

3 Results and Discussion

3.1 Electrocoagulation in Synthetic Textile Effluent

After the electrocoagulation assays, the initial and final concentrations and the percentage of color removal were calculated according to the experimental design, as shown in Table 3. In the data presented, an efficient removal of color from the effluent is observed, with all values above 80%.

Table 3 Color removal by electrocoagulation of textile dye in synthetic effluent using a scrap iron electrode

Exp. run no	Initial concentration (mg L^{-1})	Final concentration (mg L^{-1})	Color removal (%)
1	109.37	10.39	90.50
2	65.29	11.61	82.21
3	108.76	4.27	96.08
4	65.08	0.59	99.09
5*	84.47	2.02	97.61
6*	81.82	2.02	97.53
7*	82.22	3.24	96.05
8	84.27	3.04	96.39
9	80.80	3.04	96.24
10	61.41	1.82	97.04
11	123.86	2.43	98.04

*$SD = 0.70$ mg L^{-1}

The statistical analysis of the data showed that the electric current density and dye concentration (parameters applied in the 11 experimental runs) did not present statistical significance in the dye oxidation present in the synthetic effluent, as evidenced by the Pareto chart with 95% confidence (Fig. 3). However, Neto et al. highlights the importance of the electrical density applied to the electrochemical treatment, justifying that the higher the electrical current applied, the greater is the formation of coagulant species, which accelerate the process of colloidal particle formation and consequently increase treatment efficiency (Neto et al., 2011). According to Saqer and Farhat (Abu Ghalwa et al., 2016), the current density determines the coagulant production rate and the size of bubble production, and therefore affects their growth. After increasing the current density, the amount of hydroxides for precipitation and adsorption will also increase.

The pH and electrical conductivity values of the 11 experimental runs are represented in Fig. 4, showing that the initial and final values follow the same range, with the presented pH values starting at an average of 8 and ending at 12. These pH values obtained (Fig. 4a) were above the 5–9 pH range allowed for effluent discharge according to Resolution No. 430 of the Brazilian regulatory body National Council of the Environment (Conama, 2005). Results obtained by Akhtar et al. (2020) using iron electrodes, with pH 3.0, had the highest oxidation efficiency for Congo Red dye (1000 mg L^{-1}), with 89% of TOC removal efficiency and 97% of color removal. Akhtar et al. also reported a significant drop in efficiency when the experiments were investigated at alkaline pH ($pH = 8$, 10, and 12) (Akhtar et al., 2020). Daneshvar et al. (2004) studied the application of electrocoagulation in treating a solution of the acid dye Red 14

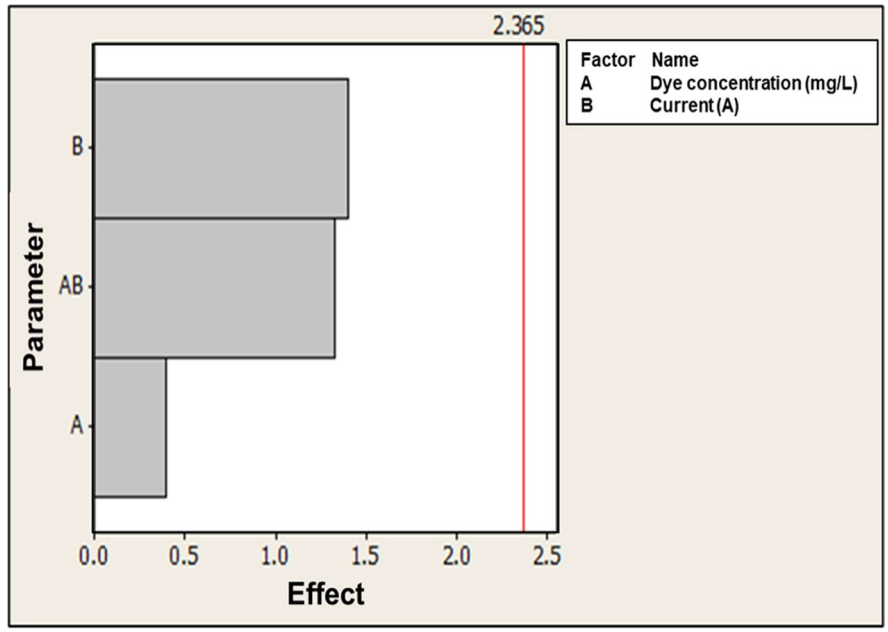

Fig. 3 Pareto chart of the analysis of experimental data obtained for color removal as a function of the current density and dye concentration variables, with 95% confidence

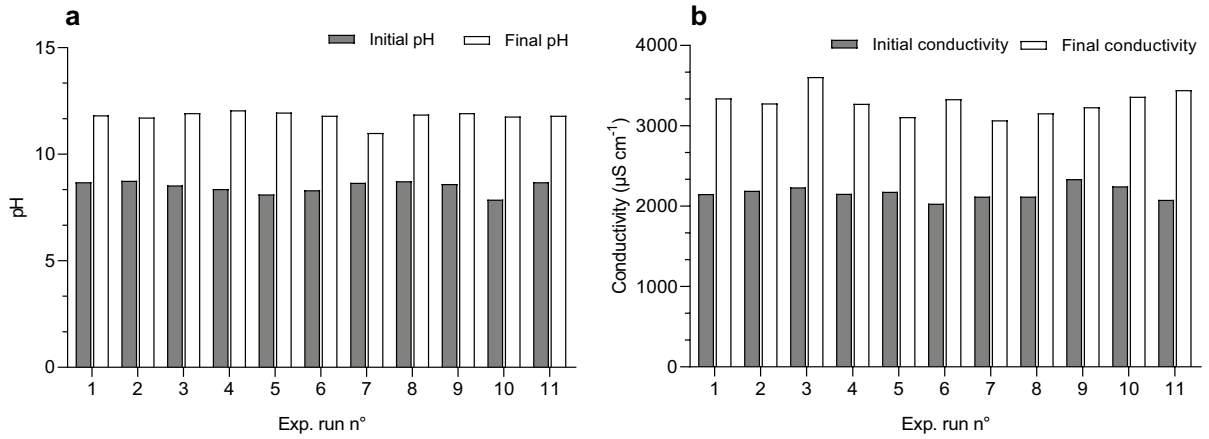

Fig. 4 Initial and final values for pH (**a**) and electrical conductivity (**b**) in the oxidation process of textile dye in synthetic effluent through electrocoagulation

using iron electrodes and observed that the maximum color removal efficiency was at a pH in the 6–9 range. Zazou et al. (2019) studied the pollutant removal efficiency using two pH ranges, an acidic range of pH 3 and another of pH 8.75, as the initial pH of the textile effluent to be treated. Zazou et al. (2019) reported that decreasing the pH from 8.75 to 3 resulted in a decrease in color removal, approximately from 93 to 65%, indicating less efficient removal at acidic pH. This was attributed to the amphoteric character of iron hydroxide, which hardly precipitates at very low pH.

The values of electrical conductivity (Fig. 4b) presented the same initial and final ranges after treatment. This parameter does not present a limit in the discharge standards required by the Brazilian resolutions in force.

The use of scrap iron as an electrode for the removal of phenols and TOC (Fig. 5) was also evaluated. Its use proved to be very efficient in the removal of phenol, with initial concentration in the synthetic effluent from 2 to 7 mg L^{-1}. Only 4 of the tests carried out did not present 100% removal but were in an average of 90%. The final concentrations of phenol in the tests where it was not fully removed (around 0.1 mg L^{-1}) were below the concentration required by National Environment Council—CONAMA resolution No. 430 (0.5 mg L^{-1}) (Conama—Brazil, 2005).

The process showed low efficiency in TOC removal (Fig. 5), with it occurring only in five of the tests performed, ranging from 9 to 66%. This may

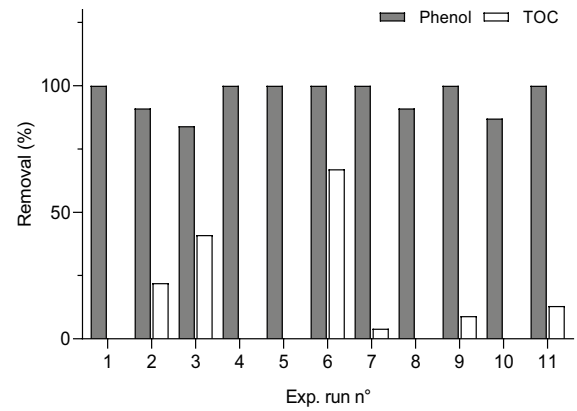

Fig. 5 Removal of phenol and TOC in synthetic textile dye effluent by electrocoagulation process

be due to the chromophore group responsible for the color and functional group, which binds the dye to the fiber (Volkov et al., 2020). The electrocoagulation treatment using scrap iron as an electrode may only be able to break the bond of the chromophore group, thus removing color, but not organic matter. Su et al. state that, although the chromophore group of the dye is destroyed by •OH radicals, some colorless degradation intermediates can be formed in the solution during the reaction (Su et al., 2011).

In Fig. 5, it is possible to observe that, for the TOC parameter, removal was not reproducible, since the results in the central point of the schedule (exp. runs No. 5, 6, and 7) presented great variation, with a

standard deviation of 38 mg L^{-1}. Figure 6 shows the wear of the scrap iron electrodes, with high wear in the anodes used in comparison to the cathodes. The wear is directly related to the density of the electric current applied, presenting an increase in the wear of the sacrificial electrodes (anode) with the increase of the electric current.

The highest sludge production (Fig. 7) was obtained in all tests where a current of 0.6A was applied, contrary to what was expected in the electrocoagulation process, where the production of sludge would increase with the electric current density and the wear of the electrodes. This expected behavior was not observed in this study. This effect possibly occurred because the maximum level of current used was 9.0 A, a value that was not considered high when compared to other studies using electrocoagulation.

3.2 Electrocoagulation in Real Textile Effluent

Effluent collected from an equalization tank in a textile industry was submitted to the initial characterization process, to observe the properties of a raw effluent. These characteristics are presented in Table 4.

The initial and final values of pH and electrical conductivity obtained in the electrocoagulation tests with the real effluent are presented in Fig. 8. All pH values were in the same range, starting at an average of 8 and ending at 12, the same values found in the treatment of the synthetic effluent, and above

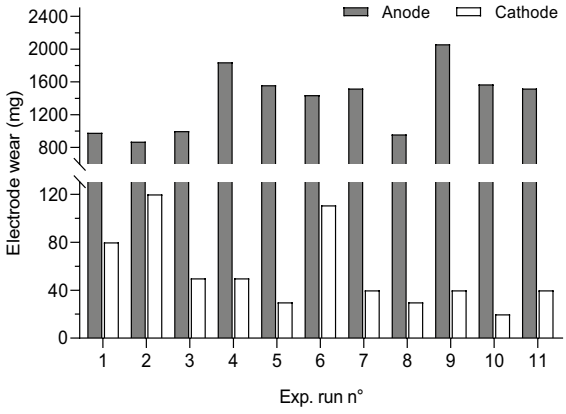

Fig. 6 Wear of scrap iron electrodes used in the electrocoagulation process of synthetic textile dye effluent. *Standard deviation of center points, exp. runs Nos. 5, 6, and 7 = 61 mg (anode) and 43 mg (cathode)

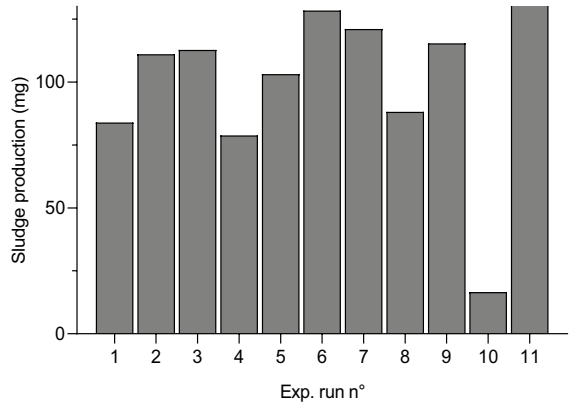

Fig. 7 Sludge production in the electrocoagulation process for textile dye oxidation in synthetic effluent

the acceptable 5–9 pH range allowed by the Brazilian regulations established by CONAMA resolution No. 430 (Conama, 2005). This indicates the need for adjustments in this parameter after application of the treatment, so that this effluent is in condition to be discharged into a body of water. Electrical conductivity follows the same pattern, but no limits are found in the Brazilian discharge resolutions.

According to Tak et al. (2015), in the electrocoagulation process, pH is a parameter that affects treatment performance, since different types of metal hydroxides are formed in electrocoagulation and changes can occur in the process depending on the initial pH. Akhtar et al. (2020) evaluated initial pH as an influencing parameter in electrocoagulation using iron electrodes and found it to be a determining factor

Table 4 Characterization of raw textile effluent from the equalization tank after the sieve system

Characterization of raw effluent	
Color	1209.20 Pt Co L^{-1}
Turbidity	1461 NTU
pH	7.89
Conductivity	702.10 µS cm^{-1}
Temperature	22 °C
COD	689.7 mg O_2 L^{-1}
BOD_5	906.1 mg O_2 L^{-1}
Total solids	83.70 mg L^{-1}
Phenol	1.35 mg L^{-1}
TOC	216.3 mg L^{-1}

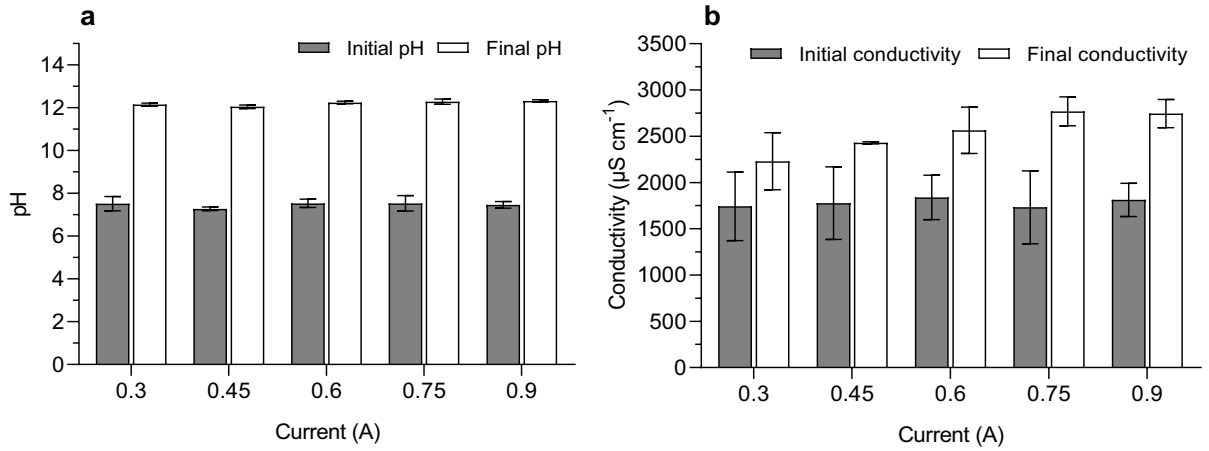

Fig. 8 Initial and final values of pH (**a**) and electrical conductivity (**b**) for the oxidation process of textile dye in real effluent using electrocoagulation

in the efficiency of the process. Torres et al. (2019) tested different pH ranges (3–8) in the removal of color, turbidity, and TOC in a textile effluent with a commercial $Ti/Ti_{0.7}Ru_{0.3}O_2$ electrode, finding better results with an initial pH close to neutral. In addition, it is advantageous to use the original pH value of the effluent, without the need to implement a process to change the pH, thus reducing costs.

Electrical conductivity is directly proportional to the quantity of conducting ions present in the solution. The higher the concentration of ions in the effluent, the greater its capacity to conduct an electrical current, thus increasing the possibility of reactions between the substances present in the effluent. Torres et al (2019) observed that, by increasing electric current intensity, it was possible to remove up to 75% of TOC in 45 min of reaction time using 300 mA at pH 6.0.

Figure 9 shows the removal of color, turbidity, TOC, and COD in each current applied. The values represent the average of removal in triplicates.

In analyzing the removal of color and turbidity of the real textile effluent, it is observed that the removal averages remained at the same levels, regardless of the electric current intensity applied. This shows a higher current is not required to obtain good removal efficiency, and the lowest current (0.3 A) can be applied, saving electricity costs, which could be interesting in scaled-up processes. These results deviate from what has been reported in the literature, as in the works of Kobya and Demirbas (2015) and Abu Ghalwa et al.

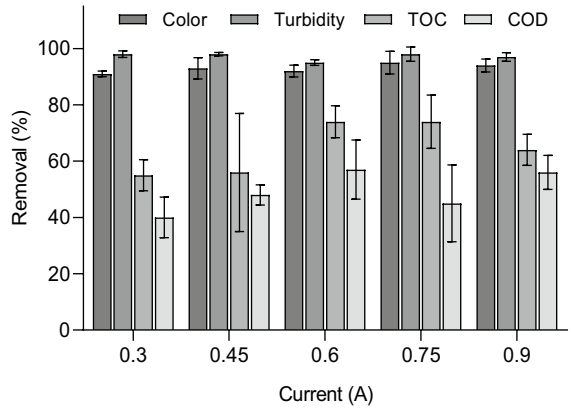

Fig. 9 Removal of color, turbidty, TOC, and COD in real textile dye effluent using the electrocoagulation process

(2016). In these works, when electrocoagulation was applied for the treatment of industrial effluents, an increase in electric current density caused an increase in the coagulant production rate, consequently resulting in greater oxidation of the electrodes and also an increase in the generation of hydrogen and oxygen gases on the surface of the electrodes, thus leading to the transport of coagulated material.

Regarding TOC removal, the averages presented little variation, from 55 to 75%, with the best result obtained with a current of 0.6 A. This shows that the electric current is not significant for this parameter. COD removal by electrocoagulation also showed similar efficiency among the electric currents applied,

ranging from 40 to 57%, with the highest removal value found with the 0.6 A current. Once again, it is evidenced that higher current densities are not required to obtain good results. Bener et al. (2019) evaluated the removal of TOC and COD in a textile effluent using aluminum electrodes. After 120 min of electrolysis at an initial pH of 5, they found an efficient removal of TOC, at approximately 65%, and low removal of COD, with the maximum value obtained being 18%. As for the presence of phenol, the treatment using scrap iron as electrodes showed a treatment efficiency of 100%, with total removal of this parameter occurring in all experiments.

It was also observed that the final treatment effluent presented a yellowish color (Fig. 10) in all the tests performed. Therefore, the researchers decided to analyze the presence of iron at the end of the electrocoagulation process, since that element is present in the scrap electrodes.

The soluble iron analysis (Fig. 11) showed that higher iron concentrations were found at lower electric current densities, with a maximum value of 8 mg L^{-1} at a current of 0.3 A. The concentration of dissolved iron can be directly related to color removal, according to Abu Ghalwa et al. (2016). When there is an increase in pH, consequently, there is an increase in the concentration of dissolved iron present in the samples during the electrochemical process, increasing the formation of iron hydroxides that absorb the dye molecules and make their removal more efficient.

Although the treated effluent presents dissolved iron, the values are below those allowed by the

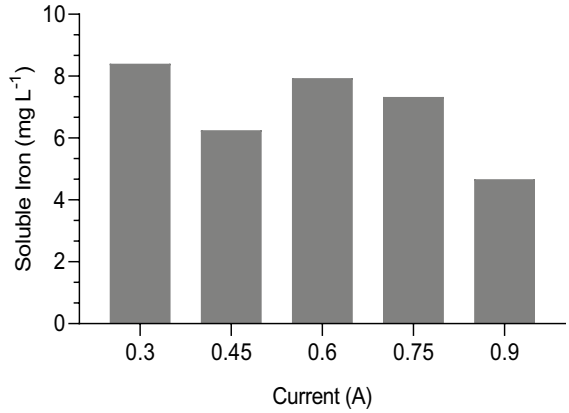

Fig. 11 Concentration of dissolved iron in real textile industry effluent after electrocoagulation process using scrap iron electrodes

Brazilian regulations established by CONAMA resolution No. 430 (15 mg L^{-1} Fe) (Conama, 2005), not requiring a secondary treatment before its release into a water body.

The wear of the electrodes was another parameter analyzed in the electrocoagulation of the real textile effluent. Results can be observed in Fig. 12. The anode wear stands out in relation to the cathode and is directly linked to the applied electric current density and the iron species formed. In the case of Fe (III), this species could be directly generated from the sacrificial electrode, depending on the applied voltage by direct charge transfer involving electro-dissolution of the anode. In addition, Fe (II) can be easily oxidized

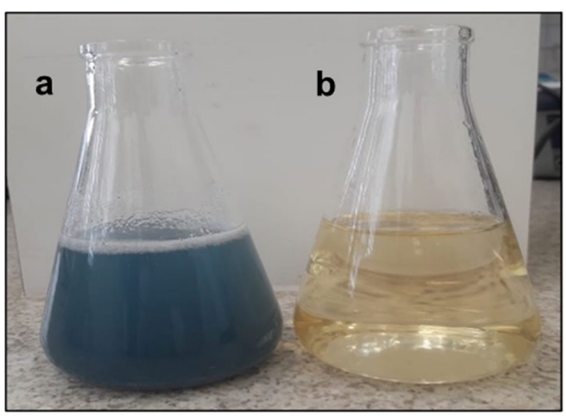

Fig. 10 Raw effluent from the textile industry (**a**) and after the electrocoagulation process (**b**)

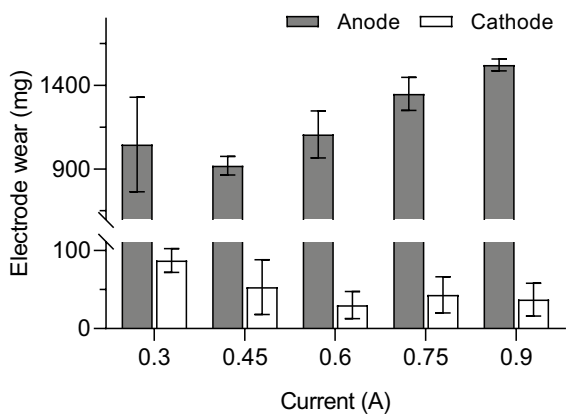

Fig. 12 Wear of scrap iron electrodes by electrocoagulation process in real textile dye effluent

into insoluble $Fe(OH)_3$ in the presence of O_2, which is usually dissolved in water (Garcia-Segura et al., 2017). Therefore, the higher the electric current applied in the treatment, the greater the wear of the sacrificial electrode, which is evident in the results obtained using scrap iron as electrodes.

The production of sludge during the electrocoagulation process of the real textile effluent (Fig. 13) showed a higher amount produced at the higher current densities, related to the higher wear of the scrap iron electrodes and the higher production of coagulants generated during the electrocoagulation treatment. Bener et al. reported similar results, where the amount of sludge generated during electrocoagulation increased as the current density was increased (Bener et al., 2019).

4 Conclusions

Electrocoagulation using scrap iron as electrodes proved to be an efficient treatment for effluents containing dyes in synthetic and real effluents. The main advantages of the process are the lower generation of sludge and not requiring the addition of chemicals. The electrochemical process showed good efficiency in the treatment of the analyzed effluents, presenting an average removal of 95% of color, 95% of phenol, and low production of sludge, in the synthetic effluent; in the real effluent from the textile industry, an average of 92% of color removal, 97% of turbidity, 100% of phenol, up to 65% of TOC, 49% of COD, and low production of sludge were obtained.

The statistical analysis showed the applied electric current density parameter is not significant for the oxidation of textile dye in synthetic and real effluents.

Studies involving the use of industrial waste are increasingly important, adding value to products without the need to consume pure raw materials, as is the case of scrap iron.

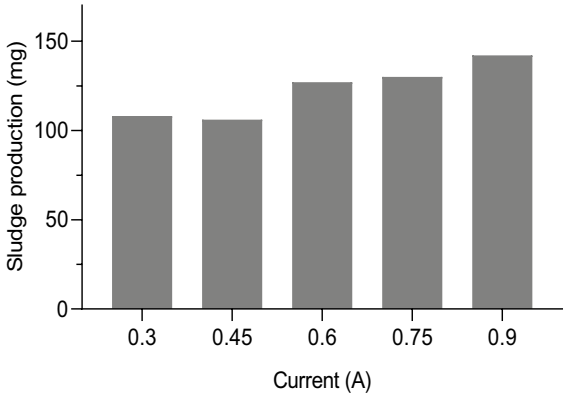

Fig. 13 Sludge production by electrocoagulation process for textile dye oxidation in real effluent

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Data Availability All data generated or analyzed during this study are included in this published article (and its supplementary information files).

Declarations

Ethics Approval and Consent to Participate Not applicable.

Consent for Publication Not applicable.

Competing Interests The authors declare no competing interests.

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