Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2022.28956

276 (2022) 116–123
November

Color and dissolved organic carbon removal from textile industry wastewater through oxidation and electrocoagulation

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Received 2 May 2022; Accepted 1 October 2022

ABSTRACT

Electrocoagulation in combination with an oxidizing agent is a very promising process for the treatment of water in the textile industry. In the present study, an experimental design was carried out to compare the influence of oxygen and ozone as oxidizing agents, in the removal of color, UV and dissolved organic carbon (DOC) present in textile wastewater of industrial origin. The results show that there is no significant difference in the removal efficiency of true color, DOC and UV when both oxidants are used with electrocoagulation, with average values of 88.7% ± 2.5%, 49.7% ± 3.6%, 28.5% ± 3.4%, respectively. The amount of iron generated by electrocoagulation, and its complete precipitation by oxidation in the presence of air or ozone, are the key factors for the removal of contaminants. This indicates that the process is carried out by adsorption of organic matter on the iron precipitates and not by its direct oxidation with ozone, with the advantage that the oxidation of iron with air is cheaper than with ozone. ANOVA analysis of the design of experiments showed that the applied current has a statistically significant effect on DOC removal, but not on color and UV removal.

Keywords: Wastewater; Textile industry; Electrocoagulation; Ozonation; Air; Oxidation

1. Introduction

The textile industry is dedicated to the production of fabrics, fibers, yarns, and products derived from these. It is one of the most important in the world; however, it is the one with the highest water consumption and its liquid waste contains many contaminants of different nature [1]. Some of the damage caused by these effluents are: decreased growth of aquatic flora by preventing the penetration of light, suppression of the reoxygenation capacity of streams, contamination of groundwater by leaching and infiltration, aesthetic problems due to high coloration and high degree of toxicity, in particular because certain azo dyes can be carcinogenic and mutagenic [2,3].

Mexico is one of the largest denim producers in the world, and a very important supplier to the US market.

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This has caused the presence of a wide range of chemicals in wastewater samples from this type of industry in Mexico, many with known dangerous impacts. Most of these substances are used during textile manufacturing processes or are created because of the breakdown of chemicals used in textile processing. These contaminants hinder the purification processes and cause a growing problem in the bodies of water that are used as sources of supply to the population. Due to the environmental pollution generated by the effluents of the textile industry, the elimination of dyes represents a technological challenge in water treatment processes.

There are different biological and physicochemical methods used in the treatment of wastewater containing dyes. These methods have technical and economic limitations, and it has been reported in the literature that a single process is not efficient for color degradation and mineralization of the compounds contained in these effluents [4,5]. Electrocoagulation is an emerging technology for water treatment based on an electrochemical process, in which pollutants are removed by applying the principle of chemical coagulation. However, in this case, the coagulant is generated by oxidation by inducing an electric current through parallel metal plates (sacrificial electrodes) immersed in the liquid medium to be treated [6]. The metal anode generates cations that destabilize the colloids, due to the formation of polyvalent polyhydroxide complexes [7].

In the case where iron electrodes acts as an anode, two types of coagulants can be formed in situ: ferrous hydroxide $Fe(OH)_2$ (under alkaline conditions) or ferric hydroxide $Fe(OH)_3$ (under acidic conditions) [8]. At the end of the formation of iron hydroxides, the colloids agglomerate (mainly those with negative charge) and, subsequently, other contaminant particles interact with the agglomerates being removed by complex formation or due to electrostatic attraction between them [9]. These precipitates, together with the pollutants removed from the water, are separated by sedimentation and then properly disposed of as contaminated solid waste. This phenomenon is favored by the presence of oxidants that ensure the complete precipitation of the formed hydroxides.

In the particular case of textile dyes, the predominant mechanism of elimination includes the adsorption of dye molecules, either by electrostatic attraction or physical trapping and precipitation, the former occurring at pH values greater than 6.5 and the latter in the range of 4–6 [10].

One of the advantages of electrocoagulation is that it produces up to 50% less sludge than chemical coagulation and does not increase the concentration of dissolved solids in the treated water.

On the other hand, ozonation is considered as one of the potential methods to be used in in the oxidative process of wastewater from the textile dyeing industry [11]. Ozone has been used for the removal of inorganic species due to its high oxidation potential, but also because it increases the efficiency of the electrocoagulation–flocculation process and degrades organic matter [12,13].

In addition, the oxygen in the air has the ability to oxidize other substances by accepting their electrons and thus forming oxides, but little research has been done studying oxidation with air and electrocoagulation together; therefore, when combined with electrocoagulation, it is important to compare the behavior of air oxidation with respect to ozone oxidation, as it is not clear what the main color removal mechanism is with this process. If color is removed by adsorption of contaminants on hydroxides formed during electrocoagulation, both oxidants can be effective in promoting their precipitation, with the advantage that air oxidation is cheaper than ozone oxidation.

2. Materials and methods

The wastewater considered for this study was obtained from a textile industry located in the state of Puebla, Mexico. Its initial characteristics were as follows: 10.74 pH units, 9.025 mS/cm electrical conductivity (EC), 4,422 mg/L total dissolved solids (TDS), 96,000 UPt-Co true color units, 320 nephelometric turbidity units (NTU), 812.50 mg/L dissolved organic carbon (DOC) and 75.37 UV absorbance at 250 nm.

2.1. Measuring techniques and equipment

A Thermo Scientific Orion STAR A329 portable meter was used to measure pH and electrical conductivity (EC). True color was measured using a HACH-DR/2800 spectrophotometer (Method 8025 at 455 nm); standard method 5310B was followed for DOC analysis with a SHIMADZU-TOC-L instrument, and UV absorbance at 250 nm with an Agilent Technologies UV-VIS double beam spectrophotometer equipped with a 1 cm path length quartz cell. DOC, color and absorbance measurements were performed after vacuum filtration of the samples through a 0.45 μm pore size membrane. For color and absorbance measurements, the samples were diluted with demineralized water, with a factor of 200 and 25, respectively.

Regarding UV absorbance, in spectroscopy, the conventional unit of wavelength is the nanometer (abbreviated nm). Absorbance (sometimes called optical density, abbreviated OD) is a measure of the amount of radiant energy absorbed. The absorbance (dimensionless) at a given wavelength depends on the number of molecules in the light path length. Absorptions by compounds containing only single bonds and unshared electron pairs are generally quite weak. However, intense absorption of UV and/or visible radiation occurs when a compound contains molecules with conjugated double or triple bonds. Compounds with isolated double bonds, triple bonds, peptide bonds, aromatic systems, carbonyl groups and other heteroatoms have their maximum absorbance in the UV region, which is very important for the determination of organic compounds. In this case, a maximum peak was detected in the UV region ($\lambda max = 250$ nm), which was taken as an indirect measure of the amount of organic compounds present in the solution. In this document, the variable indicating the absorbance at 250 nm will be referred to as UV.

True color implies that substances absorb light at visible wavelengths, indicating compounds with poly aromatic structures, substituted aromatics, condensed molecules or complex ions. In the visible region (400–800 nm) we see the visible color of a solution, which corresponds to the wavelengths of light that it transmits, not absorbs. The absorbing color is the complementary of the transmitting color. Most of the compounds that give water its color contains one or more aromatic rings, although not all aromatic rings produce color. The platinum-cobalt (UPt-Co) method measures at a single wavelength which is characteristic of yellow hues. DOC is a method of measuring the carbon content of dissolved organic substances.

2.2. Experimental model set-up

The experiments were carried out in a device (a schematic diagram of the experimental setup is shown in Fig. 1) composed of two process units: (1) an oxidation column and (2) an electrocoagulation reactor with a quadrangular pyramid-shaped hopper at the bottom for the accumulation of the sludge formed during the process; both units were made of acrylic.

The water was recirculated from the top of the reactor to the middle of the oxidation column with a peristaltic pump (7583-02, Cole-Parmer). A perforated plastic vessel containing a porous stone diffuser immersed in fine sand was installed inside the oxidation column. The diffuser was connected via a hose to an ozone generator (Ozone Ecological Equipment's) generating 2.11 g/h of O_3 by means of a compressor (WCF609196, Atlas Copco). This same compressor was used to supply 2 L/min of air at a pressure of 68.9 KPa in the tests that did not require ozone. In the oxidation column the flow was downward, counter-flowing with the gas to achieve a longer gas–water contact time. The volume of water in the column was 3 L. From the bottom of the column the water was returned by gravity to the bottom of the reactor.

Inside the reactor, 32 carbon steel electrodes were installed, with the following dimensions: 21 cm long, 15 cm wide and 1.7 cm thick. The electrode spacing was set at 0.03 cm. A plastic grid was placed to seat the electrodes and allow the water to pass freely upwards through the reactor. The plates were connected in parallel monopolar to a direct current (DC) power supply (DCS10-120E, Sorensen) with a maximum capacity of 10 V and 120 A, working galvanostatically. During the tests, the polarity of the electrodes was changed manually every hour to avoid passivation. In the reactor the flow was upward, with a treatment volume of 22 L and a residence time of 22 min.

2.3. Description of the tests

The system was operated in continuous flow, with 100% recirculation of the water between the reactor and the oxidation column. The water passing through the reactor received the iron produced by the oxidation of the electrodes. Part of the iron generated continuously, on contact with the water, formed poorly soluble hydroxides that precipitated and were deposited at the bottom of the reactor. The water from the top of the reactor, when sent to the oxidation column, encountered ozone or air (depending on the test being carried out) for about 3 min, which favored the oxidation of the still dissolved iron. The water, upon returning to the reactor, first passed through the conical sludge blanket zone, which favored the precipitation and retention of the hydroxides formed.

Six experiments (in duplicate) were performed to evaluate oxidation with air or ozone (Table 1), at three different current intensities (equivalent to three coagulant generation rates and three current densities). To establish whether the iron production rate affected the final efficiency of the process, the same theoretical iron production at the end of the operation time was considered.

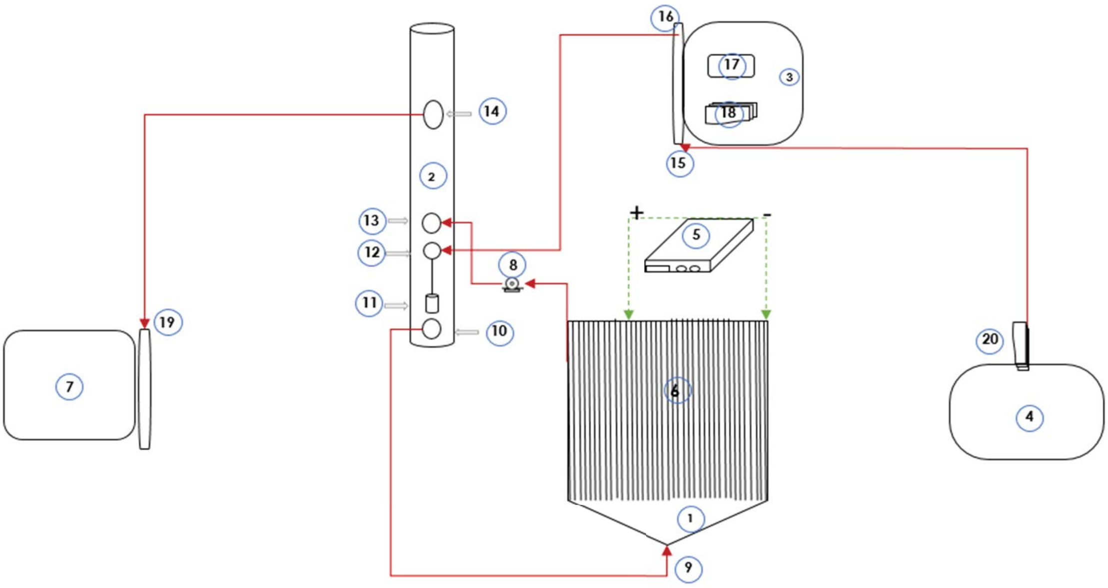

Fig. 1. Experimental set-up. 1.- Electrocoagulation reactor. 2.- Oxidation column. 3.- Ozonator. 4.- Compressor. 5.- Power supply. 6.- Iron electrodes. 7.- UV lamp for destruction of residual ozone. 8.- Peristaltic pump. 9.-Water inlet to the reactor coming from the column. 10.- Water outlet and sampling in the column. 11.- Diffuser. 12.- Air or ozone inlet. 13.- Inlet of water to the column coming from the reactor. 14.- Residual air or ozone outlet. 15.- Air inlet from the compressor. 16.- Outlet of air or ozone to the column. 17.- Regulation knob for ozone generation. 18.- Flow meter. 19.- Residual air or ozone inlet. 20.- Flow meter.

Table 1
Test conditions

Test	Electric current (A)	Current density (A/m²)	Ozone applied* (g)	Air applied* (L)	Iron applied* (g)	Test time (min)
1	30	30.7	4.9	0	72.87	140
2	30	30.7	0	280	72.87	140
3	15	15.4	9.5	0	70.27	270
4	15	15.4	0	540	70.27	270
5	7.5	7.7	19.0	0	70.27	540
6	7.5	7.7	0	1080	70.27	540

* Total at end of the test

The design of experiments shown in Table 1 corresponds to a 2^2 factorial (FD), with two additional runs to have enough degrees of freedom to allow the ANOVA to be analyzed. One variable is numerical with three levels (the applied electrical current) and one variable is categorical (the type of oxidant) with two levels (ozone and air). For purposes of the analysis of the design of experiments, a coded nomenclature of the study variables was used. In the case of the applied electrical current, the coded levels were for 30 A (+1), for 15 A (–0.333) and for 7.5 A (–1); regarding the type of oxidant used: ozone (–1), air (+1).

The parameters that were kept constant in all tests were: pH (7.0 ± 0.5), total problem water volume (25 L), recirculation flow rate (1 L/min), and air flow rate supplied (2 L/min) either to the ozone generator or directly to the oxidation column. The pH value equal to 7 was chosen due to the result of preliminary tests (not shown here) which indicated that the best color removal efficiencies with this type of water were obtained at this pH. The electrodes were washed with 10% sulphuric acid dissolution, rinsed repeatedly with demineralized water, and dried before each test.

The response variables for each experiment were UV (absorbance at 250 nm), DOC (mg/L) and true color (UPt-Co). For analysis of response curves, samples were taken at the outlet of the oxidation column, at time intervals that depended on the current intensity applied (30 A every 20 min, 15 A every 60 min, and 7.5 A every 120 min).

Since the concentrations (*C*) were measured as a function of time (*t*) during the tests, we can compare their dependence with appropriate kinetic models, to obtain the rate constants (*k*). In this case, zero [Eq. (1)], first [Eq. (2)], and second-order [Eq. (3)] models were compared.

$$\frac{dC}{dt} = -k \tag{1}$$

$$\frac{dC}{dt} = -kC \tag{2}$$

$$\frac{dC}{2dt} = -kC^2 \tag{3}$$

On the other hand, the removal efficiencies of the experiment (at the end of each test) were analyzed by multiple linear regression [14], for a general model of the type shown in Eq. (4).

$$Y = Xb + e \tag{4}$$

where X is the test matrix of the design of experiments (in coded variables), Y is the response vector of the design of experiments (removal percentages of color, DOC or UV), b is the vector of coefficients of the model, and e is the error vector implicit in the model. Vector b was calculated by minimizing the sum of squares of the errors in vector e, via Eq. (5).

$$b = \left(X^T X\right)^{-1} X^T Y \tag{5}$$

where X^T is the transpose of the design matrix into coded variables.

3. Results and discussion

The purpose of the study was to establish if the removal of the textile dye, via electrocoagulation–oxidation, resulted from an attack on the chromophore groups of the dyes (responsible for the color), or only due to its effect on the oxidation of the iron generated during the electrochemical process, and the subsequent formation of hydroxides responsible for the adsorption of contaminants from the water. The results showed that the removal of organic matter was not due to an oxidation process of organic matter, but to an adsorption phenomenon, since oxidation with air gave the same results. The oxygen present in the air cannot be expected to break the double and triple bonds in the dye molecules.

The removal of true color, DOC and UV (Figs. 2–4, respectively), represented as the ratio of the final value with respect to the initial one ($\text{Color}_f/\text{Color}_i$, $\text{DOC}_f/\text{DOC}_i$, UV_f/UV_i) show that, regardless of the oxidant used (ozone or air), or the magnitude of the current applied, the results are practically the same and the same decrease of the contamination indicators was reached at the end of the tests: on average, 88.7% in true color, 49.7% in DOC and 28.5% in UV (Table 2). This indicates that the total amount of iron produced at the end of the experiment is the key factor, and that this precipitates completely with the help of any of the oxidants applied.

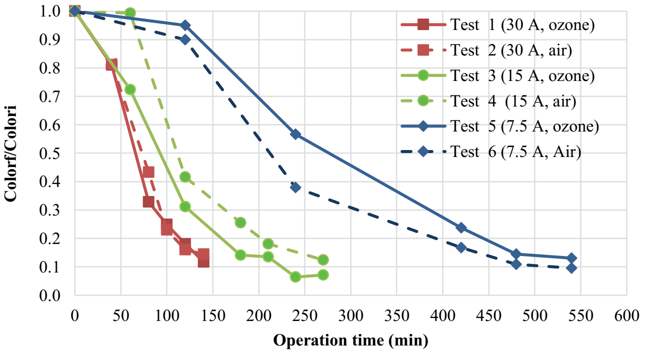

Fig. 2. Relative removal of true color with respect to time.

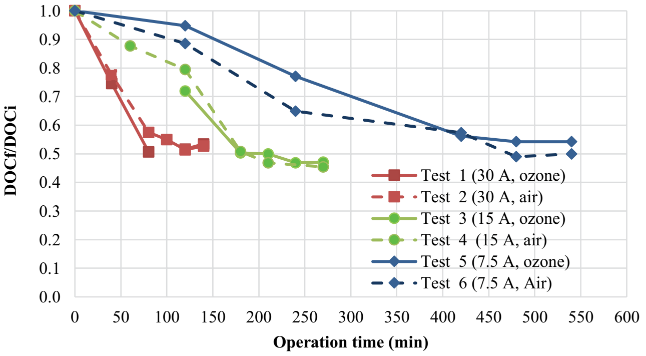

Fig. 3. Relative DOC removal over time.

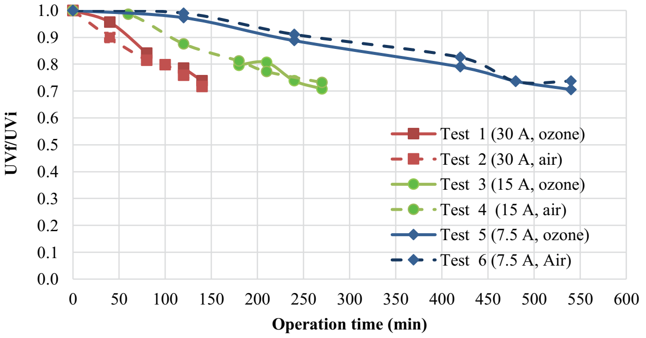

Fig. 4. Relative UV decreases over time.

Table 2
Results of the tests carried out

Test	Applied current (A)	Oxidant used	Removal efficiencies at the end of the test (%)		
			True color	DOC	UV
1	30	Ozone	88	47	26
2	30	Air	86	47	28
3	15	Ozone	93	53	35
4	15	Air	88	55	27
5	7.5	Ozone	87	46	29
6	7.5	Air	90	50	26
Average			88.7 ± 2.5	49.7 ± 3.6	28.5 ± 3.4

In all the analyzed cases, the studied process (electrocoagulation + oxidant) was efficient in reducing the true color, followed by DOC, and finally UV. The application of air or ozone was equivalent, as both encourage the conversion of Fe^{2+} to Fe^{3+} and thus the precipitation of the poorly soluble iron hydroxides.

Besides the fact that no clear difference was observed between the two oxidants at the end of the process, both presented the same behavior during the tests, that is, they exhibited practically the same rate of decrease of the process response variables (color, DOC, and UV) at the end of the elapsed operation time for the three applied currents. In all cases, a good fit with first-order kinetics was observed (Table 3). The value of the rate constant k depends on the electrical current applied, but not on the type of oxidant. This confirms that, for the three response variables (Color, DOC and UV), the efficiency is independent of this study factor.

It is important to note that the theoretical iron production with respect to time, according to Faraday's Law [15], has different slopes depending on the current applied (as shown in Figs. 5 and 6). Fig. 5 shows the response surface of the iron production as a function of the current and the application time. Fig. 6 shows the operating conditions of the experiments carried out and the approximate amount of iron produced in all the tests (dotted line) with which the maximum decrease of the true color and DOC was reached (Figs. 2 and 3). It is interesting to note that both parameters

Table 3
Decrease rate constants of response variables as a function of time

Test	First-order model [Eq. (2)]					
	Color		DOC		UV	
	k (min^{-1})	R^2	k (min^{-1})	R^2	k (min^{-1})	R^2
Test 1 (30 A, Ozone)	0.0142	0.99	0.0055	0.94	0.0021	0.99
Test 2 (30 A, Air)	0.0137	0.98	0.0055	0.98	0.0024	1.00
Test 3 (15 A, Ozone)	0.0102	0.99	0.0031	0.99	0.0017	0.96
Test 4 (15 A, Air)	0.0076	0.98	0.0032	0.97	0.0012	0.99
Test 5 (7.5 A, Ozone)	0.0036	0.97	0.0012	0.99	0.0006	0.99
Test 6 (7.5 A, Air)	0.0043	0.99	0.0014	0.99	0.0005	0.97

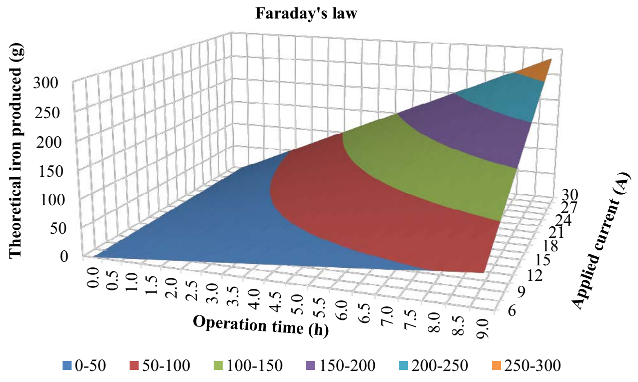

Fig. 5. Response surface of the theoretical iron production, as a function of time and applied current, according to Faraday's Law.

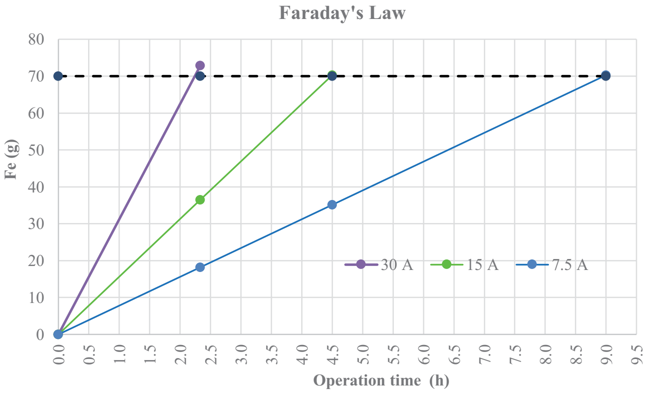

Fig. 6. Behavior of the theoretical production of iron as a function of the operation time, and of the three values of applied current, according to Faraday's Law. The dotted line indicates the approximate amount of iron established in the study tests.

reached a plateau in which their decrease was no longer significant, even if more iron continued to be added. On the other hand, UV appeared to be able to decrease more if the test had been left for longer (Fig. 4), that is, with higher iron production.

When designing the tests, the hypothesis was raised that the iron application rate would influence the efficiency of the process, however, the results show that there is no clear effect that confirms this idea. Figs. 7–9 show that the behavior of all tests was similar for each response parameter, and the final efficiency to reach the maximum iron produced was basically the same. This indicates that regardless of how quickly iron is added to water, it is the final amount that is important to have a significant effect on organic matter removal. On the other hand, these results provide enough information for practical purposes where short residence times may be required, the process still works, as long as the dose of iron is available.

The selected multiple linear regression models are indicated by Eqs. (6)–(8) with coded variables, where A is current applied and B is oxidant used. The procedure to estimate the parameters of the models was that of least squares. These models were used to determine the factors that

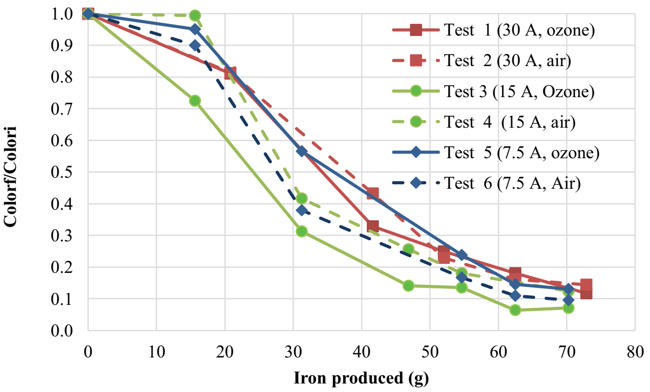

Fig. 7. Relative removal of true color to iron produced.

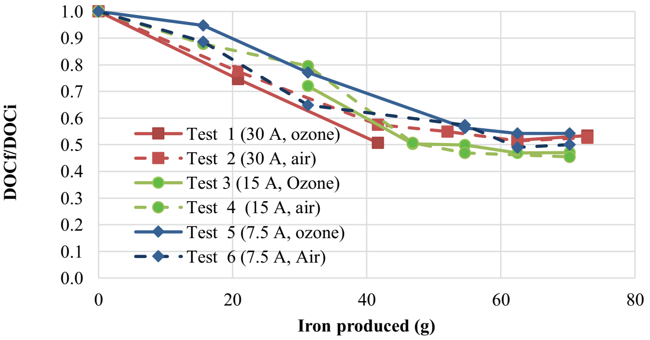

Fig. 8. Relative removal of DOC with respect to iron produced.

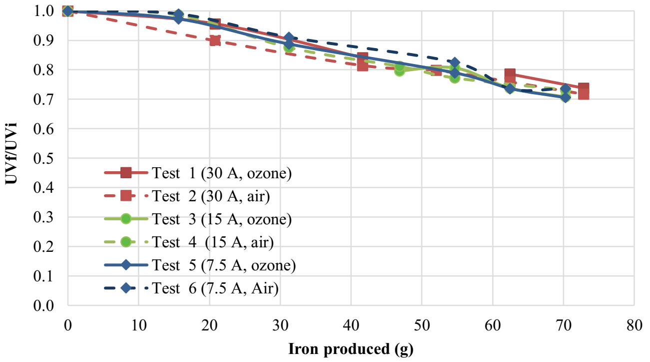

Fig. 9. Relative decrease of UV with respect to iron produced.

significantly affect the performance of the process, in terms of percent true color removal (%*R*Color), DOC (%*R*DOC) and UV (%*R*UV), with a 95% confidence interval (Table 2).

$$\%R\text{Color} = 90.56 - 0.7499A - 0.6666B - 2.8125AA \tag{6}$$

$$\%R\text{DOC} = 54.6249 - 0.5A - 1.0B - 7.1244AA \tag{7}$$

$$\%R\text{UV} = 31.3749 - 0.2499A - 1.4999B - 4.125AA \tag{8}$$

Table 4 shows the ANOVA results of the selected models. The R^2 statistic explains 47.9% of the variability in true

Table 4
ANOVA for true color, DOC an UV removal using FD

	SS	DF	MS	*F*	R^2	σ	MAE	*P*-value
%*R*Color model	15.0	3	5.0	0.61	0.479	2.86	1.44	0.669
Error	16.3	2	8.17					
Total	31.3	5	6.27					
%*R*DOC model	63.3	3	21.11	10.56	0.940	1.41	0.66	0.088
Error	4.0	2	2.0					
Total	67.3	5	13.47					
%*R*UV model	32.5	3	10.83	0.87	0.565	3.54	1.66	0.575
Error	25.0	2	12.5					
Total	57.5	5	11.5					

SS – sum of squares, DF – degrees of freedom, MS – mean square, F – MS/MSerror, σ – standard error of the estimate, MAE – mean absolute error.

color, 94% in DOC and 56.5% in UV. The value of p greater than the pre-established significance = 0.05 indicates that there is no statistically significant association between the response variable and any of the study factors.

These results indicate that none of the study variables (applied current and type of oxidant) have a statistically significant effect on the response variables; in other words: the limits selected for the applied electrical current do not substantially modify the results, and using ozone or air gives the same process efficiencies. However, the best models obtained for the three response variables gave a quadratic component with variable A (applied current), which indicates that maximum efficiency has already been reached with the limits used for this study and it would be useless to increase further iron production.

Some studies report DOC removal efficiencies between 50%–60% when using electrocoagulation coupled with ozone to treat textile water (Bilińska et al. [11]), which confirms the values obtained in this study.

Regarding the efficiencies using air, Ya et al. [16] worked with a synthetic dye (Eriochrome Black T-EBT), with which they obtained color removals of 100% and 78% of organic carbon when applying pressurized air in an electrocoagulation system with iron electrodes. The authors showed that the highest removal efficiencies were obtained at acidic and neutral pH conditions due to the effective conversion of Fe^{2+} to Fe^{3+} in the presence of dissolved oxygen under pressure. Furthermore, they attributed these results to the lower solubility of Fe^{3+}, whose precipitates adsorbed the dyes effectively. The efficiencies obtained in this work using air at atmospheric pressure were similar, even though in this case a real water and not a synthetic water was used.

4. Conclusions

- In this study, an oxidation and EC treatment was applied to a sample of real wastewater from the textile industry, with the aim of comparing the use of air or ozone as oxidants, as well as evaluating the effect of the application rate of iron (through three different current intensities) on color, DOC and UV removal efficiencies.
- In all the cases analyzed, the performance of electrocoagulation + oxidant was similar when using air or ozone, in terms of its ability to reduce true color, followed by DOC and finally UV. Both oxidants have practically the same rate of decrease of the response variables with the three currents applied, and the results fit to a first-order kinetic model with respect to time.
- The statistical analysis of the results obtained at the end of the operation time shows that neither the applied current nor the type of oxidant used significantly influence the efficiency of the system. For each contaminant, the removal percentage varies very little in the study area, therefore, there is no difference between the use of ozone or air during the electrocoagulation process for color, DOC and UV removal. This suggests that the elimination is carried out by adsorption on the iron hydroxides formed, and not by oxidation of the organic matter. For this reason, it is recommended to use air instead of ozone, due to its lower operating and handling costs.
- On the other hand, the regression models indicate that there is a quadratic behavior in the current intensity. This proves that maximum efficiency has already been reached and better results will not be achieved by increasing the amount of iron in the system.
- Electrocoagulation, ensuring the total oxidation of the iron produced during electrolysis, allows a significant removal of organic contaminants present in wastewater from the textile industry. This can allow the reuse of water in the dyeing process itself, since most of the color is removed.

Symbols

A	—	Current applied
B	—	Oxidant used
b	—	Vector of coefficients of the model
C	—	Concentrations
DOC	—	Dissolved organic carbon
e	—	Error vector implicit in the model
EC	—	Electrical conductivity
k	—	Rate constant
t	—	Time
TDS	—	Total dissolved solids
UV	—	UV absorbance at 250 nm
X	—	Matrix of the design of experiments
X^T	—	Transpose of the design matrix into coded variables

Y — Response vector
$\%R$ — Removal efficiency

Acknowledgments

The authors acknowledge the Mexican Institute for Water Technology (IMTA) for financial support and the Water Quality laboratory team for valuable help in the experimental and analytical procedures.

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