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Degradation of Dyes in Real Textile Industry Wastewater Samples by Fenton Process

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Abstract: This research proposes a solution to the problem of industrial safety and the improvement of environmental processes in the textile industry through the degradation of dyes from its processes using the Fenton reaction. This advanced oxidation technology makes it possible to reduce the possible environmental impacts caused by the organic matter generated in industrial activity. Sampling was conducted under real operating conditions in the homogenization tank of the Industrias Printex SAS wastewater treatment plant. Advanced oxidation was carried out applying the Fenton process using iron from ferrous sulfate heptahydrate as a catalyst and hydrogen peroxide at different concentrations in a 6-post flocculator. Among the results obtained, an optimal dose of 571 ppm of iron and 38 ppm of hydrogen peroxide was found through statistical analysis with the STATGRAPHICS program using the 2k factorial design and constructing response surfaces. A comparative study between the Fenton treatments, treatment with hydrogen peroxide, and treatment with ferrous sulfate heptahydrate showed the Fenton reaction at neutral pH, attributed to the presence of some organic acids such as citric acid, used as a chelating agent (Fenton modified). The proposed treatment was compared with the one that uses chlorine, applying it in three (3) different tanks through a multifactorial ANOVA with the data collected when scaling the treatment, finding that the proposed Fenton treatment is more efficient. This research was innovative because it demonstrated the feasibility of scaling up the Fenton process to neutral pH in a textile wastewater treatment plant, reducing operating costs and minimizing environmental impact.

Keywords: wastewater, colorants, Fenton, textile industry, real conditions.

芬頓法降解實際紡織工業廢水樣品中的染料

摘要：在這項工作中，通過使用芬頓反應從其工藝中降解染料，提出了解決紡織行業工業安全問題和改善環境過程的方法。這種高級氧化技術可以減少工業活動中產生的有機物可能對環境造成的影響。在“工業 Printex SAS”污水處理廠的均化池中，在真實運行條件下進行採樣，採用芬頓法，以七水硫酸亞鐵中的鐵為催化劑，以不同濃度的過氧化氫為原料，採用芬頓工藝進行高級氧化。六 柱絮凝器。在獲得的結果中，通過採用 二ķ 響應面設計的 統計數據 程序進行統計分析，找到了 五百七十一 pp 米 鐵和 三十八 pp 米 過氧化氫的最佳劑量；在芬頓處理、過氧化氫處理和七水硫酸亞鐵處理之間進行了比較研究，顯示芬頓反應在中性酸鹼度 值下，這歸因於某些有機酸的存在，例如作為螯合劑的檸檬酸（芬頓修改）; 將提議的處理與使用氯的處理進行比較，將氯應用在三 (三) 個不同的罐中，通過多因素 方差分析與縮放處理時收集的數據，發現提議的處理更有效 (芬頓)。這項研究的創新之處在於它可以證明在紡織廢水處理廠中將芬頓工藝擴大到中性 酸鹼度 值的可行性，從而降低運營成本並最大限度地減少對環境的影響。

关键词：廢水、著色劑、芬頓、纺织工业、真實情況。

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1. Introduction

Organic wastewater treatment is becoming an increasingly critical issue in sustainable development as it contains a large number of organic, refractory, and toxic pollutants. Advanced oxidation processes (AOP) have been widely applied and developed for the direct mineralization of organic contaminants or for improving the biodegradability of organic contaminants through oxidation [1]. Compared to other AOPs, the Fenton process is the most popular technique due to its interesting advantages, such as wide application range, strong anti-interference ability, simple operation, and fast degradation and mineralization [2].

Fenton's reagent was discovered in 1894 by HJH Fenton. It is a mixture of H_2O_2 and the Fe(II) salt, which, under certain conditions, shows strong oxidizing properties relative to most organic compounds [3].

In the Fenton oxidation, organic matter and color can be degraded by producing an OH radical using H_2O_2 and Fe^{2+} at a low pH value. However, in the Fenton oxidation, the regeneration of Fe^{2+} ions is very slow, significantly reducing the oxidation efficiency due to the lower formation of hydroxyl radicals. The reaction of generation of hydroxyl radicals is as follows [4]:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{\cdot} + OH^{-} \qquad (1)$$

The hydroxyl free radical is the primary oxidizing species, formed by the decomposition of hydrogen peroxide, catalyzed by Fe^{2+} in the absence of ferrous ion chelating agents. It is the second oxidizing agent after fluorine ($HO^{\cdot}$ Eo=-2.8 V vs. fluorine, Eo=-3.0 V), and it can carry out non-specific oxidations of some organic compounds. When a sufficient concentration of hydroxyl free radical and other radicals is generated, the oxidation reactions of organic compounds can reach total mineralization.

Among the domain of AOPs, the classical Fenton process (CFP) is well recognized in literature due to its notable advantages like ease of operation, fewer chemicals requirement, fast degradation/removal of contaminants, and non-sophisticated instruments requirement, etc. In the CFP, ferrous iron (Fe^{2+}) acts as a catalyst, and hydrogen peroxide (H_2O_2) acts as an oxidizing agent [5]. The oxidation mechanism for the Fenton process is shown in Figure 1. Based on this principle, the Fenton process has been widely used in various types of organic wastewater treatment [2].

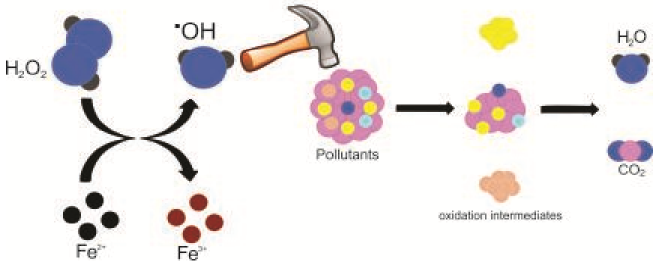

Fig. 1 Reaction mechanism for the Fenton process (Adapted from [6])

The efficiency of degradation of organic contaminants in the Fenton process depends on the operating parameters, such as the pH of the wastewater, the concentration of the Fenton reagent, and the initial concentration of organic contaminants. Homogeneous Fenton reaction is the most efficient around pH ~ 3, which can be challenging to implement at a large scale [7].

The Fenton process has three obvious flaws: the narrow working pH range, the high costs and risks associated with reagent handling, transportation, and storage (H_2O_2 and homogeneous solution of iron ions), and significant iron sludge related to the second contamination. To overcome the shortcomings, the Fenton process is optimized and improved to form various optimized Fenton processes [2]. However, the Fenton process has the shortest reaction time among advanced oxidation processes. Therefore, it can be implemented and integrated into existing water and wastewater treatments such as coagulation, filtration, and biological oxidation [3].

The industrial effluents generated constitute textile dyes that can contaminate bodies of water, deteriorate water quality, and disturb aquatic ecosystems, in addition to seriously affecting human health. Therefore, the existence of colored effluents in the environment has raised one of the main concerns of environmental problems, and it is necessary to decontaminate them before disposal [8].

Effluents from the textile industry are of concern from an environmental point of view since they are generated in large volumes and have a high concentration of organic matter and toxicity. In addition, dyes stand out among the substances present in these effluents because part of them are not fixed on the fiber during the dyeing process, leaving the effluent with a strongly colored appearance and difficult to treat [9].

A significant amount of polluting effluent is produced by the textile industry owing to the high water consumption (100–200 L/kg in textiles production) of fabric processing [10]-[11].

These industries use huge quantities of water and are a significant source of dye color pollution. The dye colors contain 2–3% phenolic compounds and 3–4% organic colorants [12].

The presence of dyes in water bodies has serious environmental consequences. On the one hand, they hinder the diffusion of oxygen and light while producing an unsightly appearance. On the other hand, they are considered persistent in the environment due to their chemical nature. In addition, they can present toxicity and carcinogenic and/or mutagenic potential [9].

The most important problem in applying the Fenton process on an industrial scale is the lack of standardized procedures. For example, different research teams used various doses of chemicals and other optimal ranges of reaction parameters. Therefore, a standardized procedure is required to compare the efficiency of a catalyst [3].

Wastewater treatment plants do not work or use conventional wastewater treatment techniques in developing countries [13].

There is the degradation of dyes within the industrial wastewater treatment processes, where chlorine is used as an input. This chemical input represents the greatest risk for the personnel working in these industries, their surroundings, and the environment. In addition, chlorination-based systems lead to the formation of chlorinated disinfection by-products (DBPs) with potential negative health effects [14]; a different alternative is sought to carry out the process of degradation of dyes from industrial wastewater.

In view of the above, this work seeks to find the best operating conditions for the Fenton process, which allow achieving the efficient and economic degradation of the dyes present in wastewater from the textile industry in the department of Quindío, using the lower amount of chemical substances for water treatment, avoiding pH adjustment; drawback that could be overcome by introducing ligands to chelate Fe(II) to maintain Fe(II) dissolution and generate subsequent reactive oxygen species (ROS) at neutral or weakly alkaline pH [15], which can be done taking advantage of the presence of citric acid [16] in the real wastewater, as a chelating substance and in this way achieve the scaling of the process. Furthermore, this treatment will improve the degradation of dyes in textile wastewater and eliminate the use of chlorine in the said process because the water discharged into the water sources complies with the parameters established in the current environmental regulations [17]. Furthermore, the purification plants capture water that is easier to treat, seeking water recycling within the same industries, and aquatic fauna and flora are less in danger of extinction. Therefore, industrial processes can be developed in such a way that they generate employment and development of our department in a sustainable and friendly way with the planet, conserving our territory. In the same way, they provide the industrial sector with the possibility of applying processes that minimize the risk of industrial safety and occupational health, generating an economic benefit.

The Fenton-based treatments reduce organic matter, toxic and recalcitrant compounds, making the effluent more susceptible to subsequent biological treatments and being able to be used as a final polish [9]. Furthermore, it has been successfully applied to reduce dissolved organic carbon (DOC) in municipal and groundwater and treat leachate effluents from municipal landfills and paper and textile companies. Therefore, the objective of this research was to degrade dyes in wastewater from the textile industry from the Fenton reaction.

2. Methods and Materials

The study was carried out at the industrial wastewater treatment plant facilities of the Industrias Printex S.A.S.

2.1. Experimental Design

The experimental design used to determine the optimal dose of the Fenton reagent was a 2k factorial design with k factors, each with two quantitative levels (concentration of ferrous sulfate heptahydrate and concentration of peroxide). The color is the research variable, considering that what is sought is to degrade the color in this type of water. The results were statistically processed with a confidence level of 95%, and response surfaces were constructed.

Three different tanks were taken to evaluate the behavior of the Fenton. Tests were carried out in triplicate and compared with the current treatment (chlorine). The results were statistically analyzed through variance - multifactorial ANOVA at 95% statistical confidence for the DOC and real color variables. To determine differences between tanks and compare the two treatments (conventional process and Fenton).

2.2. Sampling

Samples are taken under real operating conditions of the industrial wastewater treatment plant. The plant has two homogenization tanks operating in batches for the specific case. In this way, the sample is taken once the tank is full and just before carrying out the chlorination process (the conventional process used in Industrias Printex SAS).

2.3. Determination of pH and Temperature

The pH and temperature values were determined with a portable HACH sensION + 5059 potentiometer, an electrode with multiple parameters: pH, conductivity, and temperature, as shown in Figure 2.

Fig. 2 HACH sensION + 5059 portable potentiometer, multi-parameter electrode: pH, conductivity and temperature

The analyses were carried out in the Industrias Printex S.A.S wastewater treatment plant laboratory.

2.4. Determination of DOC

The DOC content was analyzed by the closed reflux method per the standard water analysis methods provisions, ed. 22. The equipment used was the Spectroquant® TR series Turbojet, MERCK. Model TR320, as shown in Figure 3. Similarly, in Figure 4, the Spectroquant NOVA 60, MERCK photometer is shown.

The tests were carried out in the wastewater treatment plant laboratory of Industrias Printex S.A.S, and the samples of the plant test were analyzed in an accredited laboratory, which in this case was LAIMAQ S.A.S.

Fig. 3 Spectroquant® TR series thermoreactor, MERCK. TR320 model.

Fig. 4 Spectroquant NOVA 60 photometer, MERCK

2.5. Fenton Processes

2.5.1. Determination of the Optimal Dose of the Fenton Reagent

By varying each of these parameters, tests were carried out to determine the treatment conditions: the concentration of the iron reagent and the oxidizing agent. The tests were carried out at pH < 5, considering that at this value, the water is found in the homogenization tanks, and the increase in costs can be avoided by adding acids. The reagents used for the treatment were: hydrogen peroxide and ferrous sulfate heptahydrate. It is important to highlight that adding ferrous sulfate heptahydrate lowers the pH, taking the water to values below 4UN. The different concentrations used to determine the optimal dose of Fe(II) and H_2O_2 are shown in Table 1.

Table 1 Concentrations used to determine the optimal dose of Fe(II) and H_2O_2

Ferrous sulfate heptahydrate concentration (ppm)	**Peroxide concentration (ppm)**
50	15
250	15
450	15
650	15
850	15
50	25
250	25
450	25
650	25
850	25
50	35
250	35
450	35
650	35
850	35
50	45
250	45
450	45
650	45
850	45
50	55
250	55
450	55

Ferrous sulfate heptahydrate concentration (ppm)	Peroxide concentration (ppm)
650	55
850	55
50	65
250	65
450	65
650	65
850	65

All the tests were carried out in a 6-station flocculator with the digital time control of 0-999 min/0.99 h or continuous and programmable speed of 20-300 RPM, Brand Y.Q, as shown in Figure 5, using volumes of 2000 mL, with stirring at a speed of 100RPM, for 15 min. The pH was adjusted to 6 UN (minimum admissible in Resolution 0631 of 2015) and allowed to settle for 10 min. The working temperature was 35°C (the temperature of the water in the homogenization tank). The times and speeds were taken according to the current operating conditions of the plant in order not to generate additional costs when scaling the process. The initial conditions of the sample are shown in Table 2.

Table 2 Initial conditions of the water in the homogenization tank

Tank	pH(UN)	Temperature (°C)	Color Real (m^{-1})		
			436 nm	525 nm	620 nm
1	4.71	35	5.6148	8.1627	5.0651

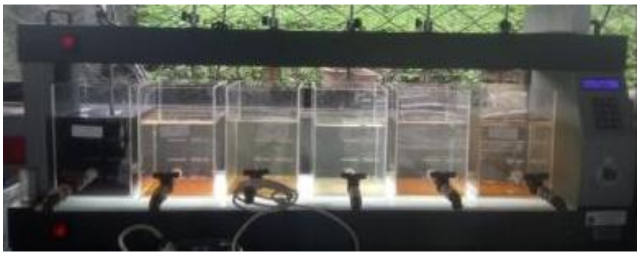

Fig. 5 Six-flocculator station with digital time control from 0-999 min/0.99 h or continuous and programmable speed from 20-300 RPM, Y.Q Brand

2.5.2 Application of the Optimal Dose in Three Tanks and Comparison with the Conventional Process (Chlorine)

Once the optimal dose of ferrous sulfate heptahydrate and peroxide has been obtained, tests are carried out in triplicate with the dosage found in 3 different tanks to determine differences between tanks. Additionally, triplicate samples are taken from the same chlorinated water (conventional process). The initial conditions of the water in each of the homogenization tanks are shown in Table 3.

Table 3 Initial conditions of the water in each homogenization tank

Tank	pH (UN)	Temperature (°C)	DOC (mgL^{-1})	Color Real (m^{-1})		
				436 nm	525 nm	620 nm
1	4,98	35	435	11,835	8,8813	8,2617
2	4,93	35	605	26,459	22,342	20,517
3	5,00	35	534	79,471	70,215	64,306

2.5.3. Minimum Dose Test and Scaling to the Real Conditions of the Treatment Plant

Although an optimal dose of ferrous sulfate heptahydrate and peroxide was determined, the tests demonstrated that color degradation is also generated in high percentages with low doses. It should be noted that the higher the dose, the higher the cost, making scaling unfeasible.

Due to the above, it was decided to set up a plant test using minimum doses of ferrous sulfate heptahydrate, and peroxide. This allows for equalizing the color degradation carried out with the conventional process (chlorine) and, in turn, generates less environmental risk and occupational impact.

A jar test is carried out using the water from a new tank previously homogenized and without chlorine. For this test, the minimum doses are applied. When verifying that the test works in the jar, the mathematical calculations were made to dose in the homogenization tank, and the process was applied in the plant.

Water pH, DOC, and color were measured before and after the treatment with Fenton.

3. Results and Discussion

3.1. Fenton Reagent Formulation Optimization

Figure 6 shows the equipment of jars used to carry out the reaction, materials used for taking samples, and evident color degradation.

Fig. 6 Jar test used to carry out the reaction, materials used for taking samples, and evident color degradation

Table 4 shows the true color results obtained by analyzing the samples after treatment with the Fenton reagent.

Table 4 Real color results after the application of Fenton at different concentrations

Ferrous sulfate heptahydrate concentration (ppm)	**Peroxide concentration (ppm)**	**Color Real (m^{-1})**		
		436 nm	**525 nm**	**620 nm**
50	15	10,607	8,5724	4,3664
250	15	0,02179	0,46506	1,0247
450	15	-0,95296	-0,67983	-0,6453
650	15	-0,18482	0,24052	0,85325
850	15	-0,12136	0,31524	0,91157
50	25	11,255	8,4479	5,2028
250	25	-0,47164	-0,77634	-1,0234
450	25	-0,43583	-0,85568	-1,0925
650	25	-0,65131	-0,98968	-1,2044
850	25	-0,11516	-0,60611	-0,90094
50	35	8,125	6,3269	2,7853
250	35	-0,4117	-0,67549	-0,8595
450	35	-0,12827	-0,47417	-0,67267
650	35	-0,98844	-1,3324	-1,5533
850	35	-0,16131	0,12078	0,67167
50	45	6,3774	4,9274	2,2089
250	45	-0,06022	-0,54502	-0,809
450	45	0,15206	-0,51107	-1,4403
650	45	-1,2256	-0,76241	-0,033188
850	45	0,63257	0,13947	-0,19598
50	55	11,686	8,4112	3,1946
250	55	1,5789	0,1009	-0,60782
450	55	0,79594	0,28	-0,10009
650	55	1,1877	0,65155	0,41375
850	55	-0,35696	-0,012684	0,62857
50	65	12,149	9,1769	0,49367
250	65	-1,7727	-1,7121	-1,7102
450	65	1,9463	1,6294	1,9288
650	65	1,0213	0,48265	0,19159
850	65	-0,98033	-0,56629	0,19817

The color analysis of the initial sample was taken as a reference (before treatment), and removal percentages were calculated at the three wavelengths. The percentages of color removal are observed in Table 5.

Table 5 Percentages of real color removal after applying Fenton at different concentrations

Ferrous sulfate heptahydrate concentration (ppm)	**Peroxide concentration (ppm)**	**% Color removal**		
		436 nm	**525 nm**	**620 nm**
50	15	-89	-5	14
250	15	100	94	80
450	15	100	100	100
650	15	100	97	83
850	15	100	96	82
50	25	-100	-3	-3
250	25	100	100	100
450	25	100	100	100
650	25	100	100	100
850	25	100	100	100
50	35	-45	22	45
250	35	100	100	100
450	35	100	100	100
650	35	100	100	100
850	35	100	99	87
50	45	-14	40	56
250	45	100	100	100
450	45	97	100	100
650	45	100	100	100
850	45	89	98	100
50	55	-100	-3	37
250	55	72	99	100
450	55	86	97	100
650	55	79	92	92
850	55	100	100	88
50	65	-100	-12	90
250	65	100	100	100
450	65	65	80	62
650	65	82	94	96
850	65	100	100	96

Optimization of the response surface experimental design indicated the optimal concentrations of ferrous sulfate heptahydrate and peroxide that result in the lowest absorbance, which is directly related to the lowest color value. For data analysis, the Statgraphics Centurión XVI software was used (Table 6). Optimal concentrations of ferrous sulfate heptahydrate and peroxide show the optimal values for each wavelength studied and the averages obtained at the end.

Table 6 Optimum values of concentrations of ferrous sulfate heptahydrate and peroxide

Wavelengths	**436 nm**	**525 nm**	**620 nm**	**Averages**
Peroxide concentration (ppm)	34	37	41	38
Ferrous sulfate heptahydrate concentration (ppm)	595	583	536	571

Therefore, the optimal concentration parameters of ferrous sulfate heptahydrate and peroxide to obtain a greater color degradation measured at 436 nm is 595:34; for the color measured at 525 nm, it is 583:37, and for the color measured at 620 nm, it is 536:41. Finally, the average optimal values were calculated to establish a value that achieves the best removals in the three wavelengths, for which the result 571:38 was obtained.

Figure 7 shows the absorbance response surface at 436 nm, a graphical representation of the response surface design optimized for the lowest sample absorbance at 436 nm.

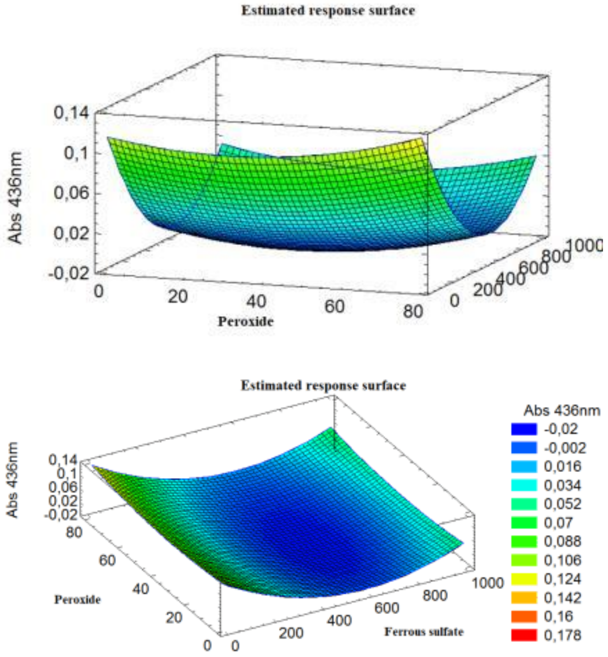

Fig. 7 Absorbance response surface at 436 nm

Analysis of variance – ANOVA of the response surface model for absorbance at 436 nm shows that Fenton degrades color over a wide range of concentration combinations of ferrous sulfate heptahydrate and peroxide, making it possible to minimize costs when scaling the project.

The regression equation (Eq. 2) to calculate the absorbance at 436 nm, which has been fitted to the model data, is:

$$\text{Abs 436 nm} = 0{,}115212 - 0{,}000766083 \times \text{Peroxide} - 0{,}000411148 \times \text{Ferrous sulfate} + 0{,}0000124816 \times \text{Peroxide}^2 - 1{,}3125\text{x}10^{-7} \times \text{Peroxide} \times \text{Ferrous sulfate} + 3{,}49397\text{x}10^{-7} \times \text{Ferrous sulfate}^2 \quad (2)$$

Figure 8 shows the absorbance response surface at 525 nm, a graphical representation of the response surface design optimized for the lowest sample absorbance at 525 nm.

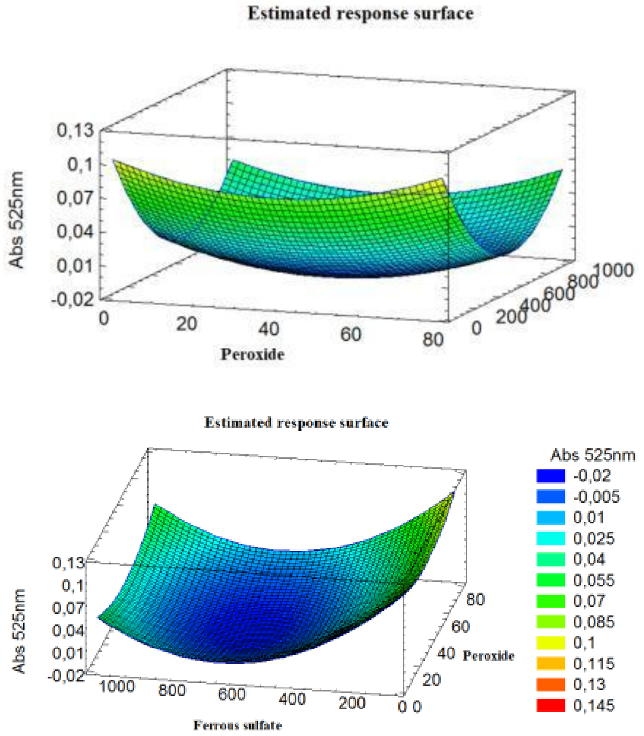

Fig. 8 Absorbance response surface at 525 nm

Analysis of variance – ANOVA of the response surface model for absorbance at 525 nm shows that Fenton degrades color over a wide range of concentration combinations of ferrous sulfate heptahydrate and peroxide, making it possible to minimize costs when scaling the project.

The regression equation (Eq. 3) was used to calculate the absorbance at 525 nm, which has been fitted to the model data:

$$\text{Abs 525 nm} = 0{,}103205 - 0{,}00128903 \times \text{Peroxide} - 0{,}000338745 \times \text{Ferrous sulfate} + 0{,}0000164681 \times \text{Peroxide}^2 + 9{,}42482\text{x}10^{-8} \times \text{Peroxide} \times \text{Ferrous sulfate} + 2{,}87458\text{x}10^{-7} \times \text{Ferrous sulfate}^2 \quad (3)$$

Figure 9 shows the absorbance response surface at 620 nm, a graphical representation of the response surface design optimized for the lowest sample absorbance at 620 nm.

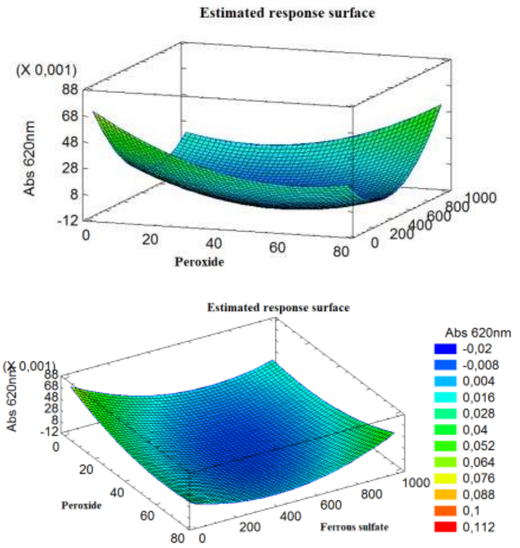

Fig. 9 Absorbance response surface at 620 nm

Analysis of variance – ANOVA of the response surface model for absorbance at 620 nm shows that Fenton degrades color over a wide range of concentration combinations of ferrous sulfate heptahydrate and peroxide, making it possible to minimize costs when scaling the project.

The regression equation (Eq. 4) was used to calculate the absorbance at 620 nm, which has been fitted to the model data:

$$\begin{aligned}&\text{Abs 620 nm}\\&= 0{,}0709351 - 0{,}00149661 \times \text{Peroxide}\\&- 0{,}000194582 \times \text{Ferrous sulfate}\\&+ 0{,}0000118375 \times \text{Peroxide}^2 + 9{,}66865\text{x}10^{-7}\\&\times \text{Peroxide} \times \text{Ferrous sulfate} + 1{,}44319\text{x}10^{-7}\\&\times \text{Ferrous sulfate}^2 \qquad (4)\end{aligned}$$

3.2. Multifactorial Analysis of Variance to Compare the Conventional Process (Chlorine) and Fenton in Three Tanks

Three samples from three different tanks were treated with the optimal dose values found in the previous analysis. In addition, DOC and color analyses were performed on these samples to evaluate the treatment's efficiency with Fenton and compare it with the conventional treatment (chlorine).

The results of the DOC analysis before and after the treatments are shown in Table 7.

Table 7 Results of the DOC analysis of the samples before and after the treatments

Tanks	**Initial**	**Analysis of DOC (mgL^{-1})**	
		Conventional treatment	**Treatment Fenton**
1	435	265	189
		252	200
		271	198
2	605	394	345
		396	362
		394	348
3	534	351	308
		352	285
		346	296

The statistical analysis of the experimental data was carried out using a variance analysis - multifactorial ANOVA (Table 8).

Table 8 Statistical analysis multifactorial ANOVA of the experimental data of DOC

Source	**Sum of Squares**	**Df**	**Mean Square**	**F-Ratio**	**P-Value**
Main Effects					
A: Treatment	13338,9	1	13338,9	225,45	0,0000
B: Tank	64114,8	2	32057,4	541,82	0,0000
Interactions					
AB	434,778	2	217,389	3,67	0,0569
Residual	710,0	12	59,1667		
Total (Corrected)	78598,4	17			

According to the values of P ($p < 0.05$), it is found that the tank and treatment factors have a significant influence on the response variable; that is, the DOC varies in each tank and differs in the treatments studied. Therefore, it is shown that there are statistically significant differences at a confidence level of 95.0%

Figure 10 shows the variation of the DOC in each tank and the two applied treatments. This research evaluated the conventional treatment in the wastewater treatment plant where chlorine is employed to degrade the dyes and the Fenton treatment technique. It revealed that the Fenton treatment works better in the three tanks since lower DOC values were obtained. It should be noted that the initial DOC values in each tank are different, the highest being that of tank 2.

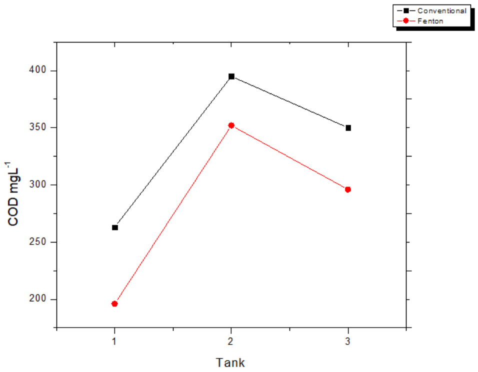

Fig. 10 DOC variation in each tank and the two applied treatments

The results of the color analysis m^{-1} at 436 nm are shown in Table 9. Color analysis results m^{-1} at 436 nm to the samples before and after the treatments.

Table 9 Results of the color analysis m^{-1} at 436 nm to the samples before and after the treatments

Tanks	Initial	Color 436 nm (m^{-1})	
		Conventional treatment	**Treatment Fenton**
1	11,835	0,040	0,164
		1,912	0,000
		6,054	1,760
2	26,459	0,000	3,810
		1,222	1,762
		10,143	0,000
3	79,471	0,000	0,000
		2,974	0,396
		0,000	1,042

The statistical analysis of the experimental data was carried out using a variance analysis - multifactorial ANOVA, as shown in Table 10. P values ($p>0.05$) indicate that the tank and treatment factors do not significantly influence the response variable; that is, the color m^{-1} at 436 nm does not vary significantly in each tank and does not differ in the treatments studied. Therefore, it is shown that there are no statistically significant differences at a confidence level of 95.0%.

Table 10 Statistical analysis multifactorial ANOVA of the experimental data of color at 436 nm

Source	Sum of Squares	Df	Mean Square	F-Ratio	P-Value
Main Effects					
A: Treatment	9,99194	1	9,99194	1,25	0,2853
B:Tank	13,1346	2	6,56728	0,82	0,4629
Interactions					
AB	2,15954	2	1,07977	0,14	0,8749
Residual	95,8742	12	7,98952		
Total (Corrected)	121,16	17			

In Figure 11, the color variation is shown as m^{-1} at 436 nm in each tank during the applied treatments. This research evaluated the conventional treatment in the wastewater treatment plant where chlorine is employed to degrade the dyes and the Fenton treatment technique. It revealed that the Fenton treatment works better in the three tanks since lower color values m^{-1} at 436 nm. It should be noted that the initial color values m^{-1} a 436 nm in each tank are different, the highest being that of tank 2.

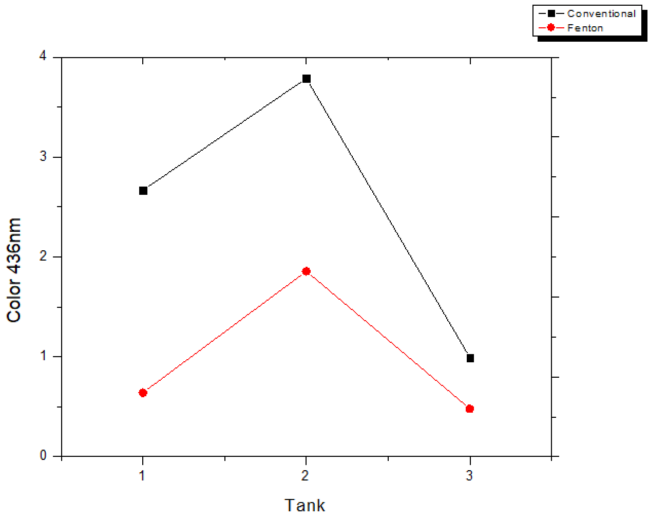

Fig. 11 Color variation at 436 nm in each tank during the two applied treatments

The results of the m^{-1} color analysis at 525 nm are shown in Table 11.

Table 11 Results of the color analysis m^{-1} at 525 nm to the samples before and after the treatments

Tanks	Initial	Color 525 nm (m^{-1})	
		Conventional treatment	**Treatment Fenton**
1	8,8813	0,000	0,000
		0,837	0,000
		5,264	0,265
2	22,342	0,000	2,949
		0,000	1,537
		7,556	0,000
3	70,215	0,000	0,000
		1,475	0,372
		0,000	0,754

The statistical analysis of the experimental data was carried out using a variance analysis - multifactorial ANOVA, as indicated in Table 12. P values ($p > 0.05$) demonstrated that the tank and treatment factors do not significantly influence the response variable; that is, the color m^{-1} at 525 nm does not vary significantly in each tank and does not differ in the treatments studied. Therefore, it is shown that there are no statistically significant differences at a confidence level of 95.0%.

Table 12 Statistical analysis multifactorial ANOVA of the experimental data of color at 525 nm

Source	**Sum of Squares**	**Df**	**Mean Square**	**F-Ratio**	**P-Value**
Main Effects					
A: Treatment	4,75861	1	4,75861	0,95	0,3493
B:Tank	7,52915	2	3,76457	0,75	0,4931
Interactions					
AB	2,50899	2	1,25449	0,25	0,7827
Residual	60,1974	12	5,01645		
Total (Corrected)	74,9941	17			

Figure 12 shows the color variation m^{-1} at 525 nm in each tank during the applied treatments. This research evaluated the conventional treatment in the wastewater treatment plant where chlorine is employed to degrade the dyes and the Fenton treatment technique. It revealed that the Fenton treatment works better in the three tanks since lower values of color m^{-1} were obtained at 525 nm. It should be noted that the initial color values m^{-1} at 525 nm in each tank are different, the highest being that of tank 2.

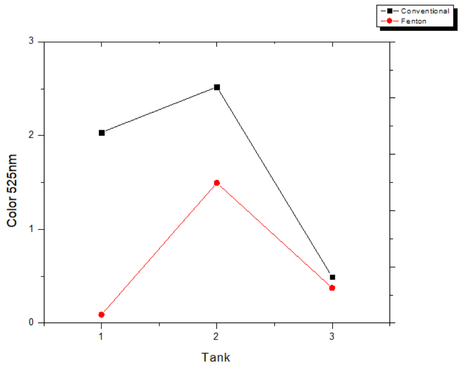

Fig. 12 Color variation at 525 nm in each tank during the two applied treatments

The results of the m^{-1} color analysis at 620 nm, before and after the treatments, are shown in Table 13.

Table 13 Results of the color analysis m^{-1} at 620 nm to the samples before and after the treatments

Tanks	**Initial**	**Color 620 nm (m^{-1})**	
		Conventional treatment	**Treatment Fenton**
1	8,2617	0,000	0,000
		0,334	0,000
		4,838	0,000
2	20,517	0,000	2,068
		0,000	1,316
		5,851	0,000
3	64,306	0,000	0,000
		0,916	0,332
		0,000	0,544

The statistical analysis of the experimental data was carried out using a variance analysis - multifactorial ANOVA, as indicated in Table 14. P values (p>0.05) demonstrated that the tank and treatment factors do not significantly influence the response variable; that is, the color m^{-1} at 620 nm does not vary significantly in each tank and does not differ in the treatments studied. Therefore, it is shown that there are no statistically significant differences at a confidence level of 95.0%.

Table 14 Statistical analysis multifactorial ANOVA of the experimental data of Color at 620 nm

Source	**Sum of Squares**	**Df**	**Mean Square**	**F-Ratio**	**P-Value**
Main Effects					
A: Treatment	3,27595	1	3,27595	0,97	0,3430
B:Tank	4,62948	2	2,31474	0,69	0,5210
Interactions					
AB	2,19693	2	1,09847	0,33	0,7274
Residual	40,3251	12	3,36043		
Total (Corrected)	50,4275	17			

In Figure 13, the color variation m-1 is shown at 620 nm in each tank during the applied treatments. This research evaluated the conventional treatment in the wastewater treatment plant where chlorine is employed to degrade the dyes and the Fenton treatment technique. It revealed that in the first two tanks, the Fenton treatment works better since lower color values m^{-1} are obtained at 620 nm; in tank 3, the two processes degraded the color without presenting differences. It should be noted that the initial values of color m^{-1} at 620 nm in each tank are different, the highest being that of tank 2.

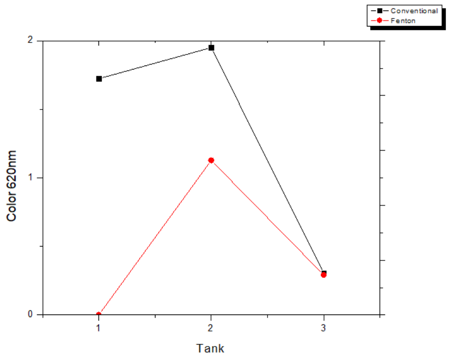

Fig. 13 Color variation at 620 nm in each tank during the two applied treatments

3.3. Scaling to the Minimum Doses

Previous tests, especially investigating the response surfaces, were taken as a reference. This research evaluated the conventional treatment in the wastewater treatment plant where chlorine is employed to degrade the dyes and the Fenton treatment technique. It revealed that where it is evident that the color degradation begins when applying concentrations of ferrous sulfate heptahydrate of 200 ppm and peroxide concentrations of 25 ppm. A jar test was carried out with the water contained in a homogenization tank, showing color degradation. With these concentration values and knowing the volume of water stored in the tank, the mathematical calculations are made to scale the doses and carry out the application in the plant.

The values taken for the plant test are shown in Table 15.

Table 15 Values taken for the plant test

Tank volume (m^3)	**Ferrous Sulfate Heptahydrate (ppm)**	**Peroxide (ppm)**	**Kg Ferrous Sulfate Heptahydrate**	**Liters of peroxide 50%**
108	200	25	22	5,4

Figure 14 shows the initial conditions of the water in the homogenization tank of the water treatment plant of Industrias Printex S.A.S.

Fig. 14 Color variation at 620 nm in each tank during the two treatments applied

Figure 15 shows ferrous sulfate heptahydrate added in the gutter that feeds the homogenization tank to be treated. At this point, ferrous sulfate heptahydrate is mixed with the water due to the turbulence generated in the Parshall.

Fig. 15 Application of ferrous sulfate heptahydrate in the gutter of the water treatment plant of Industrias Printex S.A.S.

It can be seen in Figure 16 that peroxide was similarly applied in the gutter to generate a good mixture.

Fig. 16 Application of peroxide in the gutter of the water treatment plant of Industrias Printex S.A.S.

The water passing through the gutter reaches the homogenization tank, which has constant agitation. There the Fenton reaction takes place, as described in Figure 17. The water takes coloration in the tank after the addition of chemical inputs.

Fig. 17 Homogenization tank after Fenton application

Subsequently, the water is sent by pumping to the landfill, where the anionic flocculant is applied prior to pH conditioning at 6UN. As can be seen in Figure 18 and Figure 19, the floc is formed after applying the flocculant, and the floc increases in size for its subsequent sedimentation.

Fig. 18 Application of anionic flocculant in discharge dump to flocculation tanks

Fig. 19 Floc formation after flocculant application

A sample was taken to compare it with the initial conditions and conduct real color and DOC analyses on both samples before and after treatment with Fenton. For example, in Figure 20, a sample from the homogenization tank before treatment can be seen on the left, and a sample treated with Fenton and previously settled on the right.

Fig. 20 On the left, the sample from the homogenization tank before treatment, and on the right, the sample treated with Fenton and previously settled

Table 16 shows the water analysis results in the homogenization tank (before treatment) and the weir (after the Fenton). We can observe a removal of 30% in terms of DOC, a parameter compared to Resolution 0631 of 2015, which establishes a maximum of 400 mgL^{-1} of DOC, and meets the quality criteria for discharges. Unfortunately, the color cannot be compared since it is not a variable with a parameter set in the standard; however, removal percentages up to 65% are shown in this parameter.

Table 16 Water analysis results in the homogenization tank (before treatment) and in the landfill (after the Fenton)

Sample	**pH (UN)**	**Temp. (°C)**	**DOC (mgL^{-1})**	**Color Real (m^{-1})**		
				436 nm	**525 nm**	**620 nm**
Homogenization tank	5,00	35	560	12,906	10,815	10,536
Dump	6,00	35	391	8,005	5,246	3,654
% Removal			30	38	51	65

3.4. Cost-Benefit Analysis of the Oxidation Process

The minimum concentrations of the inputs were taken as a reference. They were used to produce the Fenton reaction, the cost information of said inputs, and the cost-benefit study of the oxidation process.

The estimated cost only includes the Fenton reagent; that is, it does not include acidulants or alkalinizers. Taking as a reference that the pH found in the homogenization tanks is less than 5.00UN, every time nylon garments are dyed, the use of acidulants would not be necessary. However, when the dyeing process includes cotton garments and the pH rises, an acidulant must be applied, and the minimum pH must be 5.00UN.

In normal operation at the end of the Fenton process, the minimum pH must be adjusted to 6.00UN, which is what is established in the discharge regulations. For the said adjustment, tests were carried out with hydrated lime, finding that the average consumption is equivalent to the current consumption of the said product.

Table 17 presents the differences between Fenton vs. Chlorine treatments and economic savings when implementing the Fenton process.

Table 17 Differences between Fenton vs. chlorine treatments

Fenton Treatment		**Chlorine Treatment**	
Cost Ferrous Sulfate Heptahydrate ($/Kg)	$ 1.175	Coagulant cost ($/Kg)	$ 1.605
Peroxide Cost ($/Kg)	$ 2.680	Chlorine cost ($/Kg)	$ 4.200
Dosage Ferrous Sulfate Heptahydrate (ppm)	200	Coagulating dose (ppm)	110
Peroxide Dosage (ppm)	25	Chlorine dose (ppm)	52
Consumption Ferrous sulfate heptahydrate (Kg/m^3)	0,200	Coagulant consumption (Kg/m^3)	0,109

Continuation of Table 17

Peroxide Consumption (Kg/m^3)	0,060	Chlorine consumption (Kg/m^3)	0,052
Cost Consumption Ferrous sulfate heptahydrate (\$/m^3)	\$ 235	Cost Coagulant consumption (\$/m^3)	\$ 175
Peroxide Consumption Cost (\$/m^3)	\$ 160	Cost Chlorine consumption (\$/m^3)	\$ 218
Total Fenton Cost (\$/m^3)	\$ 395	Cost Chlorine Treatment (\$/m^3)	\$ 393
		Additional (energy cost \$/m^3)	\$ 31
		Total cost with chlorine (\$/m^3)	\$ 424
Saving (\$/m^3)		\$ 30	

Considering that, on average, the plant treats 360 m^3/day, the daily savings with the Fenton treatment amounts to USD 10,800=, which indicates that in the 30 days of the month, USD 324,000= will be saved, for a total of USD 3,888,000= in one year.

The foregoing indicates that the implementation of the Fenton process is feasible in terms of costs.

3.5. Analysis of the Fenton Treatment vs. Treatment with Peroxide and Ferrous Sulfate

A comparative study was carried out between the Fenton treatments, the peroxide treatment, and the ferrous sulfate heptahydrate treatment.

In each of the cases, the following concentrations were used (based on the study previously carried out to determine the optimal doses):

Fenton treatment:

Ferrous sulfate heptahydrate 571 ppm

Hydrogen peroxide 38 ppm

Hydrogen peroxide treatment: 38 ppm

Treatment with ferrous sulfate heptahydrate: 571 ppm

3.5.1. First Analysis (1)

For this study, the initial pH of the reaction (2.8 UN) was considered, and the three treatments were carried out.

3.5.2. Second Analysis (2)

For this study, the initial pH of the reaction (2.8 UN) was considered, and the three treatments were carried out. Later, it was adjusted to pH 7.00 UN. This step was done because it is a suitable pH for dumping water into water sources.

3.5.3. Third Analysis (3)

For this study, the initial pH of the reaction (7.00 UN) was considered, and the three treatments were carried out. Later it was adjusted again to pH 7.00 UN. This step was done because it is suitable for dumping water into water sources, and applying the reagents lowers the pH.

After the treatments, each sample was scanned between 320nm and 1100nm, obtaining the results observed in Figure 21.

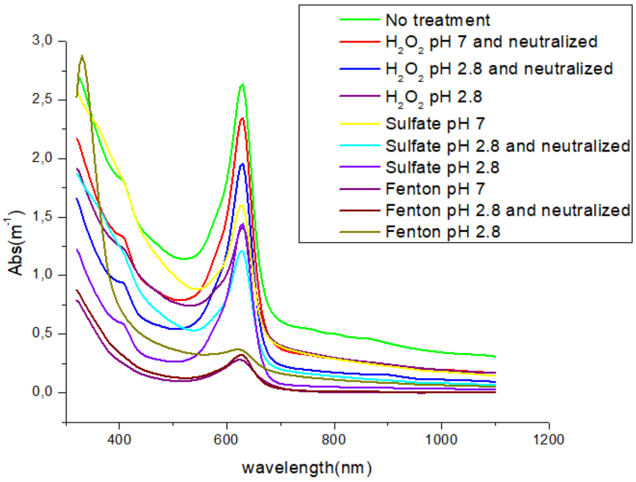

Fig. 21 Absorbances in the visible spectrum according to each treatment

An analysis of DOC and SST was conducted, obtaining the results shown in Table 18 and Figure 22, showing a greater efficiency of the Fenton (2) and Fenton (3) treatments.

Table 18 Chemical oxygen demand and total suspended solids in the treatments

Treatment	**DOC**	**DOC**	**SST**
	% Rem	**mgL^{-1}**	**mgL^{-1}**
No Treatment	0	526	229
Peroxide (1)	25	393	133
Peroxide (2)	26	388	76
Peroxide (3)	5	498	137
Sulfate (1)	53	247	24
Sulfate (2)	64	188	56
Sulfate (3)	63	196	108
Fenton (1)	61	205	48
Fenton (2)	68	170	4
Fenton (3)	68	169	3

When analyzing the results, we can observe that since it is water without prior treatment, it has a high content of solids that are partially removed by coagulation with ferrous sulfate heptahydrate, which leads to a % color removal, without ignoring that it does lead to. A Fenton process is carried out because the % color removal is even higher when applying this treatment and, in turn, reduces the DOC. The data obtained can be seen in Table 19.

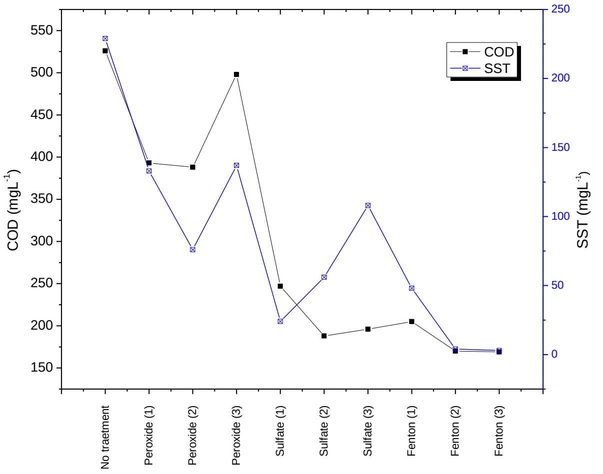

Fig. 22 Chemical oxygen demand and total suspended solids in each of the treatments

Table 19 Real color in the three total wavelengths in the treatments

Treatment	**Color 436 nm**	**Color 525 nm**	**Color 620 nm**	**Color 436 nm**	**Color 525 nm**	**Color 620 nm**
	m^{-1}	**m^{-1}**	**m^{-1}**	**% Rem**	**% Rem**	**% Rem**
No Treatment	29,6	23,8	51	0	0	0
Peroxide (1)	21	15,5	27,8	29	88	45
Peroxide (2)	13,6	11,3	36,3	54	91	29
Peroxide (3)	20,6	16,4	44,7	30	87	12
Sulfate (1)	8,13	6,45	27,4	73	95	46
Sulfate (2)	14,9	9,01	21,9	50	93	57
Sulfate (3)	20,6	12,8	27	30	90	47
Fenton (1)	18,5	12,2	11,4	38	90	78
Fenton (2)	4,22	2,48	6,32	86	98	88
Fenton (3)	3,29	1,95	5,44	89	98	89

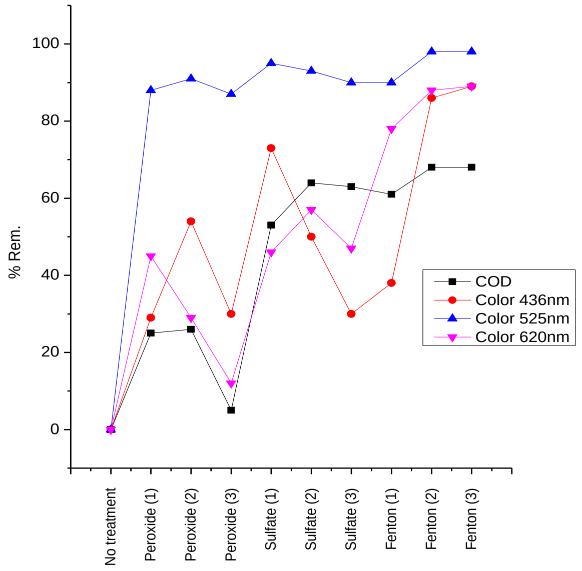

Fig. 23 Percentage of color removal and chemical oxygen demand in each of the treatments

In this case, the pH of the Fenton reaction, which is theoretically 2.8UN, is compared with the neutral pH, and it is evident that the reaction occurs. This result is attributed to some organic acids, such as citric acid, a chelating agent (Fenton modified). These results can be seen in Figure 23.

4. Conclusion

The efficiency of Fenton reactions for wastewater treatment from the textile industry was evaluated using water samples under real conditions. The samples were taken in the homogenization tanks of the industrial wastewater treatment plant of Industrias Printex S.A.S. The color variable was analyzed, finding values of; 5.6148 m^{-1} at wavelengths of 436 nm, 8.1627 m^{-1} at wavelengths of 525 nm, and 5.0651 m^{-1} at wavelengths of 620 nm. Once the Fenton treatment was applied, color removal percentages of up to 100% were found.

It was determined that a concentration of 571 ppm of ferrous sulfate heptahydrate with 38 ppm of peroxide was optimal for the Fenton reaction to produce the best color degradation in wastewater from the dyeing process in Printex S.A.S. After analyzing 30 different combinations of ferrous sulfate heptahydrate and peroxide doses, when performing an analysis of variance - ANOVA, constructing the respective response surface, and optimizing, it is found that after applying said doses, the best color removal was achieved. It is important to keep in mind that the method reports low absorbance values from 200 ppm of ferrous sulfate and 25 ppm of peroxide, leading to lower costs when scaling the process.

The Fenton reaction was compared with the use of gaseous chlorine in the process, finding that the Fenton process is more efficient than the one currently used (chlorine). In terms of DOC removals, according to the values of P ($p < 0.05$), it is found that the tank and treatment factors have a significant influence on the response variable; that is, the DOC varies in each tank and differs in the treatments studied. Therefore, it is shown that there are statistically significant differences at a confidence level of 95.0%. In terms of color degradation, the differences between the two processes are very small since both treatments, when applied, report very low color values; however, treatment with Fenton in most cases shows a better removal of said parameter. This will allow the purification plants to capture water that is easier to treat, and the aquatic fauna and flora will run less risk.

The cost-benefit analysis of the oxidation process was carried out to determine the viability of implementation at the pilot level. However, given that the conditions of the Industrias Printex S.A.S. water treatment plant allow scaling to the plant level, the study of costs in real operating conditions was proposed. Therefore, the scaling was done by treating 100 m^3 of wastewater from the company's dry cleaners, using the minimum doses for the Fenton reaction. Thus, it is economically viable with savings amounting to U SD30 per cubic meter (m^3) of treated water.

Compliance with current environmental regulations for discharges was verified by analyzing the water resulting from the Fenton process. The parameter compared was DOC since it has a maximum value established in current environmental regulations regarding discharges. A DOC of 391 mgL^{-1} was found, and the standard establishes that it should be a maximum of 400 mgL^{-1}. It should be noted that the company has a biological reactor where the water enters after the Fenton treatment, which further reduces the value of this parameter.

The results indicate that since it is water without prior treatment, it has a high content of solids that are partially removed by coagulation with ferrous sulfate heptahydrate, which leads to a % color removal without ignoring that it does lead. A Fenton process is carried out because the % color removal is even higher when applying this treatment and, in turn, reduces the DOC.

In this case, the pH of the Fenton reaction, which is theoretically 2.8UN, is compared with the neutral pH, and it is evident that the reaction occurs; this is attributed to the presence of some organic acids such as citric acid, which is a chelating agent (Fenton modified). The limitations of this study lie in the variations of the water that enters the treatment system because it is a real matrix and the production processes usually present modifications according to the type of dyed garments. This finding modifies the physicochemical and biological characteristics of water. For this reason, from a research perspective, the combination of treatment systems and the application of advanced oxidation processes after conventional treatment processes is proposed; additionally, to explore the application of electrochemical processes that eliminate the use of chemical products in the search for green chemistry.

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