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Reduction of Organic Pollutants Present in Wastewater from the Tanneries of La María in the Department of Quindío Using Photo-Fenton

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Abstract: Wastewater containing toxic organic pollutants does not comply with the legislation; therefore, there is an urgent challenge in developing efficient oxidation processes that ensure its organic elimination to preserve the water quality in the environment. Fenton and photo-Fenton techniques, which are part of advanced oxidation processes, are currently very innovative and effective for decontaminating organic pollutants in industrial wastewater. That is why we have studied the use of the Fenton reagent (Fe^{+2}/ H_2O_2) and photo-Fenton (Fe^{+2}/ H_2O_2 + Ultraviolet) to apply to wastewater produced by the tanneries. For this, 1L water solutions taken after the wastewater treatment plant were prepared, adjusting the pH to 4.0, then the Fenton reagent was added and stirred for 2 hours at 150 rpm, then these same solutions were exposed to ultraviolet light at 269 nm for 2 hours to perform the photo-Fenton process. Finally, it was possible to obtain better decontamination of these effluents with the photo-Fenton process. In turn, the wastewater turbidity decreased by 92%, chemical oxygen demand, total organic carbon, and color also decreased considerably, making this technique sustainable and environmentally friendly to improve the ecosystem and the people's quality of life around these industries.

Keywords: tanneries, Fenton, photo-Fenton, wastewater.

使用照片-芬頓減少金迪奧省瑪麗制革廠廢水中的有機污染物

摘要：含有有毒有機污染物的廢水不符合法規；因此，開發有效的氧化工藝以確保其有機物的消除以保護環境中的水質是一項緊迫的挑戰。芬頓和照片芬頓技術是高級氧化工藝的一部分，目前在淨化工業廢水中的有機污染物方面非常創新和有效。這就是我們研究使用芬頓試劑 (鐵+ 2/過氧化氫) 和光芬頓 (鐵+ 2/過氧化氫 + 紫外線) 來處理制革廠產生的廢水的原因。為此，在污水處理廠後製備 1 大号水溶液，調節酸鹼度至 4.0，然後加入芬頓試劑並以 150 每分鐘轉數攪拌 2 小時，然後將這些相同的溶液暴露於 269 納米儀表的紫外線下 2 幾個小時來執行照片芬頓過程。最後，有可能通過光芬頓工藝對這些流出物進行更好的淨化。反過來，廢水濁度降低了 92%，化學需氧量、總有機碳和顏色也顯著降低，使這項技術可持續和環保，以改善這些行業的生態系統和人們的生活質量。

关键词：制革廠，芬頓，照片芬頓，廢水。

1. Introduction

Fenton process has its origin in the discovery reported in 1894 that ferrous ion strongly promotes the oxidation of tartaric acid by hydrogen peroxide [4]. However, only much later, the oxidation activity has been ascribed to the hydroxyl radical [8]. The mechanism of Fenton's process is quite complex, and some papers can be found in the literature where tens of equations are used for its description [5, 3, and 14]. Nevertheless, it can be summarized by the following steps: first, a mixture of H_2O_2 and ferrous iron in an acidic solution generates the hydroxyl radicals Eq. (1) [2, 16], which will subsequently attack the organic compounds present in the solution.

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

As iron (II) acts as a catalyst, it has to be regenerated, which seems to occur through the following scheme:

$$Fe^{+3} + H_2O_2 \leftrightarrow Fe\text{-}OOH^{+2} + H^{+} \quad (2)$$

$$Fe\text{-}OOH^{+2} \rightarrow Fe^{+2} + HO_2 \quad (3)$$

Oxidation with Fenton's reagent has proven effective and promising for the destruction of several compounds and consequently for the treatment of a wide range of wastewaters, as described in several reviews [1, 6, 9, and 14].

As its name suggests, the photo-Fenton process is similar to the Fenton one but also employs radiation [7, 13, 11, and 14]. Its effectiveness is attributed to the photolysis of Fe(III) cations in acidic media yielding Fe(II) cations Eq. (4), in conjunction with the reaction between Fe(II) and H2O2 to yield hydroxyl radicals Fenton's reaction, step (5):

$$Fe\ (OH) + 2 + hv \rightarrow Fe+2 + OH \quad (4)$$

$$Fe\ +2 + H_2O_2 \rightarrow Fe\ (OH)+2 + OH \quad (5)$$

In this process, the regeneration of Fe^{2+} by photo-reduction of Fe^{3+} Eq. (4) is accelerated. This photo-reduction is an additional source of highly oxidative hydroxyl radicals, compared with the "simple" Fenton's process. As discussed elsewhere, the overall regeneration rate and process efficiency are considerably low in Fenton process [9]. Nevertheless, the importance of Fe^{2+} regeneration through another step - photo-reduction of the photochemical organic substrate or its degradation intermediates, has been pointed out by some authors [15]:

$$Fe^{+3}\ L + hv \rightarrow Fe^{+2} + L^{\cdot}(L=RCOO^{-}, RO^{\cdot},OH) \quad (6)$$

Besides Fe, other transition metals can also catalyze the reactions mentioned above, e.g., copper. The reaction system using Cu as the photo-Fenton catalyst follows a similar network as Fe and is referred to as a photo-Fenton-like reaction [10]. Recently, much attention has been paid to the photo-Fenton and photo-Fenton-like processes, mainly focusing on the heterogeneous process [15]. The above is much related to the disadvantages associated with the homogeneous process, namely the formation at the end of the process of high amounts of metal-containing sludge with high environmental impact and costs associated. In addition, a large amount of the catalytic metals are lost in these sludge. In the opinion of some authors, the costs associated with the processing of these sludge's might limit the further application of the homogeneous photo-Fenton process [10, 12], the reason by which numerous attempts have been made to find heterogeneous systems for the Fe species, as pillared clays, for subsequent use in photo-Fenton-based processes.

Finally, according to the articles mentioned above, the photo-Fenton has greater effectiveness in producing hydroxyl radicals with the help of ultraviolet light, either natural (sunlight) or synthetic, or with the use of UV lamps. This technique has had great relevance in the decontamination of wastewater produced by many industrial effluents, including the tanning industries. So, this research is of vital interest in highlighting advanced oxidation processes relevant for the decontamination of wastewater belonging to tanneries and, in turn, improving the sector and contributing to the improvement of this precious effluent that is vital for life.

2. Materials and Methods

2.1. Reagents and Preparation of Solutions

All reagents used in this work were analytical reagent grade and were used without further purification. Ferric sulfate hexahydrate ($FeSO_4.6H_2O$) 98%, hydrogen peroxide (30% w/w), not stabilized), and H_2SO_4 98% were purchased from Aldrich. All solutions were prepared in ultrapure water (Milli-Q water, Millipore).

2.2. Equipment

A brand METTLER TOLEDO UV-visible spectrophotometer was used to determine color and turbidity, and also a set of jars for the realization of the Fenton with UV lamp couplings (wavelength of 269 nm) for photo-Fenton, another of the equipment used, was the equipment for the determination of total organic carbon (TOC-V CSH brand SHIMADZU).

2.3. Reaction Conditions

All the experiments were carried out using six reactors with continuous stirring (jar test). For the reaction process with the Fenton reagent, the solutions of the wastewaters of the tanneries of La María in the department of Quindío were homogenized, adjusting the pH to 4. Initially, different concentrations of Fe^{+2} and hydrogen peroxide were used.

When the best reaction ranges for Fenton were generated, the photo-Fenton process was subsequently carried out; for this, the solutions were exposed to UV light lamps (wavelength of 269 nm) for 2 hours.

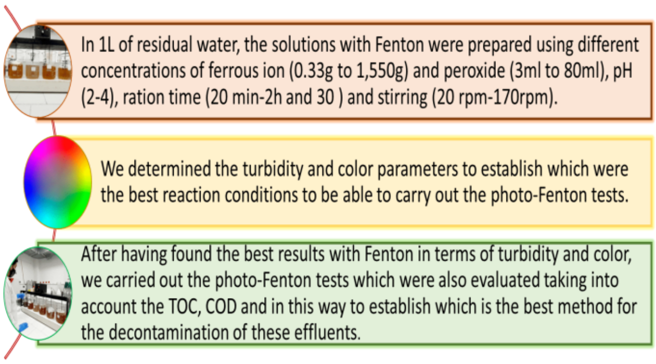

Fig. 1 Methods

3. Results and Discussion

3.1. Fenton Process Results

For the Fenton process, an experimental setup was carried out with a set of jugs containing six reactors in which one liter of wastewater from the tanneries was added, extracted from the tanks located after the WWTP (wastewater treatment plant). Different concentrations of iron and hydrogen peroxide, taken from theoretical backgrounds that had treated similar waters with these methods, made variations in pH, reaction time, and stirring, which were the parameters evaluated for this system, as shown in Fig. 2.

Fig. 2 Experimental setup of the Fenton process using jar equipment

The table below lists the preliminary data for the Fenton process, considering data found in the literature for this type of wastewater. The preliminary results of the removal percentage found in Table 1 were obtained from the following equation.

$$\% \text{ Rem} = (Wo-WF) \times 10 / Wo \qquad (7)$$

where % Rem is the removal percentage, Wo is the original water obtained from the tanneries without treatment, and WF represents the water treated with Fenton; when photo Fenton was used, WPF changed the variable.

Table 1 Preliminary data of Fenton process

Fe^{+2} (g/L)	H_2O_2 (ml)	pH	Time (min)	Agitation rpm	Remain (%)
0.823	42	3	75	21.1761	32.26
0.333	67	4	30	150	86.59
0.823	42	4.5	75	100	65.15
0.823	81	3	75	100	71.92
1.595	42	3	75	100	90.45
0.333	17	2	30	50	91.61
1.313	67	4	30	50	57.71
0.823	42	1.4	75	100	16.85
1.313	67	2	30	50	3.74
1.313	67	2	120	150	87.10
0.823	42	3	75	100	81.81
0.333	17	2	120	50	55.20
0.823	42	3	75	100	39.27
1.313	17	2	30	150	86.30
0.333	67	4	120	50	76.71
1.313	17	4	120	150	89.11
1.313	17	4	120	50	56.73
0.823	42	3	146	100	58.12
0.823	42	3	4.1	100	7.59
0.823	2.2	3	75	100	66.35
0.333	17	4	30	150	75.09
0.823	42	3	75	178	26.32
0.051	42	3	75	100	82.02
0.333	67	2	120	150	84.15

After subjecting the real wastewaters from tanneries to the Fenton reagent, the removal results were obtained for the turbidity specified in the table. From Table 1, the results with better removal greater than 65% were taken, and the experiments with photo-Fenton were carried out using these data.

Next, we can appreciate the results of the preliminary parameters evaluated for Fenton in removing turbidity and color, using the statistical analysis of the response surface.

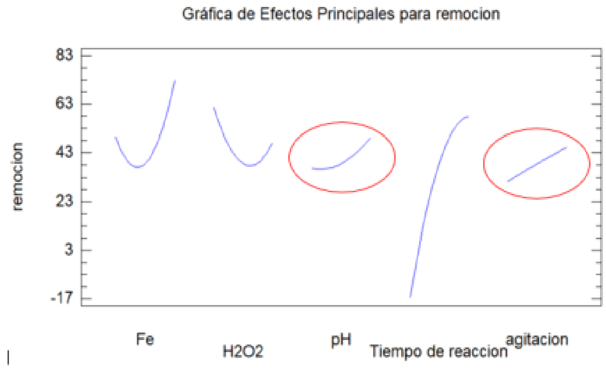

Fig. 3 Effects of removal in preliminaries

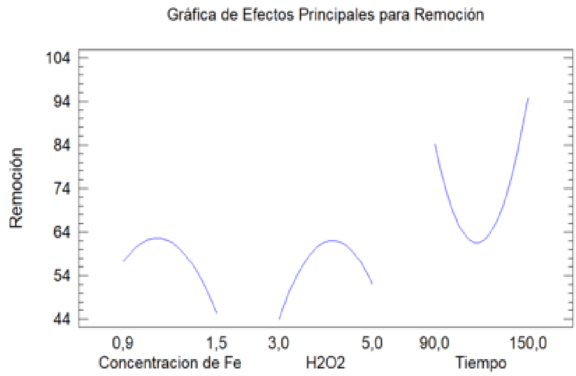

Fig. 4 Main effects of removal with fixed pH and agitation

These preliminary analyzes provided us with important information regarding the parameters evaluated. In Fig. 2, it is evident that both the pH and the agitation do not have significant changes compared to the other variables evaluated. Therefore, for the following analyzes, we leave the pH fixed, and the stirring and only the iron and peroxide concentrations and the reaction time were varied in intervals close to the initial ones, obtaining the results shown below.

For these analyzes, the turbidity and color removal indices were taken as main variables to determine the optimal parameters of Fenton and photo Fenton to evaluate for said wastewater product of the industrial processes of the tanneries. After the preliminary analyses, we can show the optimal values of the concentration of iron, peroxide, and the reaction time, as shown in Fig. 5. Below are the results of the response surface analysis for each of the variables evaluated.

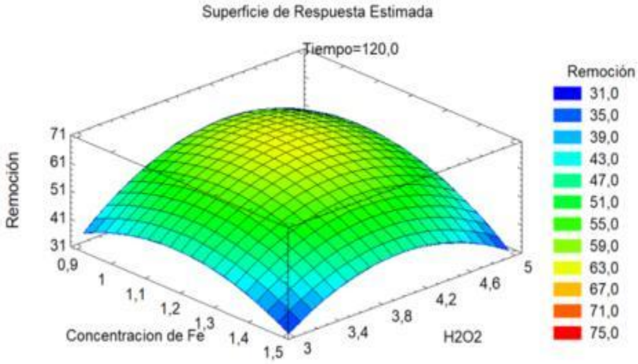

Fig. 5 Response surface of Fe^{+2} concentration vs. H_2O_2

In Fig. 5, we can observe the turbidity removal index for Fenton, obtaining that the optimal concentration of Fe^{+2} is 1.2 g/L and of peroxide – 3 ml/L. It is also shown that the optimal removal time is 2 hours, since when the time increases, the removal rate decreases, as shown in Fig. 4, and the concentrations of Fe^{+2} and H_2O_2.

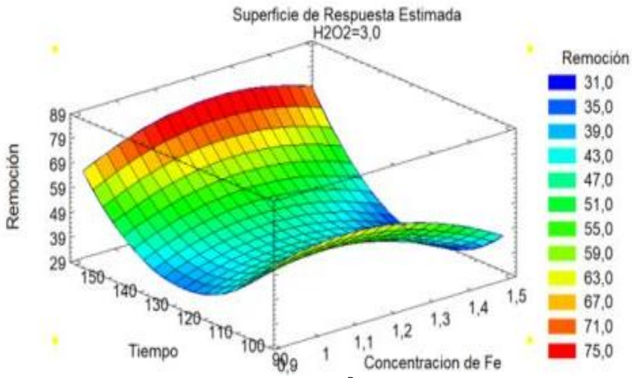

Fig. 6 Response surface of Fe^{+2} concentration vs. time

In Fig. 6, we leave the peroxide concentration constant and observe the behavior of the iron concentration versus time. It can be seen that the iron concentration where the best removal is observed is Fe^{+2} = 1.2g / L, and the best removal time ranges from 2 hours to 2 and a half hours at most; after that time, removal begins to diminish.

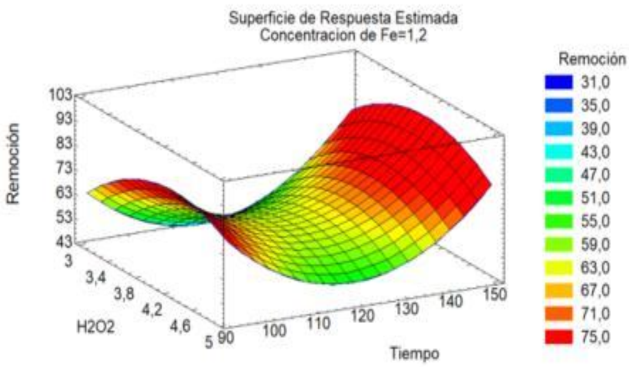

Fig. 7 Response surface of peroxide concentration vs. time

Finally, we evaluate the peroxide concentration versus time, leaving the optimal iron concentration constant (Fig. 7). In this way, it was possible to obtain that in regions where the time oscillates between 2 and 2.5 hours, and the peroxide concentration is 3 ml/L, it is achieved better haze removal.

In the case of color, three colors, yellow, red, and blue, at wavelengths of 436, 525, and 620 nm, were evaluated in the ultraviolet spectrophotometer. The results obtained for each color are analogous to the turbidity analysis, obtaining better color removal at Fe^{+2} concentrations = 1.2 g/L, H_2O_2 concentration = 3 ml/L, stirring times of 2 hours, pH of 4, and stirring of 150rpm. In addition, it was noted that there is a better removal for the blue and red colors and a lower one for yellow since the Fe^{+2} iron turns the solutions a light-yellow color. The removals achieved for these colors exceeded 90%, as evidenced in Table 2.

Table 2 Color results after using Fenton

Samples in triplicate	Original Water	Fenton 1	Fenton 2	Fenton 3	Fenton 4
Wavelengths	436	436	436	436	436
Absorbance	0.546	0.086	0.07	0.08	0.065
Removal (%)		84.249	87.179	85.347	88.095
Wavelengths	525	525	525	525	525
Absorbance	0.311	0.017	0.011	0.014	0.009
Removal (%)		94.533	96.463	95.498	97.106
Wavelengths	620	620	620	620	620
Absorbance	0.191	0.008	0.003	0.005	0.003
Removal (%)		95.811	98.429	97.382	98.429

After performing analyzes for Fenton and obtaining the optimal removal values for each of the evaluated parameters, we performed analyzes for the photo-Fenton process as shown below.

3.2. Photo-Fenton Process

To carry out the photo-Fenton analyzes, those concentrations of Fe^{+2} and hydrogen peroxide that had worked more efficiently in the Fenton process were chosen. The jar equipment assembly was used. A box with ultraviolet light lamps (269 nm wavelength) was attached so that the solutions were irradiated for 2 hours and in this way to compare the removal processes for both experiments. Fig. 8 and 9 show the mounting for photo-Fenton.

Fig. 8 Set of jars with samples for photo-Fenton

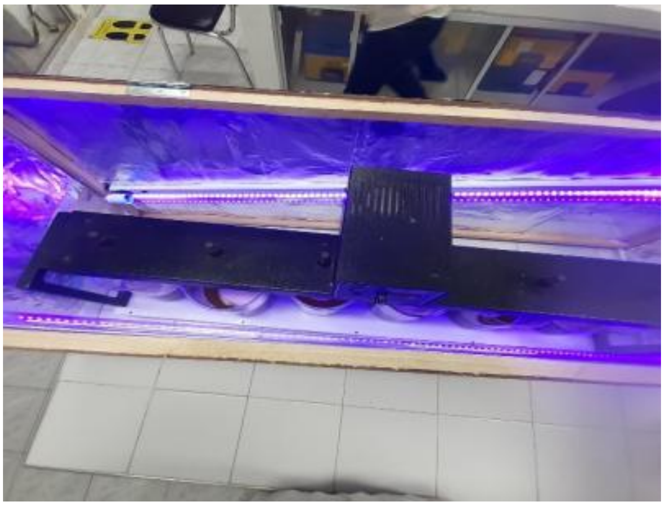

Fig. 9 Experimental setup of the photo-Fenton process

As can be seen in Fig. 8, the assembly consists of a set of jugs with the reactors containing one liter of contaminated water from the tanneries adding the Fenton reagent according to the optimal conditions that resulted in the previous experiment, and the variant is the irradiation of ultraviolet light at 269 nm for 2 hours. After irradiating the samples, we analyzed four parameters to compare the effects of Fenton and photo-Fenton in the different samples evaluated: color, turbidity, total organic carbon (TOC), and chemical oxygen demand (COD), obtaining that the samples treated with photo-Fenton achieved a better removal and reduction of organic contaminants for Fenton as shown below.

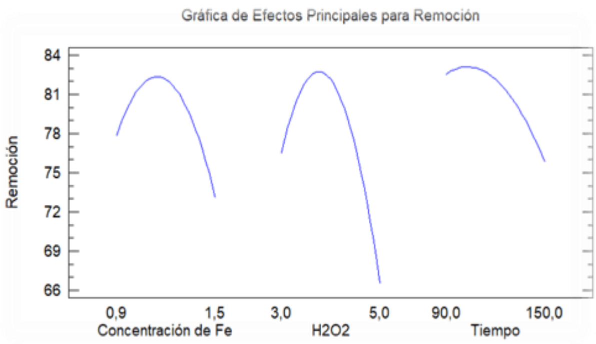

Fig. 10 Effects of removal for photo-Fenton

In the results obtained for photo-Fenton (Fig. 10), we can see, according to the response surface analysis, that the optimal ferrous ion concentration for good removal is 1.2 g/L since, when we increase this concentration, removal begins to decrease. The same happens for the amounts of peroxide: an optimal volume for a good removal is 3 ml, with a maximum of 120 minutes of reaction time.

In Fig. 11, we can observe the effects of the physicochemical parameters evaluated for photo-Fenton more clearly and concisely.

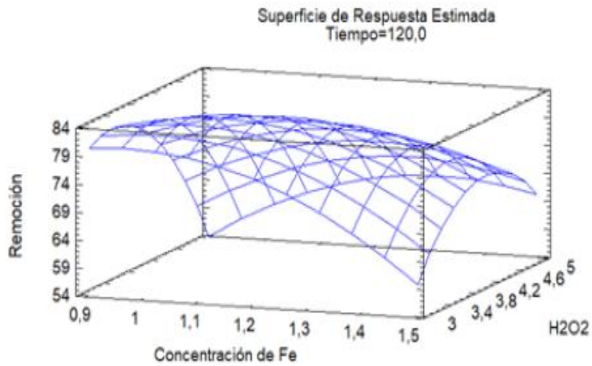

Fig. 11 Turbidity removal results using photo-Fenton

This graph shows the effects of turbidity removal from photo-Fenton treated waters. Through these response surface analyzes, the best concentrations of ferrous ion and peroxide were obtained, as well as the optimal removal time for the turbidity parameter, reaching removals above 90%, exactly a removal of 92%, compared with Fenton that the maximum turbidity removal for this process was 84%.

We also want to show below in Table 3 the results obtained for color, evidenced that there is a better removal using the photo-Fenton process, achieving color removals of more than 100%.

Table 3 Color results after using photo-Fenton

Samples in triplicate	**Original Water**	**Photo F 1**	**Photo F2**	**Photo F3**	**Photo F4**
Wavelengths	436	436	436	436	436
Absorbance	1.388	0.02	0.113	0.033	0.02
Removal (%)		99.998	99.991	99.997	99.998
Wavelengths	525	525	525	525	525
Absorbance	1.034	-0.03	0.045	-0.025	-0.032
Removal (%)		100.002	99.995	100.002	100.003
Wavelengths	620	620	620	620	620
Absorbance	0.868	-0.037	0.028	-0.033	-0.039
Removal (%)		104.262	96.774	103.803	104.493

Finally, we compare the removal of total organic carbon (TOC) and the chemical oxygen demand (COD) for Fenton and photo-Fenton processes as shown in Fig. 5 and 6, in which it is observed that there is a better COT and COD removal for the photo-Fenton process. However, the removal levels were low for both processes due to the interference of some inorganic compounds and ions, which we will deal with in a future study using electrochemical techniques such as electro-photo-Fenton.

Table 4 Comparison of the TOC for the Fenton and photo-Fenton processes

Water	**TOC (mg/L)**	**Removal**
AOF	643.46	
AOFF	645.5	
F1	270.325	57.98
F2	253.6	60.58
F3	260.36	59.53
F4	258.75	59.78
F5	249.56	61.21
F6	238.96	62.86
FOF1	244.9	62.06
FOF2	226.33	64.93

FOF3	222.2	65.57
FOF4	233.55	63.81
FOF5	224.8	65.17
FOF6	218.5	66.15

Notes: AOF - water original Fenton;
AOFF -water original photo-Fenton;
F - Fenton;
FO - photo-Fenton.

Table 5 Comparison of the COD for the Fenton and photo-Fenton processes

Absorbance	**COD**	**Water**
0.156	681.75	FF1
0.157	681.75	FF2
0.158	731.75	FF3
0.159	636.75	FF4
0.16	669.25	FF5
0.161	795.75	FF6
0.162	994.25	COD AOFF
0.163	764.25	F1
0.164	656.75	F2
0.165	671.75	F3
0.166	734.25	F4
0.167	714.25	F5
0.168	796.75	F6
0.169	861.75	COD AOF

Notes: COD AOF - water original Fenton;
COD AOFF: water original photo-Fenton;
F: Fenton;
FF: photo-Fenton.

The following figure compares wastewater before and after having been treated with the photo-Fenton process.

Fig. 12 Wastewater before being treated with the Fenton and photo-Fenton processes

Fig. 13 Wastewater after being treated with the Fenton and photo-Fenton processes

4. Conclusion

The main findings of this study are framed in the contribution that Fenton and photo-Fenton have with the environment, being very friendly techniques due to the minimum cost of reagents and efficient in the removal of organic pollutants. Comparing both techniques, it turns out to be the most significant photo-Fenton in decontamination, showing removals of turbidity greater than 92%, of color equal to 100%, and TOC – 65%.

When comparing the advanced oxidation methods studied in this research, we can conclude that Fenton and photo-Fenton have been studied very little in this type of water produced by tanneries manufacture. Therefore it is an innovative study, and removals were also obtained in 100% color and turbidity not reported in other articles.

The results obtained in this study allow us to conclude that the evaluated techniques turn out to be very efficient in removing organic pollutants in the wastewater produced by tanneries and are also very economical and environmentally friendly methods.

The innovation of this study lies in the type of waters being evaluated, as very good results in terms of removal of organic pollutants and the positive effects on the ecosystem have been obtained when studying these methods.

5. Limitations and Further Study

The study limitation was the removal of COD since there were interferences from inorganic pollutants, and therefore removal of 35% was obtained. To improve this, we want to couple electrochemical techniques such as electro-photo-Fenton.

We can conclude that both advanced oxidation processes turned out to be very relevant and effective for removing organic pollutants in wastewater produced by tanneries, being the photo-Fenton treatment much more efficient. Therefore, it is recommended to use this process for these product effluents of industrial tanning waters. We propose a line of analytical chemistry research where advanced oxidation and electrochemical processes are coupled to optimize the process and obtain better results.

Acknowledgment

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