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Abstract

The textile industry is a field which has strong environmental impacts due to its high consumption of water and emission of pollutants, specially the dyes. The dyes are organic or synthetic substances which alter the physicochemical characteristics of the water body in which they are released, increasing the toxicity, reducing the oxygen and light, intensifying the color and turbidity, and depleting aquatic life. The reduction of the ecotoxicological effects of the subproducts and the efficiency of treatment are points that still need much improvement. Research has shown that most dyes are resistant to conventional treatments, requiring the use of alternative treatments, as advanced oxidation processes. The aim is to evaluate the efficiency of applying the photo-Fenton process, with sunlight as a polishing treatment, on an effluent of a jeans laundry; and to investigate the degradation of the Drimarem red CL-5B dye and its ecotoxicity, with lettuce seeds before and after treatment. A factorial design was used on two levels, varying time, iron and hydrogen peroxide concentrations factors. After the treatment, there was over 90% mineralization of the dye and significant reduction in the evaluated physicochemical parameters were observed, as color, turbidity and total organic carbon; as well as a great reduction in the toxicity of the effluent.

Keywords:
Photo-Fenton Process
Drimaren Red CL-5B
Textile Industry Effluent

1. Introduction

The textile industry is a complex and heterogeneous chain, comprising of the processing and production of natural and synthetic raw materials, of fabrics and of the final product delivered to consumers. The steps of bleaching, dyeing, mercerizing and washing operations consume large volumes of water: about 160 m³ per ton of fiber processed [1]. Consequently, large volumes of waste water and solid waste are generated. The need to treat the effluent waste to avoid damaging the environment, and reduce the water consumption and costs associated with production, makes it imperative to implement water reuse and management practices for this waste [2]. A basic characteristic of textile effluents is the presence of dissolved dyes that were not retained by the fibers, which generates coloration [3-5]. These are identified as the most problematic compounds in textile effluents due to their high solubility in water, organic content, high salt concentration and low degradability [3, 6-13]. The total dye production in the world is estimated at 800,000 tons/year and at least 10-15% of these are discharged into the environment through effluents [8-10].

Highly colored effluents absorb sunlight and hinder the action of photosynthetic algae. Some dyes are recalcitrant and carcinogenic and the conventional biological processes, even when in sequential anaerobic-aerobic routines, do not remove such compounds [6, 7].

The removal of colored substances from the effluent is primarily based on physicochemical methods [14]. There is no universally used method for the treatment of textile effluents, probably because of the variety of chemical structures and complexity of these compounds [15].

Several studies carried out in the past decades have shown that advanced oxidation processes (AOPs) are effective alternatives for the degradation of recalcitrant compounds, usually present in textile effluents. Photochemical processes are used to degrade toxic compounds, producing CO₂ and H₂O without using additional oxidants, as the degradation is assisted by high concentrations of a hydroxyl radical. In this case, the photo excitation of TiO₂ particles, promoted from a valence band to the conduction band, generates the electron/vacancy pair [16]. Among the advanced oxidation processes, the heterogeneous ones with or without irradiation are used in the presence of titanium dioxide, and the homogeneous ones with or without irradiation – of which we highlight the Fenton and photo-Fenton processes. The Fenton reagent is obtained by the decomposition of hydrogen peroxide, catalyzed by the ferrous ion (Fe²⁺) in an acid medium. This decomposition forms hydroxyl radicals (•OH) which react with the pollutants causing their mineralization [17, 18]. The combination of the Fenton reagent with ultra violet radiation results in the enhanced oxidative process known as photo-Fenton. The basic characteristic of this process in relation to the Fenton process is an increased rate of degradation of the pollutants due to the use of UV radiation. The degradation rate increases because a photoreduction of Fe²⁺ to Fe³⁺ occurs as well as the photoysis of hydrogen peroxide, producing more hydroxyl radicals. The mechanism of the photo-Fenton process [19] is presented in the chemical equations (a-e), as follows:

\[
\begin{align*}
Fe^{3+} + 2H_2O & \rightarrow Fe(OH)_{2}^{+} + H_2O^+ \\
Fe(OH)_{2}^{+} + h\nu & \rightarrow Fe^{2+} + OH \\
H_2O_2 & \rightarrow 2\cdot OH \\
Fe^{2+} + OH & \rightarrow Fe^{3+} + H^+ \\
H_2O_2 + OH & \rightarrow H_2O + O_2H
\end{align*}
\]

Knowledge of the photo-Fenton reaction allows promising applications in some industrial practices at a moderate scale, and may be useful in the treatment of industrial waste and effluents [20].

The aim of this study is to evaluate the efficiency of the application of the photo-Fenton process, using sunlight, as a polishing treatment, on an effluent from a jeans laundry at Pernambuco, Brazil; and to investigate the degradation of the Drimarem red CL-5B dye, as well as the ecotoxicity, with lettuce seeds before and after treatment.

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2. Experimental Methods

2.1 Preparation of the Samples

An aqueous solution of 1,000 mg/L of the Drimaren red LC-SB dye (Clariant®) was prepared and used in the degradation study. This dye was chosen because of the demand in the industries of the region where the study was conducted. Fig. 1 shows the structure of the studied dye.

![Fig. 1 Structure of the drimaren red CL-SB dye (1026.41 g mol⁻¹) source: color index number CE: RR 241 [21]](image)

For the ecotoxicity study of the textile effluent subjected to the photo-Fenton process, the effluent of a jeans laundry was used. The sample was collected in polyethylene containers, properly sanitized, conditioned at 5 °C and then subjected to the photo-Fenton treatment.

2.2 Analysis

The aqueous solution of the dye was subjected to photolysis processes using hydrogen peroxide with UV light and the photo-Fenton process. Artificial light was used for the tests with the solution of the dye through the bench reactor (Fig. 2), which has three fluorescent light bulbs with power of 20 W each.

![Fig. 2 Bench reactor with fluorescent bulb](image)

The analytical curve of the dye was carried out in triplicate. The limits of quantification and detection were calculated by LD = 3.3 x (s/s) and LQ = 1.0 x (s/s), where s is the standard deviation of the lowest concentration response and S is the slope (angular coefficient) of the analytical curve [22-24].

The dye degradation was investigated by spectrophotometry (UV-Vis spectrophotometer Thermo Scientific Genesis 105). The samples were analyzed before and after the photo-Fenton treatment. Dilutions were carried out using 30%, 20%, 15%, 10% and 5% of the 1000 mg L⁻¹ solution of the dye. The linear range used for quantification of the dye was from 50 to 300 mg L⁻¹. For the real effluent, its complexity, scanning from 200 to 700 nm was carried out, to evaluate the degradation with emphasis on the reduction of absorption bands. Degradation percentage was determined by Eq. (1):

\[
\text{Degradation} \% = \frac{[Dye]_0 - [Dye]_t}{[Dye]_0} \times 100\%
\]

where, [Dye]₀ and [Dye]ₜ are the concentrations of dye before and after treatment, respectively.

To evaluate the effect of the photo-Fenton process, a 2² factorial design was used to determine the best degradation condition of the aqueous solution of the Drimaren red LC-SB dye. Three variables were chosen in order to verify its influence on the degradation of the dye: the reaction time when exposed to sunlight, the amount of Fe (mg) and the volume of H₂O₂. 25 mL of the aqueous solution were added to a beaker and then tests were carried out according to the plan (Table 1).

The same planning (or design) was used for the real effluent. The volume of hydrogen peroxide (Vetcem®, 30%) added to the sample was estimated from the total organic carbon (TOC) concentration, and the amount of iron was based on the amount determined by the current Brazilian environmental legislation (CONAMA 450/2011) for effluents, which is equal to 15 mg L⁻¹. The aqueous solution of the dye studied had no iron; heptahydrate ferrous sulphate (FeSO₄·7H₂O) was added, P.A – ACS dynamics. Table 1 shows the levels of each variable that was investigated in the factorial design. 

![Table 1 Matrix of the 2² factorial design for evaluate dye degradation using the solar photo-Fenton process](table)

2.3 Toxicity Tests

The acute toxicity of the liquid effluent was estimated through bioassays with lettuce seeds. The lettuce seeds of the variety Lactuca sativa (Lactuca sativa L.) were subjected to the diluted samples from the photolysis process, with concentrations of 1%, 3%, 30% and 100% using as support medium Petri dishes and filter paper [25]. Ten (10) seeds were used for each Petri dish. Subsequently, 5 mL of the effluent were added for each concentration tested in duplicate. Distilled water was used as negative control for toxicity. The results were expressed based on the average growth of roots (cm), Relative Growth Index (RGI) and Germination Index (GI).

The degree of toxicity of the sample was evaluated through the germination index (GI), which is the relation between the average length of the seeds, expressed by the Relative Growth Index (RGI), and the average of seeds that germinated. The RGI is the average growth of the radicle in the trial period. The growth rate of the radicle is given by Eq. (2) and the germination rate is given by Eq. (3) [26].

\[
\text{RGI} = \frac{\text{GRNC}}{\text{SG}}
\]

\[
\text{GI} = \text{RGI} \times \frac{\text{S}}{\text{SG}} \times 100
\]

In the equations, GRNC means the growth of the radicle in the sample and GRNC is the growth of the radicle in the negative control. SG is the number of seeds that germinated in the sample and SG is the number of seeds that germinated in the control. The methodology took into account the number of seeds that germinated and the length of the roots for a specified time. The seeds were incubated for seven days at 20 ± 1 °C. The amount of seeds that underwent protrusion and the elongation of roots were measured, considering germination as a growth equal or superior to 2 mm [27].

3. Results and Discussion

The solution of the dye was submitted to three processes: (1) photolysis; (2) reaction with peroxide and artificial white light; and (3) reaction with peroxide, iron, artificial white light (photo-Fenton) and sunlight. On the other hand, the real effluent, which was collected before the physicochemical treatment used in the industry, and the tests were performed in a similar manner to those employed for the solution of the Drimaren red LC-SB dye, used in the previous tests.

3.1 Study of Degradation of Drimaren Red CL-SB Dye

As in Table 2, the dye solution was characterized according to values of pH, concentration, chemical oxygen demand (COD) and total organic carbon (TOC).

![Table 2 Dye solution characterization](table)

3.2 Photolysis of Drimaren Red CL-SB Dye Solution

Qualitatively, the study observed that there was no color change in the dye solution, discarding photolysis as a treatment for the studied solution. This result is corroborated in the literature, as the direct photolysis of most textile dyes may be difficult or very slow because such compounds are resistant to degradation by exposure to UV radiation [28]. A study of
the photolysis of the red dye, with concentrations ranging between 1x10^4 mol/L and 5x10^4 mol/L, showed that the degradation of the dye decreased with increasing concentration [29]. Here, the concentration of the Drimaden red dye was 8x10^4 mol/L and we didn’t observe degradation. Therefore, the photolysis was inefficient in this case.

3.3 Hydrogen Peroxide and Artificial Light Assay

The effect of the degradation of the dye solution was evaluated in an experiment that used only hydrogen peroxide and light. In this experiment, the reaction time was 60 min. and the amount of peroxide used was 750 µL. The experiment was performed in triplicate, and an average degradation of 9.37% was observed with a standard deviation of 0.58%. The appearance of the samples after the tests also indicated that the process was inefficient, since the color of the solution remained virtually unchanged.

3.4 Photo-Fenton with Artificial Light Assay

Table 3 shows the percentage of degradation and the reduction in TOC for each entry of the experimental assay. The addition of iron salts or iron oxides increased the efficiency of the Fenton or photo-Fenton reaction, because the iron acted as a catalyst and increased the speed of reaction [30]. After the tests, the amount of residual peroxide was measured with colorimetric test strips (peroxide test), which change color according to the amount of peroxide.

Table 3 Results of the mineralization of solution after photo-fenton process with artificial and sunlight

<table>
<thead>
<tr>
<th>Entry</th>
<th>H₂O₂ (µL)</th>
<th>Iron (mg)</th>
<th>Time (min)</th>
<th>TOC (%) (artificial light)</th>
<th>TOC (%) (sunlight)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>900</td>
<td>0.4</td>
<td>90</td>
<td>73.97</td>
<td>91.74</td>
</tr>
<tr>
<td>2</td>
<td>600</td>
<td>0.4</td>
<td>90</td>
<td>74.33</td>
<td>94.12</td>
</tr>
<tr>
<td>3</td>
<td>900</td>
<td>0.2</td>
<td>90</td>
<td>72.85</td>
<td>93.01</td>
</tr>
<tr>
<td>4</td>
<td>600</td>
<td>0.2</td>
<td>90</td>
<td>72.27</td>
<td>87.41</td>
</tr>
<tr>
<td>5</td>
<td>900</td>
<td>0.4</td>
<td>30</td>
<td>51.63</td>
<td>87.76</td>
</tr>
<tr>
<td>6</td>
<td>600</td>
<td>0.4</td>
<td>30</td>
<td>41.07</td>
<td>79.42</td>
</tr>
<tr>
<td>7</td>
<td>900</td>
<td>0.2</td>
<td>30</td>
<td>51.68</td>
<td>73.87</td>
</tr>
<tr>
<td>8</td>
<td>600</td>
<td>0.2</td>
<td>30</td>
<td>59.65</td>
<td>88.91</td>
</tr>
</tbody>
</table>

Fig. 3 shows Pareto’s chart built from data matrix presented in Table 3. None of studied variables showed statistical significant for mineralization, measured by TOC. Therefore, to perform the assays, using the real effluent, we used the values employed in the entry 2 which presented the best result for mineralization.

![Pareto chart for the analysis of the effects of the factors on the mineralization of the dye after treatment of photo-Fenton with artificial light.](image)

Fig. 3 Pareto chart for the analysis of the factors on the mineralization of the dye after treatment of photo-Fenton with artificial light.

3.5 Chemical Oxygen Demand of Aqueous Solution after Treatment Photo-Fenton with Artificial Light

The result of reducing the chemical oxygen demand (COD) was obtained from the experimental condition that presented the best mineralization of the aqueous solution of the dye (Entry 2). The COD values before and after treatment by photo-Fenton process, using artificial light, are shown in Table 4.

Table 4 Results of chemical oxygen demand assay

<table>
<thead>
<tr>
<th>Sample</th>
<th>COD (mg de O₂/L)</th>
<th>Reduction</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before treatment</td>
<td>1763.4</td>
<td></td>
</tr>
<tr>
<td>After treatment</td>
<td>470.00</td>
<td>73.34%</td>
</tr>
</tbody>
</table>

A study using the Mordant Red 73 Azo dye showed that for degradation by photo-Fenton process, the COD reduction was 85% in 180 min of experiment for a dye concentration of 0.1 mM [31]. Herein, when we use a concentration of 0.09 mM of the Drimaren Red LC-5B dye in 90 minutes of experiment, a reduction of 73.34% was achieved for the COD. When used solar photo-Fenton process, the best results presented values higher than 80% for COD. These results are summarized in the Table 5.

Table 5 COD after Solar Photo-Fenton process

<table>
<thead>
<tr>
<th>Entry</th>
<th>COD initial (mg O₂/L)</th>
<th>COD (mg O₂/L)</th>
<th>Reduction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1763.4</td>
<td>237.7</td>
<td>86.52</td>
</tr>
<tr>
<td>2</td>
<td>1763.4</td>
<td>1211.1</td>
<td>31.30</td>
</tr>
<tr>
<td>3</td>
<td>1763.4</td>
<td>256.5</td>
<td>85.45</td>
</tr>
<tr>
<td>4</td>
<td>1763.4</td>
<td>324.0</td>
<td>81.63</td>
</tr>
<tr>
<td>5</td>
<td>1763.4</td>
<td>1014.1</td>
<td>42.50</td>
</tr>
<tr>
<td>6</td>
<td>1763.4</td>
<td>1092.2</td>
<td>38.03</td>
</tr>
<tr>
<td>7</td>
<td>1763.4</td>
<td>346.5</td>
<td>80.35</td>
</tr>
<tr>
<td>8</td>
<td>1763.4</td>
<td>711.5</td>
<td>59.65</td>
</tr>
</tbody>
</table>

These COD values indicate that there is significant reduction after use of solar photo-Fenton process. The reduction in COD was higher in assays of 90 minutes; therefore, time is an important factor in this result, indicating that there was a reduction in the organic matter and increase in the mineralization.

3.6 UV-Vis Spectrophotometry

Fig. 4 shows the UV-Vis spectrum of Drimaren red LC-5B dye solution. The maximum absorption was observed at 545 nm.

![UV-Vis spectrum of Drimaren Red LC-5B dye solution.](image)

Fig. 4 UV-Vis spectrum of the Drimaren red LC-5B dye solution.

Table 6 Degradation assay of the dye solution after 1 h of exposure to peroxide (750 µL) and UV light in the absence of iron

<table>
<thead>
<tr>
<th>Sample</th>
<th>Degradation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>970</td>
</tr>
<tr>
<td>1.2</td>
<td>970</td>
</tr>
<tr>
<td>1.3</td>
<td>870</td>
</tr>
</tbody>
</table>

This result demonstrates that the absence of iron [catalyst] does not allow the degradation of the dye at the maximum time of 90 min. and this is an indication that the chemical structure of the dye was not affected in the treatment with peroxide and UV light only (Fig. 5).

![UV-Vis spectra of the tests performed only with H₂O₂/UV](image)

Fig. 5 UV-Vis spectra of the tests performed only with H₂O₂/UV

After treatment with the photo-Fenton process, using artificial light, the treated solutions were submitted to analysis by UV-Vis spectrometry. Fig. 6 shows these spectra.
Fig. 6 UV-Vis spectra of seven solutions after photo-Fenton process, using artificial light.

Fig. 7 presents an analytical curve that was built to quantify the dye remaining in the solutions. The results are presented in the Table 7.

Table 7: Quantification by spectrophotometry of the degradation of the model effluent after photo-Fenton treatment using artificial and sunlight.

<table>
<thead>
<tr>
<th>Entry</th>
<th>H₂O₂ (µL)</th>
<th>Iron (mg)</th>
<th>Time (min)</th>
<th>Degradation (%)</th>
<th>Artifical light</th>
<th>Sunlight</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>900</td>
<td>0.4</td>
<td>90</td>
<td>61.11</td>
<td>62.77</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>600</td>
<td>0.4</td>
<td>90</td>
<td>59.68</td>
<td>61.46</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>900</td>
<td>0.2</td>
<td>90</td>
<td>59.08</td>
<td>68.49</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>600</td>
<td>0.2</td>
<td>90</td>
<td>59.56</td>
<td>71.94</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>900</td>
<td>0.4</td>
<td>30</td>
<td>58.85</td>
<td>71.58</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>600</td>
<td>0.4</td>
<td>30</td>
<td>56.11</td>
<td>82.18</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>900</td>
<td>0.2</td>
<td>30</td>
<td>55.51</td>
<td>95.15</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>600</td>
<td>0.2</td>
<td>30</td>
<td>49.56</td>
<td>63.01</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 7 Analytical curve (λ = 545 nm) to quantify the degradation dye after photo-Fenton process.

Fig. 8 shows the Pareto chart, indicating the factors that are statistically significant, being the time the most relevant. Also, the interactions among the factors are significant, being the most important the interactions between the amounts of iron and peroxide, and between the amount of peroxide and the time. Only the interaction between peroxide and iron showed no statistical significance (Fig. 8).

3.7 Toxicity of Aqueous Solution after Photo-Fenton Treatment with Artificial Light

The toxicity test with lettuce seeds was conducted in the assay that most mineralized the effluent. The results of germination and average growth of the seeds are expressed in (Table 8).

Table 8: Average number of seeds that germinated and average radicle growth.

<table>
<thead>
<tr>
<th>Seed</th>
<th>H₂O</th>
<th>BT</th>
<th>AT1%</th>
<th>AT3%</th>
<th>AT30%</th>
<th>AT100%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Germination</td>
<td>8.0</td>
<td>7.5</td>
<td>10.0</td>
<td>10.0</td>
<td>10.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Growth radicle</td>
<td>2.2</td>
<td>1.8</td>
<td>2.3</td>
<td>2.1</td>
<td>1.3</td>
<td>0.0</td>
</tr>
</tbody>
</table>

*BT - Before of treatment; **AT - After of treatment.

It is possible to note that germination after treatment, when the effluent concentrations were equal to 1%, 3% and 30%, was higher than that observed for the negative control (distilled water), indicating that the process products do not have enough toxicity to impede the germination of lettuce seeds at the concentrations tested (Table 8). However, when the assay was performed with the crude effluent (100%), no seed germinated, indicating the need for dilution to minimize the toxic effect. Root growth in the effluent before treatment was less than the growth observed for the negative control. Assays containing 1%, 3% and 30% showed a reduction in toxicity regarding the lettuce seeds, since they showed values close to those observed for the negative control. However, the average growth decreased at higher concentrations, indicating that the products – when more concentrated – do interfere in the growth and germination of the seeds. A more detailed analysis was performed based on the relative growth index (RGI) and germination index (GI), described in Eqs.(6) and (7). The graph in Fig. 9 shows that regarding lettuce seeds, there was better germination and greater root growth after treatment of the effluent. The chart also shows that, starting from the concentration of 1%, there is a decrease of both germination and root growth, although at the concentration of 30% germination is considered optimum [32]. However, it is important to note that the effluent when released into the water body undergoes dilution; this resembles the conditions of toxicological tests with effluent concentrations of less than 100% the root growth is similar to the negative control.

Fig. 9 Graph of GI values (%) in relation to RGI

3.8 Results Obtained for the Real Effluent after Treatment with the Photo-Fenton Process with Sunlight

Figs. 10 and 11 show UV/Vis spectra of the samples of the crude effluent and of the assays of the factorial design, respectively. A significant reduction in absorption can be observed after the treatment.

Fig. 10 Absorption spectrum of the raw effluent

Fig. 11 Absorption spectrum of the experimental assays after photo Fenton treatment with sunlight.
al effluent was reevaluated for factors and the interaction of peroxide with time, (C).
To evaluate the effects of the factors and the interaction of the th concentrations, the study observed that the maximum reduction in turbidity reached 98.46%.
3.10 Analysis of Toxicity of the Real Effluent Treated with the Solar Photo-Fenton Process
The real effluent treated with the solar photo-Fenton process showed low toxicity in relation to the bioassays carried out with lettuce seeds. The test showed that the seeds germinated in the treated effluent with the sample in natura as well as with dilutions at concentrations of 30%, 1%, and 1% (Table 10).

Table 10 Average number of seeds that germinate and average root growth (in cm).

![Table 10](image)

The effects of weather, iron and peroxide, as well as the effects of the interaction of the three factors and the interaction of peroxide with time, are statistically significant in relation to mineralization, at a confidence level of 95%. The study showed that the resulting byproducts of the Fenton process are not toxic for the environment, due to the low toxicity observed.

4. Conclusion
Based on this present study, the following conclusions were obtained:
- The photo-Fenton process was effective in changing physicochemical parameters with results of removal of up to 98.46% turbidity and 80.60% of color.
- The toxicity test with lettuce seeds showed high toxicity of the real effluent before the treatment, and a clear increase in the relative growth rate and the germination index after the treatment, demonstrating that the proposed treatment reduced the toxicity of the effluent – this indicated that the resulting by-products were not toxic for the environment, considering the seeds tested. The statistical studies applied to each treatment showed that from the investigated factors, the one that most influenced degradation was time.

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