Removal of Basic Blue 9 Dye by Hydrogen Peroxide Activated by Electrogenerated Fe$^{2+}$/Fe$^{3+}$ and Simultaneous Production of Hydrogen

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Abstract

Electrochemical techniques were used to oxidize organic pollutants by Fenton process using a mix of H$_2$O$_2$ and ferrous ions at a parallel plate reactor. The first stage was to build a micro scale reactor comprising two compartments, cathode and anode, separated by a membrane (Nafion-117). Each compartment has inlets and outlets to allow the flow of fluids (10 Lmin$^{-1}$). The function of the reactor is to oxidize organic pollutants as well as to produce H$_2$. Hydrogen is electrogenerated in the catholyte by the reduction of protons on a carbon steel cathode in acidic medium (0.05 M H$_2$SO$_4$). At the same time, a mixture of Fe$^{2+}$/Fe$^{3+}$ ions is produced in the anolyte (0.05 M Na$_2$SO$_4$, pH ≈ 2) by means of the oxidation of a sacrificial electrode made of stainless steel mesh. Fe$^{2+}$/Fe$^{3+}$ ions interact with H$_2$O$_2$ to generate strong oxidants which are responsible for oxidizing the organic matter and removing color. A voltage of 1 V was applied between the electrodes and remained constant, while the current observed was approximately 0.06 A. Under these conditions, the activation rate with different H$_2$O$_2$ concentrations (15, 20, 25, 30, 35, 40, 45 and 50 mM) was evaluated. The maximum activation rate (1.3 mM$^{-1}$min$^{-1}$) was obtained using 30 mM H$_2$O$_2$. Under these conditions, the oxidation of a synthetic industrial effluent (0.615 mM BB9) was performed and the following results were obtained: 95% of this concentration was removed in 5 minutes and 15 mL...
of H₂ was electrogenerated in 30 minutes.

Keywords
BB9 Degradation, Fenton Process, Hydrogen Peroxide, Hydrogen Production

1. Introduction
Dyes are a class of organic pollutants of aquatic ecosystems coming from the effluents of various industries, such as textiles. Apart from the aesthetic problems related to colored effluents, dyes present in the aquatic environment can reduce light penetration and affect photosynthetic activity and oxygen transfer in the water [1]. Textile processing industries are widespread in developing countries. Among the various processes employed in the textile industry, the dyeing process uses large volumes of water for dyeing, fixing and washing. Textile wastewater causes colouring of the receiving water environment [2]. Textile wastewater can have toxic effects on living organisms in the receiving water, adversely affects the ecosystem, and reduces the assimilative capacity of the environment. There are many processes to remove dyes from coloured effluents, some of which are applied in industry [3]. Electrochemistry offers promising approaches for the prevention of pollution problems in the process industry. For example; Özcan et al., (2009) [4] reported the degradation of a cationic dye, in aqueous solution by using an indirect electrochemical method, electro-Fenton process and carbon sponge and carbon felt cathodes. Moreover, Daneshvar et al., (2006) [5] used electrocoagulation for decolorizing dyes. Navarro et al., 2010 and Figueroa et al., 2009 [6] [7] used H₂O₂.

Hydrogen peroxide is a versatile and environmentally friendly oxidizing agent. It is considered to be an environmentally friendly chemical, since it leaves no hazardous residues and other oxidants. Its oxidant property is due to its high content of active oxygen [8]. H₂O₂ demand has increased in applications such as paper bleaching and industrial water treatment. Many studies have reported the efficiency of H₂O₂ as an oxidant of organic pollutants in presence of Fe²⁺ ions (Fenton process) [9]-[11]. Fenton oxidation processes, both conventional and electrochemical, have been widely applied to detoxifying hazardous organic compounds. The reaction between H₂O₂ and Fe²⁺ produces hydroxyl radical, a non-selective strong oxidant [12]-[14], as shown in equations (1) and (2). In the conventional Fenton process, both H₂O₂ and Fe²⁺ are externally applied, whereas in the electrochemical process, H₂O₂ and Fe²⁺ can be electrochemically generated on site, either separately or concurrently [7] [15].

\[
\begin{align*}
    H_2O_2 + Fe^{2+} &\rightarrow Fe^{3+} + OH^+ + H^+ \\
    H_2O_2 + Fe^{3+} &\rightarrow Fe^{2+} + HO_2^- + H^+
\end{align*}
\]

Hydrogen shows a great potential as a sustainable fuel option. The production of pure H₂ at an acceptable cost is a necessary requirement for hydrogen-based economy [16]. Hydrogen can be produced using different technologies such as chemical reactions, electrochemical reactions, fotoelectrochemical reactions, biological and thermal processes [17]-[19]. Water electrolysis is one of the important technologies for the generation of pure hydrogen [16].

The electrolysis of water is primarily carried out in acidic medium with compartments separated by a cationic exchange membrane. H₂ generation from water electrolysis is probably one of the most simple and direct electrochemical methods [20]. It is an economically promising alternative of clean energy generation when the anodic reaction is replaced by the energetically more convenient electroproduction of Fe²⁺/Fe³⁺ in presence of H₂O₂ to oxidize synthetic textile effluents [6]. The electrogeneration of Fe²⁺/Fe³⁺ ions in presence of H₂O₂ activates H₂O₂ in an industrial effluent and produces free radicals which degrade the organic matter. Hydrogen peroxide is an oxidant capable of decolorating organic molecules and once it is consumed, only water is produced, without byproducts [21]-[23]. Using this approach, it is possible to decolorize industrial wastewater and reduce organic matter, which is an indicator of mineralization of soluble organic pollutants.

2. Materials and Methods
The electrochemical model used assumed that the electrochemical reactor constantly works in a steady state and
the main electrochemical oxidation and reduction reactions involved in the process are those shown in Figure 1. In this figure, the electroproduction of Fe ions in acidic medium in presence of H$_2$O$_2$ can be operated to produce a very strong oxidant and water electrolysis can be represented by two independent electrochemical reactions carried out simultaneously in the electrodes according to the following equations [11] [12] [24]:

\[
\text{Cathode: } 2\text{H}_2\text{O}^+ + 2e^- \rightarrow 2\text{H}_2 + \text{H}_2\text{O} \\
\text{Anode: } 3\text{H}_2\text{O} - 2e^- \rightarrow 2\text{H}_2\text{O}^+ + 1/2\text{O}_2 \\
\text{Net Reaction: } \text{H}_2\text{O} \rightarrow \text{H}_2 + 1/2\text{O}_2
\]

(3)  
(4)  
(5)

Hydrogen and oxygen electrogeneration in acidic medium are represented in Equations (3) and (4), where two pieces of material immersed in an electrolyte are needed to exchange electrons with the electroactive species (H$_2$O$_2$ in the cathode and H$_2$O in the anode) [25] [26].

First, the hydraulic circuit of the electrochemical reactor was constructed, consisting of 2 columns containing the anolyte and the catholyte (1000 mL volume), pumps to send the flow to the reactor and a power source as can be seen in Figure 2. The electrochemical cell was constituted by four acrylic blocks (12 × 12 × 1 cm); the

![Figure 1. Theoretical electrochemical reactions. In the anolyte, oxidation of organic matter and in the catholyte, electrogeneration of hydrogen.](image1)

![Figure 2. Hydraulic system coupled to a parallel plate electrochemical reactor. The columns contain the electrolytes recirculated within the electrochemical cell by means of pumps.](image2)
internal blocks being adapted to form the anode and cathode compartments in which the anode and cathode were placed, respectively. Both compartments were separated by a Nafion-117 membrane in order to prevent the mixture of the anolyte and catholyte fluids in the cell. However, because of the membrane’s permeable capacity to cations, migration of protons from the anolyte to the catholyte compartment was permitted. Inlets and outlets were included in the compartments to allow the flow of fluids, and an electrode feeder was installed for connection to the power supply as shown in Figure 3.

The electrodes (cathode and anode) were made of carbon steel mesh because it is inexpensive and the oxygen layers of this material make it chemical and temperature resistant. However, when this material is in contact with water, electrochemical corrosion problems occur because of the ionic conductivity of the medium. To prevent this, a zinc coating is applied which is removed prior to the use of this material in the electrolysis. This treatment consists of immersion in a bath of 1M H2SO4, during 3 minutes. The cation permeable membrane (Nafion®) is composed of negative charges and only allows the transfer of cations (Fe²⁺, H⁺). The cationic membrane divides the components of the electrochemical cell avoiding thus the mixture of the catholyte and anolyte fluids. The catholyte was composed by 1000 mL of 1 M H2SO4, and the anolyte was composed by 1000 mL of 0.05 M Na2SO4 (Ph ≈ 2). A known concentration of H2O2 was added to the anolyte to determine the H2O2 reaction rate. Different concentrations were evaluated (15 mM, 20 mM, 25 mM, 30 mM, 35 mM, 40 mM, 45 mM and 50 mM H2O2) to determine the concentration at which the reaction rate is fastest. Therefore, a further evaluation of the degradation of the dye can be carried out. H2O2 concentration was determined by permanganometry.

H2 electrogeneration was carried out in the catholyte, where the column has a hole at the top with a hose leading to the input of a measuring tube as shown in Figure 4. The anolyte side was completely symmetrical, without a graduated cylinder for collecting gas. During H2O2 activation, electrogenerated H2 is stored at ambient pressure in a 50 mL graduated cylinder according to the following Equation (6) to (8) [16]:

\[
\text{Cathode: } 4\text{H}^+ + 4\text{e}^- \rightarrow 2\text{H}_2 \tag{6}
\]

\[
\text{Anode: } 2\text{H}_2\text{O} \rightarrow \text{O}_2 + 4\text{H}^+ + 4\text{e}^- \tag{7}
\]

\[
\text{Net Reaction: } 2\text{H}_2\text{O} \rightarrow \text{O}_2 + 2\text{H}_2 \tag{8}
\]

Synthetic wastewater was used for removal of dye Blue Basic 9. It was prepared using the dye Blue Basic 9 (distributed by Ciba Specialty Chemicals) (IUPAC: [7-(dimethylamino)phenothiazin-3-ylidene]-dimethylazan-ium; chloride) to evaluate the electro-Fenton oxidation. The anolyte was prepared in synthetic wastewater with a known BB9 concentration. 0.05 M Na2SO4 and 1 M H2SO4 were added to obtain a pH ≈ 2. The discoloration was observed by absorbance in a Thermo Scientific Genesys 10UV spectrophotometer according to the Lambert-Beer law. During the electrolysis, 3 mL of sample were taken every minute to measure its absorbance in the spectrophotometer. A calibration curve procedure was carried out before the BB9 concentration was ob-
The energy required to electrogenerate H₂ and Fe²⁺/Fe³⁺ ions in an acidic medium essentially depends on the electrode material and electrolyte. Other kinetic parameters, such as the ease of electron exchange between the material and dissolved electroactive species, and the number of steps to transform the electroactive species, are also relevant. The energy required for electrolysis was calculated according to Equation (9).

\[
E_{\text{req}} = \left( \frac{E_{\text{cell}}(I_{\text{cell}})(t_{\text{electrolysis}})}{V} \right)
\]

where: \( E_{\text{cell}} \) is the voltage in the cell (Volts), \( I_{\text{cell}} \) is the current (ampere), \( t_{\text{electrolysis}} \) is the time of the electrolysis (hours) and \( V \) is the volume of the catholyte in L (for the production of H₂) or of the anolyte (for the wastewater treatment) in Wh·L⁻¹ or KWh·m⁻³ (Navarro et al. 2010, Figueroa et al. 2009). The energy required is directly proportional to the cell voltage during the production of hydrogen by water electrolysis [16].

3. Results and Discussion

The first stage of this work was the construction of the electrochemical reactor, the hydraulic system and the electrodes, as shown in Figure 2 and Figure 3. The electrochemical reactor has two compartments: one for the anolyte and the other for the catholyte.

The electrodes used were made of carbon steel mesh obtained from a commercial galvanized mesh. This material was cut to the appropriate size for the electrochemical reactor. The Zn coating of the commercial mesh carbon steel delays Fe oxidation, but Zn metallic surface makes it complicated to obtain Fe²⁺/Fe³⁺ ions in the anode and hinders the reduction of protons to obtain H₂ in the cathode. Considering that metals react immediately with acids to form oxides and hydrogen, the electrodes were pretreated with 1 M H₂SO₄, in order to remove the protective zinc coat and thus expose the carbon steel. However, a prolonged contact time between carbon steel and 1 M H₂SO₄ could oxidize Fe and form a protective layer (passive). Through experimental results verified with electron microscopy studies, a 3-minute contact time was found to be optimum. Figure 5(a) shows the carbon steel mesh without treatment and Figure 5(b) shows the carbon steel mesh with a 3-minute treatment.
Figure 5. (a) Carbon steel mesh without treatment; (b) Carbon steel mesh with a 3-minute treatment (bath in 1 M H2SO4).

3.1. Activation of H2O2

The electrogeneration of Fe2+/Fe3+ ions in presence of H2O2 activates H2O2 in a textile effluent and produces free radicals which degrade the organic matter [27]. Fenton chemistry is based on the activation of H2O2 by Fe2+/Fe3+ ions. Thus, H2O2 is almost entirely stoichiometrically consumed to produce a strong oxidant. A voltage of 1V was applied in the electrochemical cell between the electrodes, which remained constant, while the electric current was 0.06A. Under these conditions, the electrogeneration of Fe3+/Fe2+ ions in presence of different concentrations of H2O2 was determined [28]. Eight different concentrations of H2O2 were evaluated (15, 20, 25, 30, 35, 40, 45 and 50 mM) and Figure 6 shows H2O2 activation as a function of time. Each H2O2 concentration presented different activation rates (slope of the curve). The fastest activation rate was found using a concentration of 30 mM H2O2. An activation rate reduction was observed with higher H2O2 concentrations (Figure 7). Once the oxidant (H2O2) is consumed, there is no production of byproducts, only water [4] [29].

The energy required to electrogenerate Fe2+/Fe3+ ions in acidic medium depends mainly on the material of the electrodes and the electrolyte. The anode oxidation rate is important to initiate the reduction of the protons on the catholyte side. The energy required for electrolysis is shown in Figure 7. A turning point is observed at a concentration of 30 mM H2O2. This behavior has been already reported by Navarro et al., (2010) [6] who suggest that the turning point is an indicator of optimum conditions in the electrogeneration process.

3.2. Oxidation of a Synthetic Effluent

This technology uses the Fenton reaction as the source of hydroxyl radicals, in which the Fenton reagent, a mixture of H2O2 and Fe2+, is electrochemically produced and regenerated throughout the process. The formed hydroxyl radicals lead to the oxidative degradation of BB9 (C16H18N3SCl) until its total mineralization [4].

According to Equation (10), one mole of H2O2 can accept two moles of electrons from the organic matter available to be oxidized, ranging from a highly selective oxidation in organic electrosynthesis to a mineralization of organic matter. The total mineralization of the BB9 dye is very complex due to the presence of the nitrogen, sulfur and chlorine atoms contained in its molecule. BB9 should lose 78 electrons during its mineralization according to Equation (11). Considering Equations (10) and (11), the theoretical quantity of H2O2 needed to mineralize BB9 can be calculated according to Equation (12). BB9 color is caused by nitrogen double bonds in the...
molecule.

$$\text{H}_2\text{O}_2 + 2\text{H}^+ + 2e^- \rightarrow \text{H}_2\text{O}$$  \hspace{1cm} (10)

$$\text{C}_{16}\text{H}_{18}\text{N}_3\text{SCl} + 36\text{H}_2\text{O} - 78e^- \rightarrow 16\text{CO}_2 + 78\text{H}^+ + 2\text{H}_2\text{SO}_4 + 3\text{NH}_3 + \text{HCl}$$  \hspace{1cm} (11)

$$\text{C}_{16}\text{H}_{18}\text{N}_3\text{SCl} + 39\text{H}_2\text{O}_2 \rightarrow 16\text{CO}_2 + 2\text{H}_2\text{SO}_4 + 3\text{NH}_3 + \text{HCl} + 42\text{H}_2\text{O}$$  \hspace{1cm} (12)

To evaluate the oxidation of an industrial effluent using the Fenton process, a synthetic effluent was prepared with BB9. Because the electrolysis were carried out with different H$_2$O$_2$ concentrations, different reaction rates were obtained. The fastest reaction rate obtained was 1.3 mM$^{-1}$min$^{-1}$ with 30 mM H$_2$O$_2$. The removal of the dye BB9 was continued using this H$_2$O$_2$ concentration. Stoichiometric calculations were made (considering 30 mM H$_2$O$_2$) according to Equation (10) and (12), and the following result was obtained: 39 mM H$_2$O$_2$ are needed to eliminate 1 mM BB9, therefore 30 mM H$_2$O$_2$ can eliminate up to 0.77 mM BB9, but only an 80% removal was considered (0.615 mM BB9).

The discoloration was spectrophotometrically monitored. The water was considered discolored when an absorbance ≤5% of the original value was achieved at the catholyte. A calibration curve was prepared in order to establish a relationship between absorbance and BB9 concentration. Several tests were conducted to determine the time corresponding to the desired discoloration. Hydroxyl radicals play an important role because the accu-
mulation of •OH radicals on the surface subsequently leads to the completion of the oxidation of organic species [30].

Figure 8 shows BB9 discoloration behavior. The spectrum shows the absorbance vs wavelength changes as a function of time. Maximum absorbance was found at $\lambda_{\text{max}} = 665$ nm. Samples were taken every 10 minutes, a) 0 min, b) 10 min, c) 20 min, and d) 30 min. A 98% discoloration was observed with a 30-minute electrolysis time. However, degradation rate was faster during the first five minutes achieving up to 95% of the total decoloration. The removal of Basic Blue 3 dye is reported using very similar characteristics [1] with a diamond anode, where an efficiency of 100% color removal in 30 minutes is obtained, utilizing lower dye concentrations.

Table 1 presents BB9 degradation behavior during the first 5 minutes of electrolysis. This table shows the reduction of BB9 concentration (mM, mg L$^{-1}$) and removal efficiency (%) as a function of time and charge (30 mM H$_2$O$_2$ and 0.615 mM H$_2$O$_2$ BB9). These results were satisfactory, since over 90% of the dye was removed in a short time and with a small charge. During the first five minutes of electrolysis, BB9 concentration in the reactor decreased from 0.615 mM to 0.028 mM, which indicates that up to 95% of the initial concentration was eliminated.

Since H$_2$O$_2$ activation rate was 1.3 mM min$^{-1}$, it can be deduced that in 5 minutes 6.5 mM H$_2$O$_2$ were activated. Theoretically, this quantity is needed to remove 0.587 mM BB9. It is important to mention that the time of the electrolysis was prolonged to 30 min, time needed to activate 30 mM H$_2$O$_2$, but the discoloration was not complete. In this case, it was not possible to predict the dye removal because H$_2$O$_2$ activation ends in 30 minutes; however, when the colorant is added to the anolyte, it is removed in the first 5 minutes. Furthermore, the pres-

![Figure 8. Absorbance spectrum for a solution containing 0.061 mM basic blue 9 in 0.05 M Na$_2$SO$_4$; (a) deep blue, 0 min; (b) 10 min, (c) 20 min and (d) 30 min.](image)

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>Charge (C)</th>
<th>Abs</th>
<th>BB9 (C$<em>{16}$H$</em>{18}$N$_3$SCl)</th>
<th>Removal</th>
</tr>
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<tr>
<td></td>
<td></td>
<td></td>
<td>mM</td>
<td>mg L$^{-1}$</td>
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<tr>
<td>0</td>
<td>0</td>
<td>29.99</td>
<td>0.615</td>
<td>195.1</td>
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<td>1</td>
<td>3.6</td>
<td>23</td>
<td>0.46</td>
<td>147.1</td>
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<td>17</td>
<td>0.35</td>
<td>111.9</td>
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<td>3</td>
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<td>14.4</td>
<td>7</td>
<td>0.14</td>
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<td>18</td>
<td>1.4</td>
<td>0.028</td>
<td>8.9</td>
</tr>
</tbody>
</table>
ence of Fe^{2+}/Fe^{3+} ions in the final effluent is not considered toxic as other authors have reported [31]-[33]. Chemistry oxygen demand (COD) was used to determine the amount of organic compounds. A 25% reduction was obtained at 5 minutes and an 80% reduction was obtained at 30 minutes. Dye removal is due to the role of •OH radicals. Xu et al. 2013 [34] mention that H_2O_2 decomposition forms •OH radical and, particularly, the addition of Fe^{2+} facilitates the transformation of H_2O_2 to •OH radical.

These results show that the treatment is both efficient and economical. A 5-minute electrolysis permits to remove 95% of the dye with a 25% COD drop. After 30 minutes, a 100% dye removal is observed, together with an 80% COD reduction. Thus, 30 minutes are sufficient to degrade BB9 organic molecule and the energy required is 0.03 Wh∙L^{-1}. Özcan et al., (2009) [4] removed up to 90% BB3 in 8 hours using a 300 mA current under very similar conditions.

### 3.3. H_2 Generation

This study describes a new reactor concept enabling water-gas shift reaction and the separation of the generated hydrogen in one process step by using electrical energy. When H_2 is obtained from water electrolysis, an electric current passes between two electrodes immersed in a slightly acidic solution. Then, the energy from the electrical current breaks the water molecule in order to obtain hydrogen and oxygen. Figure 1 shows, the electrochemical reactions that are carried out in the electrochemical reactor. In the anolyte, a mixture of Fe^{2+}/Fe^{3+} ions is electrogenerated to activate various H_2O_2 concentrations. Moreover, H_2 is electrogenerated during H_2O_2 activation in the catholyte. Figure 4 shows the catholyte side of electrochemical reactor. The anolyte side is a mirror image but without a graduated cylinder to collect gas. H_2 electrogenerated is stored at ambient pressure in a graduated cylinder (50 mL). During H_2O_2 activation and H_2 electrogeneration, the current is practically the same; therefore, H_2O_2 generation only depends on the electrolysis time: the longer H_2O_2 activation lasts, the larger H_2 production is.

Figure 9 shows data regarding H_2 generation, and 30 mM H_2O_2 activation. It presents a linear trend. It is observed that 15 mL of H_2 were produced in 30 minutes. The technological feasibility and the optimization of hydrogen production depend on several parameters that can be considered under two aspects: the thermodynamics and kinetics of the electrochemical reaction. It can be seen that H_2 production is a linear function of the electrical charge passed. Using the same electrolite but with a different electrochemical cell and a greater voltage and charge, Navarro et al., (2010) [6] reported a production of almost 1600 mL of H_2 in 200 min of electrolysis.

### 4. Conclusions

This process uses the Fenton reaction as the source of hydroxyl radicals, in which the Fenton reagent is a mix-
ture of H$_2$O$_2$ and Fe$^{2+}$. The formed hydroxyl radicals lead to BB9 oxidative degradation until its total mineralization. The electrochemical reactor has two compartments where electrochemical reactions are carried out. In the catholyte, H$_2$ is electrogenerated, which can be visualized as energy; meanwhile, Fenton chemistry is applied to treat synthetic textile effluents in the anolyte through the electrogeneration of a mixture of Fe$^{2+}$/Fe$^{3+}$ ions in presence of H$_2$O$_2$.

The catholyte consisted of 1000 mL of 1 M H$_2$SO$_4$, and the anolyte consisted of 1000 mL of 0.05 M Na$_2$SO$_4$ (pH $\approx$ 2). The electrodes were made of carbon steel mesh. Under these conditions, many electrolysis tests were carried out and the activation rate was evaluated for various H$_2$O$_2$ concentrations (15, 20, 25, 30, 35, 40, 45, and 50 mM). The highest activation rate was 1.3 mM$\cdot$min$^{-1}$, corresponding to a concentration of 30 mM H$_2$O$_2$. The energy required was 0.03 Wh$\cdot$L$^{-1}$ and the optimum time was 30 minutes. In this time, 15 mL of H$_2$ was electrogenerated. The experimental results showed the feasibility of the reactor concept, as H$_2$ was generated at the cathode according to Faraday’s Law.

Using the optimum concentration found in this study (30 mM H$_2$O$_2$), an evaluation of the oxidation was carried out, in which synthetic industrial effluent (0.615 mM BB9) was used. Five minutes after the start of the electrolysis, 95% of the BB9 concentration was eliminated. According to this result, textile effluent treatment was environmentally compatible, fast and efficient, from a chemical point of view.

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