

# A Comparative Study of the Degradation of the Erionyl Navy R by Different Oxidation Processes: Chemical, Fenton and Fenton-like

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## Abstract

The oxidative degradation performance of the Erionyl Navy R dye was studied in this article. The investigation mainly focused on a comparative study between chemical oxidations by sodium hypochlorite (NaClO) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), and catalytic oxidations including the Fenton (Fe<sup>2+</sup>-H<sub>2</sub>O<sub>2</sub>) and Fenton-Like (Fe<sup>2+</sup>/ Fe<sup>3+</sup>/Co<sup>2+</sup>/ Mn<sup>2+</sup>-H<sub>2</sub>O<sub>2</sub>) or modified Fenton-like (Fe<sup>2+</sup>/ Fe<sup>3+</sup> -NaClO) reactions. A discoloration and degradation of the Erionyl Navy R occurred after 30 minutes, which varies according to the oxidation system involved; 31%, 54%, <20%, 95%, and >96% losses were observed for Co<sup>2+</sup>-H<sub>2</sub>O<sub>2</sub>, Mn<sup>2+</sup>-H<sub>2</sub>O<sub>2</sub>, Fe<sup>2+</sup>-NaClO, Fe<sup>3+</sup>-NaClO, and Fe<sup>2+</sup>-H<sub>2</sub>O<sub>2</sub> and Fe<sup>3+</sup>-H<sub>2</sub>O<sub>2</sub>, respectively.

**Keywords:** Erionyl Navy R, Sodium hypochlorite, Hydrogen peroxide, Fenton, Fenton-like

## 1. Introduction

The amount of wastewater discharged by the textile industry into the environment is enormous, making water recovery a necessity[1]. Today, all the pollutants in this sector can be treated using conventional methods, except for the dye, which is not easily biodegradable and very toxic to the aquatic ecosystem[2]. Approximately 280,000 tons of textile dyes are discharged into wastewater every year[3]. Textile wastewater, which accounts for 20% of industrial wastewater [4], contains a wide range of synthetic dyes with a concentration of about 300 mg/L[5].

Textile waste can be treated using physical, chemical or biological techniques. While biological processes are sometimes preferred because they are low cost, the textile rejects contain a high concentration of synthetic dyes, and the results are not always satisfactory[6]. In most research papers published on dye removal, physical-chemical techniques have been used to improve results, and adsorption and advanced oxidation processes are the most widely studied methods[7]. Practically, oxidation by ozone is one of the most widely used oxidation methods[8]. There are, however, other viable alternatives to ozonation, such as catalytic oxidation in the Fenton type of process[9] and chemical oxidation by sodium hypochlorite[10] which continue to gain

importance in the treatment of colored water.

The purpose of this study was to examine the chemical oxidation effects of some cheap oxidizing products such as sodium hypochlorite and hydrogen peroxide, as well as to compare the mineralization of Erionyl Navy R dye in aqueous solutions, compared with the catalytic oxidation with Fenton reagents on the one hand, and modified Fenton on the other, by using catalysts other than Fe<sup>2+</sup> such as Fe<sup>3+</sup>, Co<sup>2+</sup> and Mn<sup>2+</sup>. Therefore, the results of this study will lead to a better understanding of the oxidation mechanism and thus design the best operating conditions for the treatment of acid dyes at specific sites.

## 2. Materials and methods

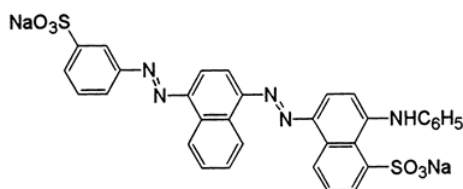
### 2.1. Chemicals and reagents

The Erionyl Navy R dye (C<sub>32</sub>H<sub>21</sub>N<sub>5</sub>Na<sub>2</sub>O<sub>6</sub>S<sub>2</sub>) was supplied by the Sarl Aldrich Lab, Algeria. The main characteristics of the dye studied (Figure 1) are presented in Table 1. The various experimental conditions carried out as part of this study required the following analytical quality products: Chemical oxidants: Sodium hypochlorite 16° (NaClO), Hydrogen peroxide 10V (H<sub>2</sub>O<sub>2</sub>). Catalysts: Iron sulfate (FeSO<sub>4</sub>, 7H<sub>2</sub>O), Cobalt chloride (CoCl<sub>2</sub>, 6H<sub>2</sub>O), Manganese chloride (MnCl<sub>2</sub>, 4H<sub>2</sub>O), Ferric chloride (FeCl<sub>3</sub>), and for pH adjustment of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) solutions. All chemicals were purchased from the Sarl Aldrich Lab, Algeria.

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**Table 1. Main Characteristics of the Erionyl Navy R Dye**

|                          |  |
|--------------------------|--|
| Commercial name          | Erionyl Navy R   |
| Manufacturer             | CIBA   |
| CAS number               | 3351-05-1  |
| Colour Index             | 26360  |
| Molecular Weight (g/mol) | 681  |
| $\lambda_{\max}$ (nm)    | 565  |
| Purity (%)               | 50   |
| Linear formula           | $C_{32}H_{21}N_5Na_2O_6S_2$  |
| IUPAC name               | disodium;8-anilino-5-[[4-[(3-sulfonatophenyl)diazenyl]naphthalen-1-yl]diazenyl]naphthalene-1-sulfonate |

**Figure 1. Chemical structure of Erionyl Navy R dye.**

## 2.2. Experimental method

All the Erionyl Navy R degradation experiments were carried out on batch mode in 250 ml beakers at a volume of 100 ml, under magnetic stirring at 550 rpm and at a temperature of 297 K°. The concentration of the colored solution was 0.0147 mmol/L prepared by dilution of a stock solution and the pH was adjusted to a value equal to 3 by the addition of some drops of 0.1 M sulfuric acid.

Chemical oxidation experiments are performed in the presence of different oxidant concentrations. For hydrogen peroxide the concentration varies from 1.8 to 7.1 mmol/L and for sodium hypochlorite it varies from 1.4 to 7.1 mmol/L. Moreover, for catalyzing oxidation experiments, the quantity of each catalyst used is added first, with stirring, and then the oxidant is introduced to initiate the radical degradation process.

## 2.3. Analysis

The reaction is monitored by spectrum-photometric analyses of a sample of the reaction mixture taken at determined time intervals, at the maximum wavelength of 681 nm. The device used is a double-beam UV-Vis spectrophotometer of the helios alpha spectrophotometer type.

The dye removal percentage was estimated using the following equation:

$$\% = \left( \frac{C_0 - C_e}{C_0} \right) \times 100 \quad (1)$$

Where,  $C_0$  and  $C_e$  are the initial and equilibrium concentrations of dye in aqueous solution.

## 3. Results and discussion

### 3.1. Chemical oxidation process

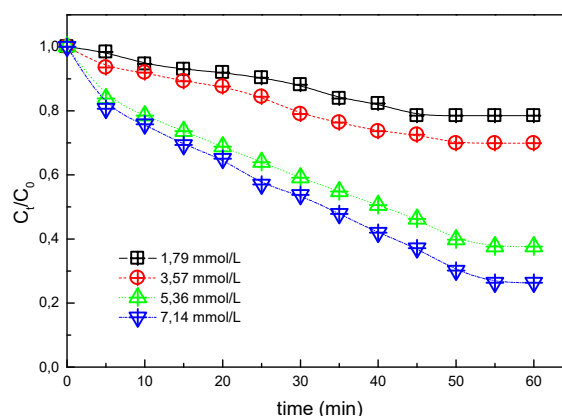
#### 3.1.1. Effect of hydrogen peroxide concentration

In Figure 2 we have represented the variation of the concentration ratio as a function of the reaction time of our four experiments. The results are shown in Table 2.

In general, the curves reported in Figure 2 seem to have the same shape, with a linear slope, low for the low doses of hydrogen peroxide added in solution, and distinct for large quantities of hydrogen peroxide used. , this slope ends in about one hour. The percentage of dis-

**Table 2. Erionyl Navy R Chemical Oxidation Results**

| Oxidant | [Oxidant] (mmol/L) | Reaction time (min) | $10^3 \cdot [Dye]_{\text{final}}$ (mmol/L) | Degradation (%) |
|---------|--------------------|---------------------|--|-----------------|
|         | 1.786              |                     | 11,54                                      | 21.50           |
|         | 3.571              | 60                  | 10,28                                      | 30.10           |
|         | 5.357              |                     | 5,53                                       | 62.36           |
|         | 7.143              |                     | 3,87                                       | 73.65           |
|         | 1.4286             |                     | 0  | 100.00          |
|         | 2.8571             | 13                  | 1,59                                       | 89.20           |
|         | 4.2857             |                     | 1,67                                       | 88.63           |
|         | 5.7143             |                     | 0,25                                       | 98.29           |
|         | 7.1429             |                     | 0,25                                       | 98.29           |

**Figure 2. Effect of hydrogen peroxide concentration ([Dye] = 0.0147 mmol/L, pH = 3, T = 297 °K).**

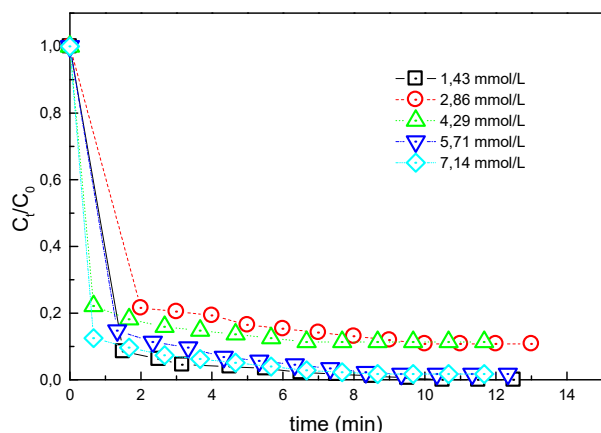


Figure 3. Effect of sodium hypochlorite concentration ( $[Dye] = 0.0147$  mmol/L,  $pH = 3$ ,  $T = 297$  °K).

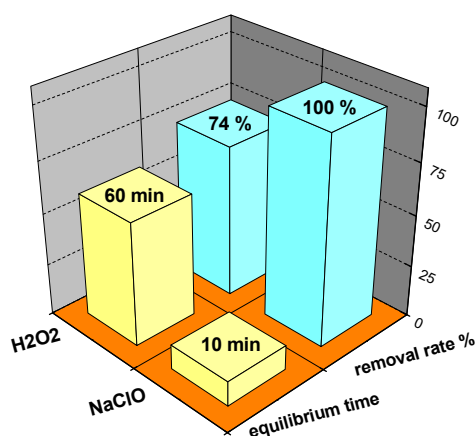


Figure 4. Percentage removal by chemical oxidation.

coloration varies between 21 and 76%.

It can be seen that the degradation reaction of Erionyl Navy R is faster the more hydrogen peroxide is present, and the final yield of each experiment is proportional to the solution concentration.

### 3.1.2. Effect of sodium hypochlorite concentration

In Figure 3 we have represented the variation of the concentration ratio as a function of the reaction time of our five experiments. The results are shown in Table 2.

Firstly, on the curves reported in Figure 3, there is a rapid drop to less than two minutes of reaction, for a degradation rate of close to 80% regardless of the concentration, followed by a slight slope that ends at about 13 minutes. The removal rate varies between 89 and 100%.

In general, the degradation reaction of Erionyl Navy R is faster the higher the concentration of sodium hypochlorite present, and the final yield of each experiment is proportional to the amount added, except for the lowest concentration of 1.4 mmol/L, in this case, we note a total discoloration for a reaction time of 10 minutes.

By comparison, between the two oxidants, peroxide and hypochlorite, the efficiency of the oxidation reaction of the Erionyl Navy R, pre-

Table 3. Erionyl Navy R Oxidant Results in the System ( $Fe^{2+}$ - $H_2O_2$ )

| $[Fe^{2+}]$<br>(mmol/L) | Time<br>(min) | $10^3 \cdot [Dye]_{final}$<br>(mmol/L) | Dye removal<br>(%) |
|-------------------------|---------------|--|--------------------|
| 0.2                     | 35            | 0,41                                   | 97.24              |
| 0.4                     |               | 0,14                                   | 99.08              |
| 0.6                     |               | 0,07                                   | 99.54              |
| 0.8                     |               | 0,54                                   | 96.33              |
| 1.0                     |               | 0,47                                   | 96.78              |

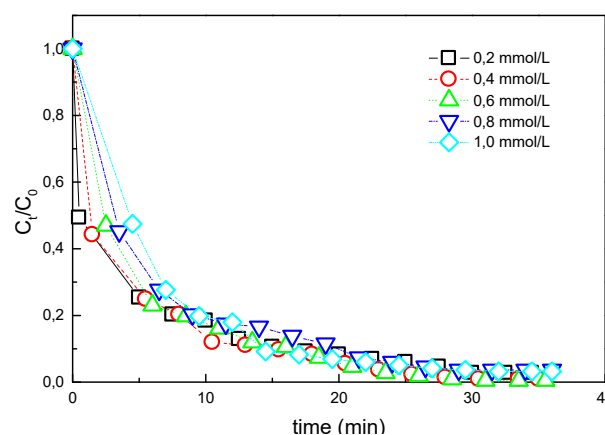


Figure 5. Effect of ferrous ion concentration on the Fenton reaction ( $[Dye] = 0.0147$  mmol/L,  $[H_2O_2] = 8.93$  mmol/L,  $pH = 3$ ,  $T = 297$  °K).

sented in Figure 4, leads us to note that the degradation of our dye and under the same conditions experimental is clearly better when it is carried out by sodium hypochlorite.

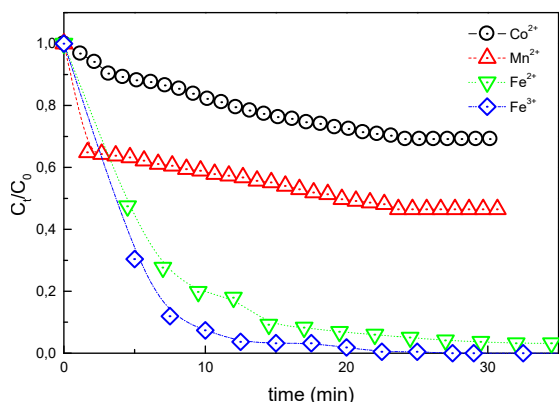
### 3.2. Fenton process ( $Fe^{2+}$ - $H_2O_2$ )

We first studied the fate of the Erionyl Navy R in aqueous solution using the Fenton process (chemical oxidation by hydrogen peroxide in the presence of ferrous ions  $Fe^{2+}$ ). The ferrous ion solution at  $pH = 3$  is always added before that of hydrogen peroxide, since in the presence of  $Fe^{2+}$  ion alone, no oxidation reaction of the dye is observed (no variation of the initial concentration of the dye), whereas in the presence of peroxide of hydrogen alone, there is an oxidation reaction of the Erionyl Navy R dye as seen before.

To evaluate the effect of the amount of ferrous ions in the oxidation process of the Erionyl Navy R dye, experiments were carried out in the presence of different catalyst concentrations ranging from 0.2 to 1.0 mmol/L. The hydrogen peroxide concentration is constant and equals 8.93 mmol/L.

In Figure 5 we have represented the variation of the concentration ratio as a function of the reaction time of our five experiments and in Table 3 the percentage of dye removed.

The results presented in Figure 5 indicate that the oxidation reaction of Erionyl Navy R by oxygen peroxide in the presence of ferrous ions as a catalyst (Fenton process) is more or less rapid since 90% of dye is eliminated between 10 and 15 minutes of reaction, and it is almost complete after 35 minutes.



**Figure 6.** Effect of different catalysts on the Fenton-like reaction ( $[Dye] = 0.0147 \text{ mmol/L}$ ,  $[H_2O_2] = 8.93 \text{ mmol/L}$ ,  $[M^{n+}] = 1.0 \text{ mmol/L}$ ,  $pH = 3$ ,  $T = 297 \text{ °K}$ ).

It is also observed that when the quantity of ferrous ions ( $Fe^{2+}$ ) added increases, the efficiency of the discoloration and degradation of the dye increases. This can be explained by the fact that the hydroxyl radicals ( $OH \cdot$ ) formed in the solution are directly related to the quantity of ferrous ions ( $Fe^{2+}$ ) added.

The rate of dye discoloration increased up to a concentration of 0.6 mmol/L ferrous ions ( $Fe^{2+}$ ), the increase in concentration led to a decrease in dye discoloration efficiency. This could be due to the limited concentration of  $H_2O_2$  in the solution[11].

### 3.3. Fenton-Like process ( $M^{n+}-H_2O_2$ )

The effect of different added catalysts (metal ions ( $M^{n+}$ )) on the degradation reaction of the Erionyl Navy R dye was carried out in the presence of a catalyst concentration equal to 1 mmol/L and a hydrogen peroxide concentration of 8.93 mmol/L.

The catalyst solutions are always added before that of the chemical oxidant, since in the presence of metal ions alone, no dye oxidation reaction is observed, whereas in the presence of the chemical oxidant alone, there is an oxidation reaction of Erionyl Navy R.

The different metallic ions studied by comparison are  $Fe^{2+}$ ,  $Fe^{3+}$ ,  $Mn^{2+}$ ,  $Co^{2+}$ .

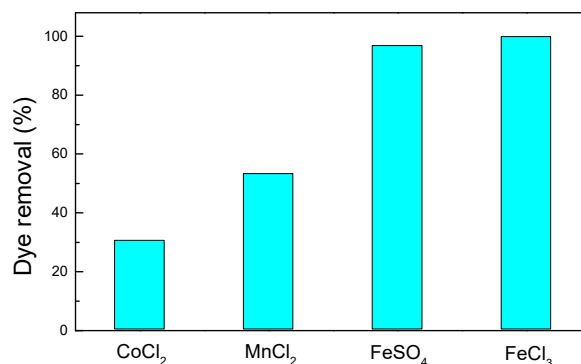
In Figure 6 we have represented the variation of the concentration ratio as a function of the reaction time for the different catalysts and in Figure 7 the percentage of dye removed. The results are reported in Table 4.

From the results found and presented in Figures 6 and 7, it is noted that even if the oxidation reaction in the presence of manganous ions is greater than that experienced by cobaltous ions, with a rate which slightly exceeds 53% for the first and 30% for the second, the oxidation reaction remains more important with ferrous and ferric ions with a percentage of degraded dye exceeding 96% and 100% consecutively after 30 minutes of reactions.

This could be due to the ratio ( $H_2O_2 / \text{metal ion}$ ) which is not optimal, in the case of the cobaltous ion, it has been reported that the optimal  $H_2O_2/Co^{2+}$  ratio is 6, and beyond this value, the percentage of degradation is much lower[12]. The decrease in dye bleaching rate is at-

**Table 4.** Erionyl Navy R Oxidant Results in the System ( $M^{n+}-H_2O_2$ )

| Catalysts | $[M^{n+}]$<br>(mmol/L) | Time<br>(min) | $10^3 \cdot [Dye]_{\text{final}}$<br>(mmol/L) | Dye removal<br>(%) |
|-----------|------------------------|---------------|---|--------------------|
| $CoCl_2$  | 1,0                    | 30            | 10,17   | 30.81              |
| $MnCl_2$  |                        |               | 6,83  | 53.51              |
| $FeSO_4$  |                        |               | 0,52  | 96.45              |
| $FeCl_3$  |                        |               | 0   | 100                |



**Figure 7.** Effect of different catalysts on dye removal of Fenton-type reactions.

tributed to the fact that the amount of unreacted peroxide acts as a hydroxide radical scavenger[13]. In the case of the ferrous ion ( $Fe^{2+}$ ) which appears to be slightly less effective than the ferric ion, this can be explained, within our experimental errors, by a limited concentration of  $H_2O_2$  in the solution and its relationship to the catalyst.

### 3.4. Fenton-Like process ( $Fe^{3+}-H_2O_2$ )

In view of the results reported in the previous paragraph, which suggest that the ferric ion ( $Fe^{3+}$ ) is in the operating conditions of our experiments and by comparison with the various catalysts, the results are satisfactory, therefore a study on the effect of Ferric ion assay on Fenton-like degradation of Erionyl Navy R was monitored. Figure 8 shows the dye degradation for different concentrations of the ferric ion ( $Fe^{3+}$ ) ranging from 0.2 to 1 mmol/L, with a fixed concentration of hydrogen peroxide equal to 8.93 mmol/L. Table 5 shows the results found.

It can be seen in Figure 8 that the oxidation reaction of our dye by the modified Fenton process is more or less rapid since approximately 90% of dye is eliminated between 10 and 25 minutes of reaction. Also, of the different concentrations of ferric ion added, the curves seem to have the same appearance.

The discoloration rate of Erionyl Navy R apparently increases with increasing ferric ion concentration from 0.2 to 0.6 mmol/L, the improved dye degradation can be mainly attributed to the faster reaction rates of the hydrogen peroxide ( $H_2O_2$ ) with higher levels of ferric ions in Fenton-type reactions[14]. For the quantities of ferric ions that were added greater than or equal to 0.6 mmol/L, the discoloration was completed in 27 minutes. Less than that, the reaction is not complete and the percentage of elimination is all less important as the quantity of ferric ions is low.

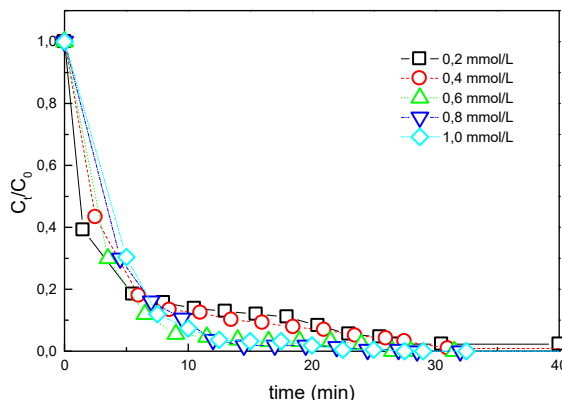


Figure 8. Effect of ferric ions concentration on the Fenton reaction ([Dye] = 0.0147 mmol/L, [H<sub>2</sub>O<sub>2</sub>] = 8.93 mmol/L, pH = 3, T = 297 °K).

Table 5. Erionyl Navy R Oxidant Results in the System (Fe<sup>3+</sup>-H<sub>2</sub>O<sub>2</sub>)

| [Fe <sup>2+</sup> ]<br>(mmol/L) | Time<br>(min) | 10 <sup>3</sup> ·[Dye] <sub>final</sub><br>(mmol/L) | Dye removal<br>(%) |
|---------------------------------|---------------|---|--------------------|
| 0.2                             |               | 0,34  | 97.70              |
| 0.4                             |               | 0,14  | 99.08              |
| 0.6                             | 40            | 0   | 100.00             |
| 0.8                             |               | 0   | 100.00             |
| 0.10                            |               | 0   | 100.00             |

### 3.5. Effect of the oxidant NaClO in the presence of Fe<sup>3+</sup> and Fe<sup>2+</sup>

The effect of the degradation reaction of the Erionyl Navy R by sodium hypochlorite in the presence of ferrous and ferric ions was carried out under the same operating conditions of the Fenton test (initial concentration of the dye, reaction volume, pH and temperature).

The two experiments were carried out in the presence of a catalyst concentration equal to 1 mmol/L and a sodium hypochlorite concentration equal to 1.429 mmol/L.

In Figure 9 we have represented the variation of the concentration ratio as a function of the reaction time for the two catalysts.

From the results found, it is noted that the degradation reaction of the Erionyl Navy R dye by sodium hypochlorite is greater in the presence of ferric ions than ferrous ions.

In the presence of ferrous ions, the degradation rate is less than 11% recorded in the first minute and reaches a maximum of 19.7% after 19 minutes 30 seconds.

In the presence of ferric ions, the degradation rate is already equal to 80.7% in the first minute and reaches a maximum of 94.82% in 18 minutes.

Even if the percentage of the dye degradation reaction by sodium hypochlorite is greater in the presence of ferric ions than ferrous ions, that carried out in the presence of sodium hypochlorite alone is greater and therefore more effective.

## 4. Conclusion

Several oxidation processes have been studied for the degradation of Erionyl Navy R acid dye. The results are very promising, as each proc-

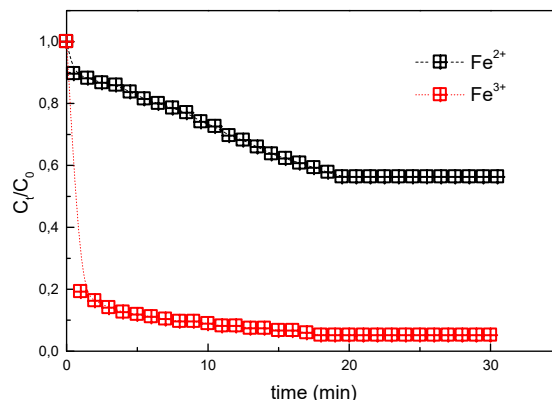


Figure 9. effect of Fe<sup>2+</sup> and Fe<sup>3+</sup> on oxidation by sodium hypochlorite ([Dye] = 0.0147 mmol/L, [NaClO] = 1.429 mmol/L, [Fe<sup>n+</sup>] = 1.0 mmol/L, pH = 3, T = 297 °K).

ess has specific attributes that can be explored for the implementation of remediation procedures. The experimental results of the chemical oxidation tests revealed that the discoloration and degradation of the dye are significantly better when carried out by sodium hypochlorite, with a high removal rate in less time. The presence of a metal ion as a catalyst for hydroxyl radicals, which are stronger oxidants than the chemical oxidants used, showed variability in the degradation efficiency of our dye, depending on the metal ion used and under the operating conditions applied. Ferrous and ferric ions are much better, nevertheless, manganous ions seem more promising than cobaltous ions in the presence of hydrogen peroxide. On the other hand, even if the degradation of the acid dye by sodium hypochlorite in the presence of iron ions seems to be reduced, it is clearly better in the presence of ferric ions than ferrous ions. Among the different oxidation methods involved, Fenton process, Fenton-like and chemical oxidation, Fenton-type degradation reactions in the presence of ferric and ferrous ions gave the best performance for the degradation of the acid dye Erionyl Navy R. Sodium hypochlorite treatment, which is probably the most common technique in practice in textile wastewater treatment plants, has proven to be an easily applied and alternative choice. Further research is needed on this topic, and mechanistic studies to further examine dye fate and degradability will be reported.

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