

# Evaluation of Fenton Process in Removal of Direct Red 81

Mohammad Ali Baghapour,  
Mansooreh Dehghani,  
Zahra Elhamiyan

Department of Environmental Health  
Engineering, School of Health, Shiraz  
University of Medical Sciences, Shiraz,  
Iran

## Correspondence:

Mohammad Ali Baghapour, PhD;  
Department of Environmental Health  
Engineering,  
School of Health,  
Shiraz University of Medical Sciences,  
Shiraz, Iran

Tel: +98-917-1121658

Fax: +98-71-37260225

Email: baghapour@sums.ac.ir

Received: 18 August 2015

Revised: 9 September 2015

Accepted: 25 October 2015

## Abstract

**Background:** Dyes are visible materials and are considered as one of the hazardous components that make up the industrial waste. Dye compounds in natural water, even in very low concentrations, will lead to environmental problems. Azo dyes are compounds with one or more  $-N=N-$  groups and are used in textile industry. Because of its low price, solubility, and stability, azo dyes are widely used in the textile industry. Direct Red 81 (DR81) is one of the azo dyes, which is removed from bodies of water, using various methods. This study aimed to assess DR81 dye removal by Fenton oxidation and the effects of various parameters on this process.

**Methods:** Decolorization tests by Fenton oxidation were performed at dye concentrations of 50, 500, 100 and 1000 mg/L; hydrogen peroxide concentrations of 0, 10, 30, 60 and 120 mg/L; iron (II) sulfate heptahydrate concentrations of 0, 3, 5, 20 and 50 mg/L; and pH levels of 3, 5, 7 and 10 for durations of 5, 10, 20, 30, 60 and 180 minutes.

**Results:** The optimal condition occurred at a dye concentration of 20 mg/L, hydrogen peroxide concentration of 120 mg/L, bivalent iron concentration of 100 mg/L, pH of 3, and duration of 30 minutes. Under such conditions, the maximum dye removal rate was 88.98%.

**Conclusion:** The results showed that DR81 could be decomposed and removed by Fenton oxidation. In addition, the removal of Direct Red 81 (DR81) depends on several factors such as dye concentration, reaction time, concentrations of hydrogen peroxide and iron, and pH.

Please cite this article as: Baghapour MA, Dehghani M, Elhamiyan Z. Evaluation of Fenton Process in Removal of Direct Red 81. *J Health Sci Surveillance Sys.* 2016;4(1):14-21.

**Keywords:** Fenton process, Direct Red 81 dye, Advanced oxidation process, Hydroxyl radical, Waste water treatment

## Introduction

Textile and dyeing industries form a major part of the economy in many countries. One of the important outcomes of this industry is production of a large volume of wastewater that contains a complex mixture of compounds and toxic dyes.<sup>1</sup> Evidence has indicated that 15-20% of dyes used in this industry enter the sewage system.<sup>2,3</sup> During the textile processes, a substantial amount of water, energy, and chemicals are consumed. Most of the dyes used in this industry are of artificial origin, have aromatic rings, are chemically stable, and are

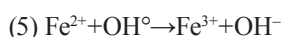
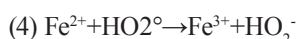
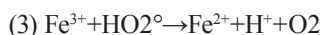
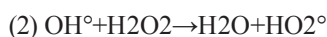
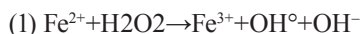
not easily biodegraded.<sup>1</sup> Therefore, their direct disposal into the environment can create numerous hazards. Studies have shown that the presence of dye compounds in natural water, even at very low concentrations, can lead to problems.<sup>4</sup>

More than 10,000 different chemical dyes are widely used in many industries, such as textile industry, publication, plastics, rubbers, leathers, pharmaceutical materials, food, solvents, and cosmetics. Thus, a significant amount of dye waste is produced at the end of the processes in these

industries, which is sometimes discharged into natural water. Release of wastewater containing dyes into water systems causes numerous adverse effects on human health and has environmental risks, as well. Dyes in wastewater can break down aromatic amines under aerobic conditions, which is carcinogenic and can cause serious health problems for humans and animals. Dyes can cause allergies, dermatitis, skin irritation, and cancer in humans.<sup>4</sup> In case dyes and other compounds are released into the environment, especially in water resources, they reduce the receiving waters' self-purification capacity. In addition, the insoluble materials resulting from dyeing prevent the light from entering aquatic environment and decrease the rate of photosynthesis.<sup>5,6</sup>

Therefore, treatment of wastewater containing dyes is very important. In this regard, several physicochemical methods, including adsorption, precipitation, coagulation, flocculation, reduction, electrochemical treatment, and photo degradation, have been used to eliminate dyes from wastewater.<sup>4</sup>

One of these advanced oxidation methods is Fenton reaction, which is a catalytic process wherein hydroxyl radicals are produced from hydrogen peroxide. These radicals are very strong oxidizing agents and are able to react with a wide variety of organic compounds under environmental conditions. The main steps of Fenton reaction process have been shown in the following equations:<sup>7</sup>



The advantage of Fenton process is that no energy input is required to activate the hydrogen peroxide and it is quickly decomposed under acidic conditions in the presence of iron ions.<sup>7</sup>

## Objectives

This study aimed to assess Direct Red 81 (DR81) dye removal via Fenton oxidation process and the influence of various parameters on its removal efficiency.

## Materials and Methods

### Chemicals and Instruments

The direct azo dye DR81 ( $\lambda_{\text{max}}$ : 510 nm, Molecular formula:  $\text{C}_{29}\text{H}_{19}\text{N}_5\text{Na}_2\text{O}_8\text{S}_2$ , and molecular weight: 675.6) was purchased from Alvan Sabet Company. The dye's chemical structure is presented in Figure 1.

Iron (II) sulfate heptahydrate, sulfuric acid (98%), hydrogen peroxide (30%), sodium hydroxide, and other materials used in laboratory experiments were purchased with the purity of 90% from Merck Company. Dye concentration in the samples was measured at  $\lambda_{\text{max}}=510$  nm using DR 5000 Spectrophotometer at Hach USA and the calibration curve. A magnetic stirrer was used for stirring the solution and pH adjustment was carried out by 1 N sulfuric acid and 1 N sodium hydroxide. Additionally, pH was measured by Metrohm 826 pH Meter-Swiss made. Besides, the decolorization rate of the dyed effluent was measured using a spectrophotometer. It should be noted that the spectrophotometer was automatically calibrated and calibration of the pH meter was checked before beginning of the work.

### Method

The current research was conducted in 2015 in Shiraz. The tests were performed in a 2000-ml batch glass reactor at room temperature while thorough mixing condition was created with a magnetic stirrer. Deionized water was used to create artificial effluent.

Decolorization experiments were performed at dye concentrations of 50, 500, 100 and 1000 mg/L; hydrogen peroxide concentrations of 0, 10, 30, 60 and 120 mg/L; iron (II) sulfate heptahydrate concentrations of 0, 3, 5, 20 and 50 mg/L; and pH levels of 3, 5, 7 and 10 for durations of 5, 10, 20, 30, 60 and 180 minutes.

In each decolorization experiment, the desired amount of dye was brought to the target volume with distilled water. Then, hydrogen peroxide and ferrous sulfate at specified concentrations were added to the reactor simultaneously. Then, the magnetic stirrer was turned for mixing. At the same time, pH was adjusted. By adjusting pH, Fenton reaction began and the sample absorption was read at zero time using UV/Vis spectrophotometer. Reading was performed

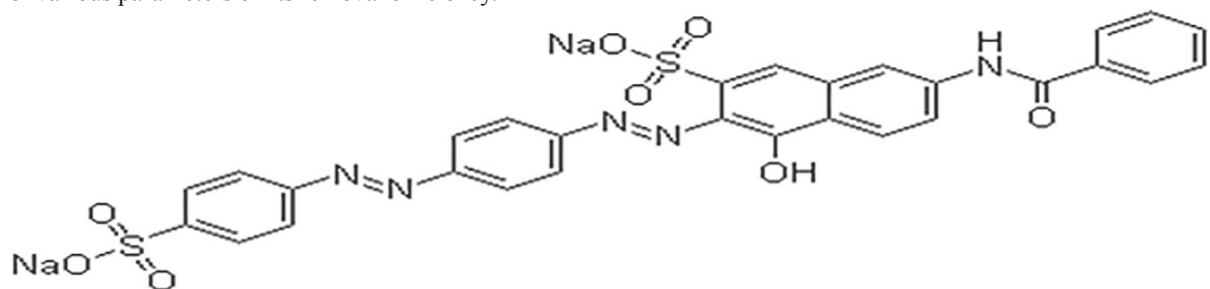


Figure 1: Chemical structure of DR81<sup>8</sup>

for samples at different times at specified intervals by UV/Vis spectrophotometer and dye removal was measured.

In the tests, a variable was changed while keeping other variables constant to determine the highest removal rate, i.e. the optimal conditions for that particular variable. The parameters affecting the dye removal were compared using SPSS statistical software and the results are presented in Table 1. This comparison was expressed by the sensitivity analysis, and the relationship (reverse or direct) between the study parameters and the effectiveness of each parameter in the dye removal was examined. The results are shown as positive or negative in column B of Table 1.

## Results

When presenting the results of the effects of the parameters on the process performance, only the effect of the same parameter at various ranges is stated, while assuming the rest of the parameters constant.

### The Effect of pH on DR81 Removal

According to studies, one of the factors that

influence the removal efficiency in the Fenton reaction is the solution's pH level.

In this study, the effects of different pH levels (3, 5, 7, 10) were evaluated at hydrogen peroxide concentration of 30 mg/L, iron concentration of 20 mg/L, DR81 concentration of 50 mg/L, and reaction time of 20 minutes. According to the results, pH=3 showed the highest efficiency in such a way that at pH levels of 3, 5, 7, and 10, the removal efficiency was 66.78%, 9.49%, 8.27%, and 4.3%, respectively. The effect of pH on DR81 removal is shown in Figure 2.

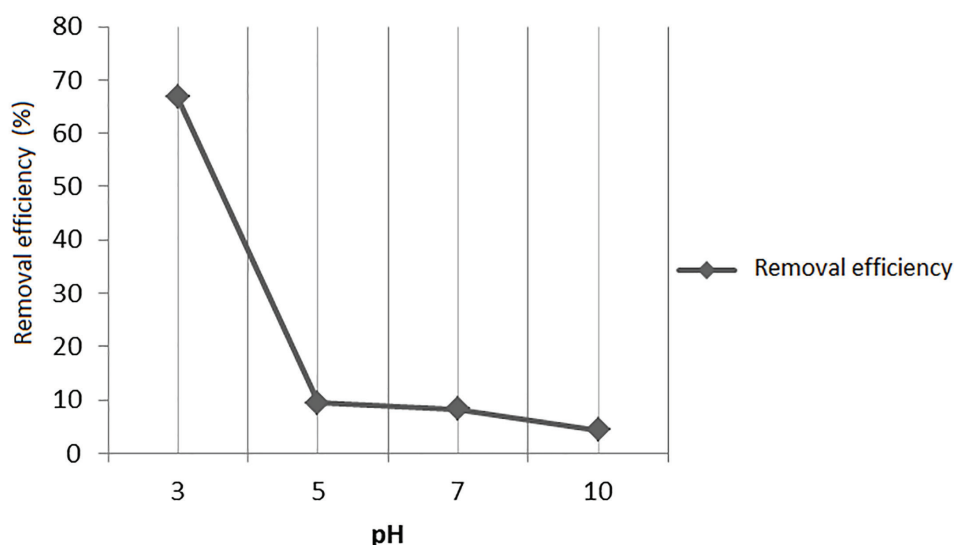
### The Effect of Bivalent Iron Ions Concentration on DR81 Removal

In this section, the effect of the iron concentration on the Fenton process was examined and the results are presented in Figure 3. In this stage, the effect of different doses of bivalent iron concentration (0, 3, 5, 20, and 50 mg/L) was investigated at pH of 3, reaction time of 20 min, hydrogen peroxide concentration of 30 mg/L, and dye concentration of 50 mg/L.

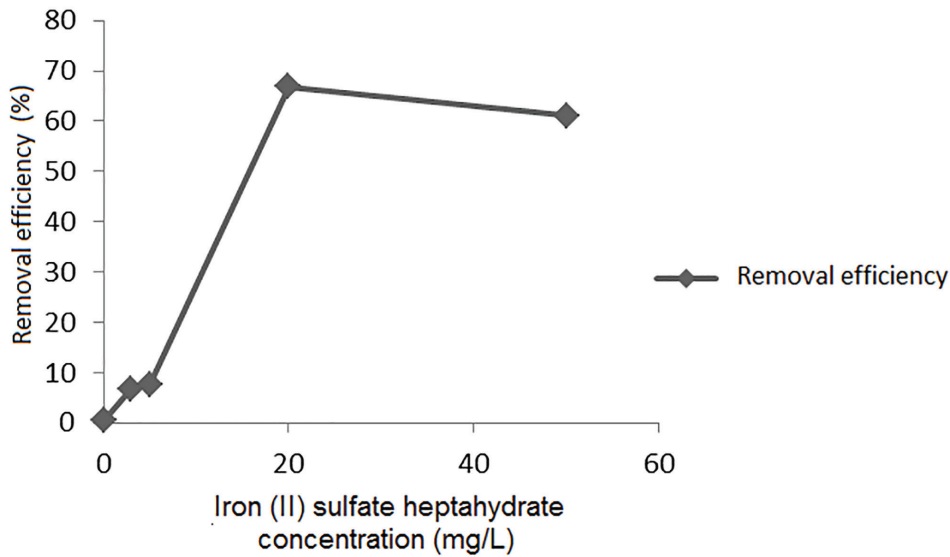
As iron concentration increased from zero to 20 mg/L, the dye removal rate increased, as well. However, at iron concentrations above 20 mg/L,

**Table 1:** Comparison of the factors affecting the Fenton process

| Model |                                     | Unstandardized coefficients |            | Standardized coefficients | t       | Sig.  |
|-------|-------------------------------------|-----------------------------|------------|---------------------------|---------|-------|
|       |                                     | B                           | Std. Error | Beta                      |         |       |
| 1     | (Constant)                          | 7.491                       | 0.924      |                           | 8.103   | 0.000 |
|       | color ,mg/l                         | -0.003                      | 0.001      | -0.098                    | -4.218  | 0.000 |
|       | Fe <sup>+2</sup> ,mg/l              | 0.136                       | 0.014      | 0.226                     | 9.720   | 0.000 |
|       | H <sub>2</sub> O <sub>2</sub> ,mg/l | 0.027                       | 0.006      | 0.105                     | 4.544   | 0.000 |
|       | pH                                  | -1.037                      | 0.101      | -0.239                    | -10.306 | 0.000 |
|       | Time, min                           | 0.134                       | 0.027      | 0.115                     | 4.960   | 0.000 |



**Figure 2:** The effect of pH on DR81 decolorization rate (at dye concentration of 50 mg/L, peroxide concentration of 30 mg/L, bivalent iron concentration of 20 mg/L, and reaction time of 20 min)



**Figure 3:** The effect of Iron (II) sulfate heptahydrate on DR81 decolorization rate (at dye concentration of 50 mg/L, hydrogen peroxide concentration of 30 mg/L, pH of 3, and reaction time of 20 minutes)

the removal efficiency slightly reduced. The lowest and highest dye removal rates occurred at iron concentrations of 0 and 20 mg/L, respectively. Dye removal rates were 0.47%, 6.85%, 7.47%, 66.78%, and 61.02% at concentrations of 0, 3, 5, 20, 50 mg/L, respectively. However, the concentrations higher than 20 mg/L did not have a significant effect on the DR81 decolorization rate. Thus, the optimum bivalent iron concentration recommended for these conditions is 20 mg/L.

The effect of iron sulfate concentration on DR81 removal rate is shown in Figure 3.

#### *The Effect of Hydrogen Peroxide Concentration on DR81 Removal*

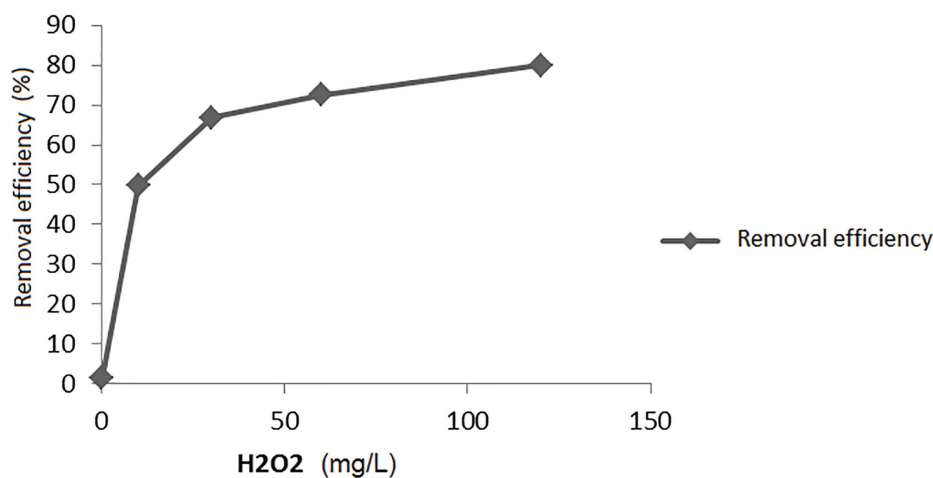
The effect of different hydrogen peroxide concentrations on DR81 removal efficiency was

investigated at iron concentration of 20 mg/L, pH level of 3, and dye concentration of 50 mg/L. By increasing hydrogen peroxide concentration, more hydroxyl radicals are produced. The results showed that by increasing the initial concentration of hydrogen peroxide from 0 to 120 mg/L (0, 10, 30, 60, 120), the dye removal rate also showed an increasing trend (1.055%, 49.67%, 66.78%, 72.57%, and 79.91%, respectively). Therefore, the hydrogen peroxide concentration of 120 mg/L was considered as optimal.

Dye removal efficiency according to changes in hydrogen peroxide concentration is presented in Figure 4.

#### *The Effect of Time on DR81 Removal*

In order to evaluate the effect of time on the dye removal rate, DR81 concentration was measured at



**Figure 4:** The effect of hydrogen peroxide concentration on DR81 removal rate (at dye concentration of 50 mg/L, ferric sulfate concentration of 20 mg/L, pH of 3, and reaction time of 20 minutes)

reaction times of 5, 10, 20, 30, 60 and 180 minutes at pH of 3; iron sulfate concentration of 20 mg/L, hydrogen peroxide concentration of 120 mg/L; and initial dye concentration of 50 mg/L. The removal efficiency rates were 34.24%, 60.67%, 79.91%, 85.41%, 89.6%, and 99.9, respectively.

The dye removal rates according to changes in the reaction time are shown in Figure 5.

Then, the experiment continued under the same conditions until 180 minutes when the removal efficiency reached 99.9%. It should be noted that the highest dye removal rate occurred in the first 30 minutes of the reaction.

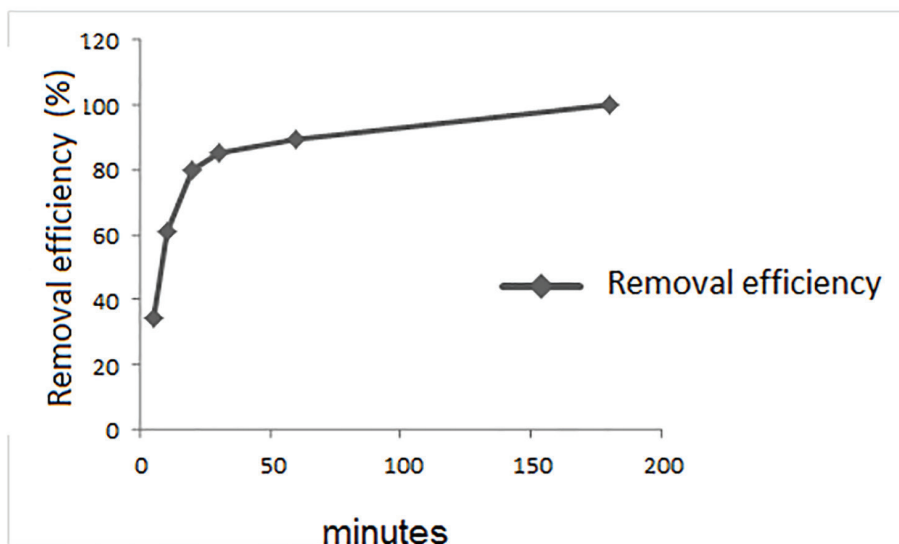
*The Effect of Dye Concentration on DR81 Removal*

The effect of dye concentration on the removal efficiency is presented in Figure 6. As shown in the

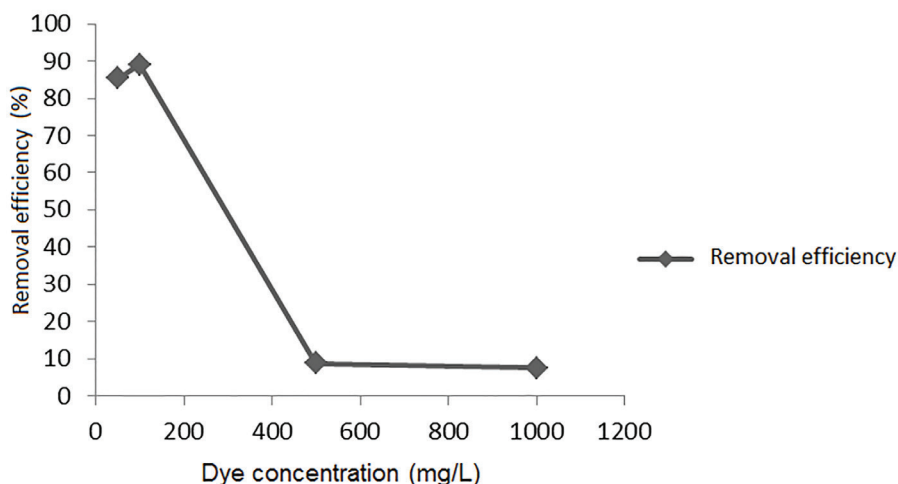
figure, at dye concentrations of 50, 100, 500, and 1000 mg/L, the removal efficiency was 85.41%, 88.98%, 8.73%, and 7.39%, respectively. Thus, the highest removal efficiency was obtained at the dye concentration of 100 mg/L.

**Discussion**

One of the conditions of the Fenton process is performing the experiments at low pH levels where the process is more efficient. At pH levels above 3, the solubility of iron is low; thus, ferric ion precipitates as hydroxide and can be removed from the catalytic cycle.<sup>9,10</sup> The optimum pH level in the Fenton process ranged from 2 to 4. At pH levels greater than 4, bivalent iron ions become unstable and change into trivalent iron ions. Within the alkaline range, the oxidation power of hydrogen peroxide reduces due to conversion into water and oxygen.<sup>11</sup>



**Figure 5:** The effect of reaction time on DR81 decolorization rate (at dye concentration of 50 mg/L, ferric sulfate concentration of 20, hydrogen peroxide concentration of 120 mg/L, and pH of 3)



**Figure 6:** The effect of DR81 concentration on removal efficiency (at ferric sulfate concentration of 20 mg/L, hydrogen peroxide concentration of 120 mg/L, pH of 3, and reaction time of 30 minutes)



According to Figure 2, in constant conditions (hydrogen peroxide concentration of 30 mg/L, iron concentration of 20 mg/L, DR81 dye concentration of 50 mg/L, and reaction time of 20 minutes), dye removal efficiency decreased with increasing pH. Besides, as pH increased (3, 5, 7, 10), the removal efficiency rates of 66.78%, 9.49%, 8.27%, and 4.3 were achieved, respectively.

Kashmiri Zade and colleagues studied the removal of Anionic Brown 14 and Cationic Blue 41 by Fenton oxidation at acidic pH (3-5) and achieved the high removal rate of 99%.<sup>7</sup>

Moreover, Sohrabi and colleagues found that at bivalent iron concentration of 0.015 Mmol, hydrogen peroxide concentration of 0.15 Mmol, and dye concentration of 20 mg/L, dye removal efficiency reached 92.7% at pH of 3.<sup>12</sup>

El-Haddad and colleagues also investigated decolorization of aqueous solutions containing an azo dye (Reactive Yellow 84) by advanced oxidative process using Fenton reagent. The Fenton process was effective at pH=3 and decolorization efficiency of 85% was attained at the reaction time of 20 min.<sup>13</sup>

According to Figure 3, at the pH level of 3, reaction time of 20 minutes, hydrogen peroxide concentration of 30 mg/L, and dye concentration of 50 mg/L, dye removal efficiency was 0.47%, 6.85%, 7.47%, 66.78%, and 61.02% at iron concentrations of 0, 3, 5, 20, 50 mg/L, respectively.

Many studies have shown that excessive concentrations of bivalent iron had an inhibitory effect on hydroxyl radicals and reduced the destruction rate of chemical compounds.<sup>14,15</sup> By increasing the initial concentration of ferrous ions, the dye decolorization rate increased due to rapid production of a large number of hydroxyl radicals. These radicals quickly react with dye molecules and can increase the decolorization rate. However, at concentrations above 20 mg/L, ferrous ions had an inhibitory effect on the production of hydroxyl radicals, which could slow down the destruction rate of the chemical compounds. This result was consistent with that obtained by Chen and colleagues in 1997<sup>16</sup> and by Joseph and colleagues in 2000.<sup>17</sup> Barbusiński also showed that as the Fe concentration increased from 3.2 to 37.5 mg/dm<sup>3</sup>, dye removal efficiency rapidly increased from 82.0% to 95.4%. However, when the concentration of Fe ions exceeded 37.5 mg/dm<sup>3</sup>, only a slight increase from 95.4% to 96.6% was observed.<sup>18</sup> In the same line, Chiou and colleagues investigated the removal of reactive black 5 and showed that as the concentration of iron ions increased from 0 to 20 mg/L, removal efficiency increased. However, increasing the concentration of iron ions to 30 mg/L decreased the

removal efficiency.<sup>19</sup> These results were also similar to those of the study by Panizza and colleagues.<sup>20</sup>

Hydrogen peroxide has been proposed as a factor affecting Fenton oxidation. This factor leads to production of hydroxyl radicals.<sup>21</sup>

According to Figure 4, the maximum removal rate (79.91%) occurred at hydrogen peroxide concentration of 120 mg/L. This was achieved at pH of 3, dye concentration of 50 mg/L, iron concentration of 20 mg/L, and reaction time of 20 minutes. This is in agreement with the results of the study by Bahmani and colleagues. They found that at Remazol Black B concentrations of 200 and 500 mg/L, as hydrogen peroxide concentration increased from 50 to 300 mg/L, the decolorization rate increased from 73% to 98% and from 52% to 96%, respectively.<sup>22</sup> Selecting the appropriate concentration of hydrogen peroxide is important for two reasons. First, increase in hydrogen peroxide concentration leads to formation of less active radical hydroperoxyl and reduces the efficiency of the oxidation process. Second, due to the high cost of hydrogen peroxide, using its excessive amounts is not cost-effective.<sup>15</sup>

As shown in Figure 5, increase in reaction time resulted in an increase in dye removal efficiency. This is due to the fact that increasing the time results in production of many hydroxyl radicals, which in turn leads to combination with and decomposition of the dye and production of water, carbon dioxide, and intermediate compounds.<sup>23</sup>

In a study conducted by Ganesan and colleagues, the decolorization rate of real dyeing wastewater by solar PhotoFenton reached 89% in 30 minutes.<sup>24</sup> This was similar to the findings of the study by Sibel Tunc and colleagues in which with an increase in iron concentration from  $2.5 \times 10^{-6}$  to  $2.5 \times 10^{-5}$  M, dye removal efficiency increased from 54.5% to 98.9% at the end of 180 minutes.<sup>25</sup>

In a study carried out by El-Haddad and colleagues in 2013, dye removal efficiency was 85% at pH of 3, bivalent iron concentration of 25 mg/L, hydrogen peroxide concentration of 250 mg/L, and reaction time of 20 minutes.<sup>13</sup>

Based on Figure 6, at dye concentrations of 50, 100, 500, 1000 mg/L, the removal efficiency was 85.41%, 88.98%, 8.73%, and 7.39%, respectively. Increasing the concentration of the dye leads to an increase in the number of dye molecules while the hydroxyl radical concentration in the solution does not change. Therefore, the decolorization rate decreases with increasing the dye concentration.<sup>25</sup> According to a study conducted by Bahmani and colleagues, increase in the dye concentration led to a competition among intermediate materials (aromatic amines) formed by decomposition of the dye on one hand and

the mother dye molecules on the other hand. Thus, when dye concentration increases, the amount of intermediate materials increases, as well.<sup>22</sup> On the other hand, the removal efficiency is low in low dye concentrations. This can be attributed to the fact that in low concentrations of dyes and pollutants, hydrogen peroxide breaks down into oxygen and water and recomposes with hydroxyl radicals.<sup>22</sup>

The findings of the research by Seibel indicated that by increasing the dye concentration from  $2.5 \times 10^{-6}$  to  $2.5 \times 10^{-5}$  Molar, the removal efficiency decreased from 73.2% to 65.1%.<sup>25</sup>

Mousavi and colleagues also reported that the highest removal efficiency of Rhodamine B occurred at a dye concentration of 100 mg/L and pH of 3.<sup>21</sup> Similarly, the results of the research carried out by Panizza and Cerisola on removing alizarin red revealed a Chemical Oxygen Demand (COD) removal efficiency of more than 90% under optimum conditions and reaction time of 120 minutes.<sup>20</sup>

In general, the factors affecting Fenton process include reaction time, pH, concentration of hydrogen peroxide, concentration of iron, and concentration of the contaminant. The present study assessed the effect of each factor on the dye removal efficiency. According to the analyses shown in Table 1, the removal efficiency was directly related to the concentration of bivalent iron, reaction time, and concentration of peroxide. Besides, greater dye concentrations resulted in higher removal efficiencies. Moreover, iron concentration was strongly associated with removal efficiency, and dye concentration and pH had inverse correlations with efficiency. An inverse relationship was also found between pH and removal rate. Therefore, it can be concluded that among these factors, pH followed by iron concentration had the strongest effects ( $P < 0.001$ ). Since the removal rate was higher in acidic pH and increase in reaction time improved the removal efficiency, pH and time were considered constant in further analyses (pH=3 and reaction time=30 minutes).

A sensitivity analysis of decolorization rate using Fenton process in relation to the parameters under the study is presented in Table 1.

## Conclusion

Test results showed that DR81 could be decomposed by Fenton oxidation. The removal rate depended on the initial dye concentration, reaction time, hydrogen peroxide concentration, iron concentration, and pH.

At initial DR81 concentration of 100 mg/L, hydrogen peroxide concentration of 120 mg/L, bivalent iron concentration of 20 mg/L, pH of 3, and reaction time of 30 minutes, Fenton oxidation method had the

highest efficiency (88.98%). Hence, these conditions were defined as optimal conditions. Moreover, pH had the highest and dye concentration had the lowest effects on the Fenton reaction.

It is also worth mentioning that increasing the reaction time up to 180 minutes could increase the dye removal efficiency to 99.9%, but this increases the reactor volume that is not economically viable.

## Acknowledgments

This article was extracted from Zahra Elhamiyan's M.Sc. thesis approved and financially supported by the Research Vice-chancellor of Shiraz University of Medical Sciences (Research project of 93-7049). Hereby, the authors would like to appreciate Ms. A. Keivanshekouh at the Research Improvement Center of Shiraz University of Medical Sciences for improving the use of English in the manuscript. They are also grateful to the Alvan Sabet Company for providing DR81. Thanks also go to all the individuals who helped the researchers during the course of this study.

**Conflict of Interest:** None declared.

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