

Decolorization of Direct Red 81 in Aqueous Solutions by Fenton Oxidation Process: Effect of System Parameters

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ABSTRACT

Dye industries and textile are among the most water-consuming industries, which severely disturb the aquatic life. Therefore, the aim of this study was to determine the feasibility of Fenton process in removing Direct Red 81 dye from aqueous solutions and the optimal conditions for maximum removal.

This research was conducted in a laboratory-scale using a one-litre photochemical reactor. The effect of the influential parameters, including pH (3–9), Fe (II) concentration (10–150mg/L), H₂O₂ concentration (20–150mg/L), initial dye concentration (25–150mg/L), and reaction time (15–120min) on the dye removal was investigated and the optimal conditions were determined according to maximum dye removal efficiency.

The results showed that the dye removal rate increased as the pH and Fe(II) concentration decreased and as the initial dye concentration and time increased. The optimal condition was at pH=3, Fe (II)=10mg/L, H₂O₂=50mg/L, initial dye concentration=100mg/L, and reaction time= 45 minutes. Although the maximum removal efficiency (98.29%) was obtained at the reaction time of 120 minutes, 45 min was the appropriate reaction time considering the cost-effectiveness.

Our results suggest that the Fenton process is a reliable and efficient method with more than 95% efficiency for decolourization of DR-81 dye and many industrial wastewaters.

Keywords: Direct Red 81 Dye Removal, H₂O₂, Fenton Process, Advanced Oxidation Processes

INTRODUCTION

Industrial wastewater, especially textile wastewater, contains large amounts of dyes that play a major role in contaminating the receiving waters [1]. Textile and dyeing industries use large amounts of water and are among the most water consuming industries [2]. During the dyeing process in the world, about 10-15% of the unfixed dye is lost in water which comes out as a coloured effluent from the industries and is released into the environment [3, 4]. Dyes are normally very large aromatic molecules consisting of many linked rings. Four common classes of dyes, classified according to the fibers to which they can be applied and their chemical nature, are anionic dyes (acidic, direct, and reactive), cationic dyes (all alkaline dyes), and non-ionic dyes (disperse dyes) [5, 6].

Dye-containing effluent can be toxic to the environment [7] because dyes directly interfere with the photosynthesis process, thereby reducing the growth of organisms. In addition, dyes may form metallic bonds and produce highly toxic compounds to fish and other aquatic organisms. Moreover, they are

liable to be carcinogenic; especially, azo dyes are known to be highly toxic [8, 9]. In order to minimize the risk of pollution problems from such effluents, it is necessary to accurately treat them before their discharge into the environment [10].

Direct Red 81 is a toxic sulphonated azo-based dye which makes it easily soluble in water. It is a widely used anionic or acidic dye among its category for colouring cellulosic fibres like cotton, rayon and jute. It is also used as a dyestuff for colouring leather and paper materials [11]. It is known for its carcinogenic nature and toxicity towards animals and humans and is selected as a synthetic model dye solution for experimentation which is widely used in many industries. It also has harmful effects on the skin and eyes [12]. The basic characteristics of DR 81 are presented in Table 1.

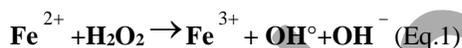
Dyes are removed from textile wastewater with great difficulty. Besides, many different physical and chemical methods, such as coagulation, flocculation, biological treatment, chemical oxidation, electrochemical technology, ion exchange, and

adsorption are used to decolorize the textile wastewaters [9, 15-20]. Combinations of processes, such as ozonation and flocculation, electrocoagulation and ion exchange, and chemical method followed by biodegradation process, are also used for efficient dye removal. These methods are somehow effective, but they are very expensive and inefficient [1].

Table 1: Basic characteristics of Direct Red 81 dye [13, 14]

Parameter	Characteristic
Chemical name	Direct Red 81
Molecular formula	C ₂₉ H ₁₉ N ₅ Na ₂ O ₈ S ₂
C.I. number	28,160
Classification	Diazo
Apparent colour	Red
Molecular weight	675.60g/mol
UV absorption	λ _{max} 397 nm λ _{max} 508 nm

Dyes are resistant to biodegradation process; therefore, conventional wastewater treatment methods are not capable of removing these compounds [21]. Advanced Oxidation Processes (AOPs) is an efficient method in which hydroxyl radicals (OH[•]) are produced which oxidizes the recalcitrant dyes to harmless end-products, such as H₂O and CO₂ [22, 23]. Fenton method is also highly applied due to its high efficiency and cost-effectiveness [24]. In the Fenton process, ferrous ions (Fe (II)) and hydrogen peroxide (H₂O₂) is used as reductants and oxidants, respectively [25]. The following reaction mainly occurs in this process [23]:



Many parameters influence the efficiency of this process. The rate of reaction highly depends on the optimization of the following parameters: pH, hydrogen peroxide concentration, Fe (II) concentration, and reaction time [13].

Robinson *et al.* showed that the dye removal efficiency of more than 90% was achieved to decolorize the textile wastewater, using a combined method of powder activated carbon-activated sludge [26]. In another study, Rahmani *et al.* demonstrated that 96% of Eriochrome Black T was removed, using electrocoagulation [27]. Dehghani *et al.* also showed that more than 98% removal of Reactive red 198 dye was obtained, using electrocoagulation [25].

Direct Red 81 dye, widely used in textile industries, is frequently detected in many water resources. In addition, great concern exists regarding water resources contamination and its adverse effects on the individuals' health. Since Direct Red 81 dye is persistent and conventional wastewater treatment is not capable of removing this dye efficiently, a suitable technique is required to remove the dyes from textile industries wastewater. Therefore, the present study

aims to (i) evaluate the feasibility of using Fenton process to remove Direct Red 81 dye from aqueous solutions and (ii) determine its removal efficiency at the optimum operating conditions.

MATERIALS AND METHODS

This cross-sectional study was performed on a laboratory scale, using a one-litre photochemical reactor. Direct Red 81 (C₂₉H₁₉N₅Na₂O₈S₂) was supplied by Hoechst Company, Germany. The rest of chemicals were purchased from Merck, Germany. The experiments were performed in triplicates. All data were presented based on the mean values. The studied parameters were showed in Table 2.

Table 2: The studied parameters and its values

parameter	values
reaction time (min)	15, 30, 45, 60, 90, and 120
H ₂ O ₂ concentration (mmol/L)	0.59, 1.18, 1.47, 2.94, and 4.41
Fe (II) concentration (mg/L)	10, 30, 50, 100, and 150
initial dye concentration (mg/L)	25, 50, 75, 100, and 150
pH	3, 5, 7, and 9

The optimal conditions were determined according to the maximum dye removal efficiency. Dye concentration was determined at the wavelength of 520 nm, using spectrophotometer (DR 5000) according to the standard method (No 2120) [28]. After all, the data were analyzed using the SPSS statistical software for Windows (version 16, SPSS Inc., Chicago, IL, USA). Pearson's correlation coefficient was used to analyze the relationship between these parameters.

The dye removal efficiency was calculated using the following equation:

$$R (\%) = \left(\frac{A_0 - A}{A_0} \right) \times 100 \quad (\text{Eq. 2})$$

where A₀ and A represented the initial dye concentration before and after the Fenton process, respectively.

RESULTS

Effect of pH

According to the results of this research, the maximum removal of the dye (5.87%) was at pH=3. In fact, negative removal efficiency occurred at pH=4 to 9.

Effect of ferrous ion (Fe (II)) concentration

The effect of Fe (II) on Direct Red 81 dye removal efficiency is shown in Fig. 1. The results indicated that as Fe (II) concentration increased from 10 to 150mg/L, the removal efficiency of Direct Red 81 dye decreased from 80.28% to 5.58%. It can be concluded that the optimum Fe (II) concentration was at 10mg/l.

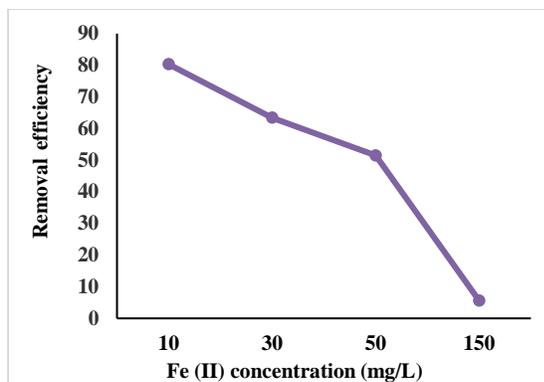


Fig. 1: The effect of ferrous ion Fe (II) on the removal of Direct Red 81 dye by Fenton process (pH=3, H₂O₂ concentration=1.47mmol/L, initial dye concentration=50mg/L, contact time=30 min)

Effect of hydrogen peroxide (H₂O₂) concentration

The effect of H₂O₂ on Direct Red 81 dye removal efficiency is presented in Fig. 2. The results demonstrated that removal efficiency was increased by increasing H₂O₂ concentration from 0.59 to 1.47mmol/L. Additionally, the maximum removal efficiency (70.02%) occurred at the H₂O₂ concentration of 1.47mmol/L. However, a further increase in H₂O₂ concentration (more than 1.47mmol/L) considerably decreased the removal rate.

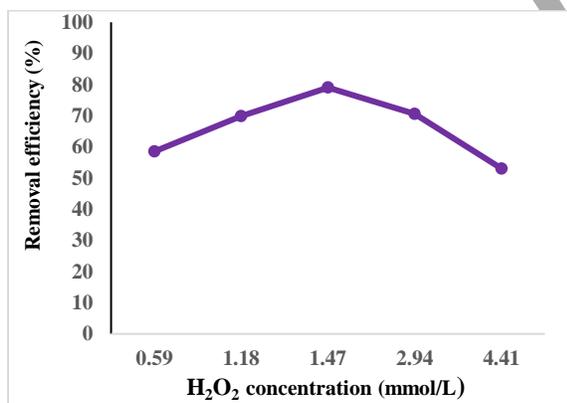


Fig. 2: The effect of hydrogen peroxide (H₂O₂) on the removal of Direct Red 81 dye by Fenton process (pH=3, Fe(II) concentration=10mg/L, initial dye concentration=50mg/L, contact time=30 min)

Effect of the initial concentration of Direct Red 81 dye

The effect of Direct Red 81 dye concentration on the removal efficiency is shown in Fig. 3. According to the results, the removal efficiency of Direct Red 81 dye was almost constant (about 59%) at the initial dye concentration of 25 to 50mg/L. After that, as the dye concentration was increased from 50 to 100mg/L, the removal efficiency increased to 83.88%. However,

increasing the initial dye concentration from 100 to 150mg/L decreased the removal rate (70.72%).

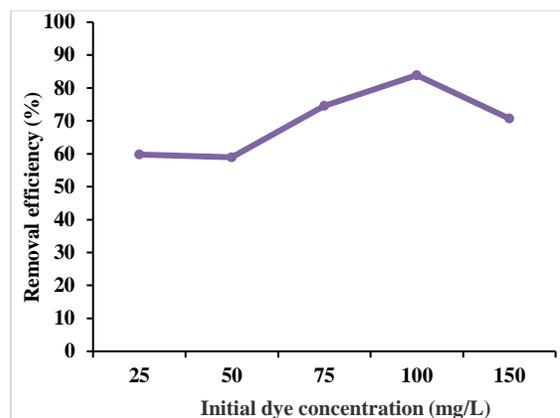


Fig. 3: The effect of initial dye concentration on the removal of Direct Red 81 dye by Fenton process (pH=3, Fe (II) concentration=10mg/L, H₂O₂ concentration=1.47mmol/L, contact time=30 min)

Effect of reaction time

The effect of reaction time on Direct Red 81 dye removal efficiency is shown in Fig. 4. Based on this Figure, the removal efficiency increased with increased reaction time. At first, the removal efficiency of dye increased significantly as the reaction time increased from 15 to 45 min. After that, the increasing trend of the removal rate became slower and its degradation reached a plateau (from 45 to 120 min).

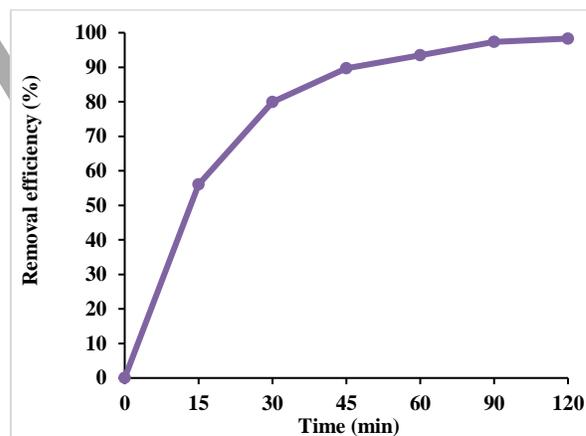


Fig. 4: The effect of reaction time on the removal of Direct Red 81 dye by Fenton process (pH=3, Fe (II) concentration=10mg/L, H₂O₂ concentration=1.47mmol/L, initial dye concentration=100mg/L)

DISCUSSION

Dyes are notorious as hazardous substances because most of them are toxic and considered to be resistant to biodegradation. Traditionally, the treatment of

solutions containing soluble dyes is performed by biochemical and coagulation processes. Recently, advanced oxidation processes (AOP) have received considerable attention because it is possible to degrade the organic compounds and colour from wastewaters. OH° radicals can be produced by fenton's reaction of H_2O_2 with Fe (II) salt. The actual dye wastewater has a wide range of initial pH values, and the solution pH is an important operating parameter affecting the removal efficiency in AOP.[29]

Basically, many chemical reaction rates depend on pH. The results of the present study indicated that the decolorization rate decreased with increasing pH. In AOPs, production of hydroxyl radicals is highly affected by pH. Based on the results of this research, higher hydroxyl radicals were produced under acidic conditions. A change in pH of the solution involves a variation of the concentration of Fe (II); therefore, the rate of production of OH radicals responsible for oxidation dyes will be restricted. The reaction rates of fenton oxidation of the dyes are rather slow in alkalinity medium, while they are fast in acidic medium.[29] The maximum dye removal efficiency was at pH=3. pH<3 may decrease the efficiency of decolorization due to the reaction of hydroxyl radicals with H^+ ions. Bahmani *et al.* declared the same results for the removal of Remazol Black-B (RB-B) [30]. Dehghani *et al.* reported a decrease in pH; also, the dye removal increased and optimum pH was at 3[31]. Another study showed that penicillin G removal in the aqueous solution was relatively high at pH = 3[32].

In our study, once the concentration of Fe (II) was increased up to the specified level (optimal concentration=10mg/L), the feasibility of OH° production and decolourization rate was increased. However, excessive increase of Fe (II) concentration had an inhibitory effect on the hydroxyl radical production, acted as OH° scavenger, and reduced the efficiency of chemical compounds degradation. Similar results were also obtained in many previous studies [30, 33]. In a study conducted to remove DB71 from azo dye wastewater by fenton's oxidation to see the effect of Fe (II) dosage in the fenton process and to determine the most appropriate concentration of Fe (II), some experiments were performed at different concentrations of Fe (II) from 0 to 50mgL⁻¹ at an initial pH of 3. Removal efficiency decreased when H_2O_2 addition was higher than 125mgL⁻¹. Excess H_2O_2 interferes with the measurement of COD. Additionally, above the limiting point, OH efficiently reacts with H_2O_2 and produces HO_2 results in negligible contribution [29]

In the current study, the optimal H_2O_2 concentration was 50mg/L. Excessive addition of H_2O_2 may promote OH° formation and act as hydroxyl radicals'

scavengers, eventually reducing the oxidation efficiency. The optimal molar ratio of H_2O_2 to Fe (II) is equal to 5. Emami *et al.* showed that the maximum removal of Reactive Red 120 using the Fenton method was at $\text{H}_2\text{O}_2/\text{Fe (II)}$ molar ratio of 10 [33]. One study showed that by increasing the concentration of Fe (II), H_2O_2 and initial dye, the removal efficiency was increased [34].

Our study revealed that the higher dye concentration increased the removal efficiency. According to the results, the maximum removal efficiency of Direct Red 81 dye was at the initial concentration of 100mg/L. The same results were obtained by Zonoozi *et al.*, reporting the maximum removal of Acid Blue 292 at the initial concentration of 150mg/L, using poly-aluminium [35]. Removal efficiencies of DB71 were evaluated at 125mg/L H_2O_2 and 3mg/L Fe (II) at pH 3. The colour removal efficiency gradually decreased with an increase in the initial concentration. [29]

Basically, optimal reaction time is a very important parameter for any chemical reaction. The results of this study showed that the dye removal efficiency was directly proportional to the reaction time. Based on the results illustrated in Fig. 4, the maximum decolorization rate (98.29%) was at 120 min reaction time. According to the results, at first, the rate of Direct Red 81 dye removal increased very fast as the contact time increased. After 45 min, however, this increasing trend slowed down until it reached a plateau (Fig. 4). At equilibrium, the degradation reached a plateau (89.68% at 45 min). Therefore, from the economic point of view, the reaction time of 45 min was optimal for removal of Direct Red 81 dye, using Fenton method. Robinson *et al.* also showed that the dye removal was optimal at 30 min using activated carbon and activated sludge simultaneously [26]. Another study also indicated that the highest removal of Yellow Alkaline dye 2 was at the optimal reaction time of 11 min [36]. Similarly, the removal efficiency of Reactive azo dye was 89.5%, using Anaerobic Batch Reactor (ABR) system [37]. Dehghani *et al.* also showed that the Reactive Red 198 dye removal efficiency from aqueous solutions was more than 99% at optimal conditions (pH = 3, Fe (II) = 10mg/L, H_2O_2 = 75mg/L, initial dye concentration = 50 mg/L, and reaction time = 120 minutes)[38].

CONCLUSION

In conclusion, our results suggested that Fenton process was an appropriate method for the removal of Direct Red 81 dye with more than 98% efficiency. Dye removal efficiency decreased by increasing pH and increased by increasing the reaction time. Overall, the optimal conditions for dye removal were pH of 3, Fe (II) concentration of 10mg/L, H_2O_2 concentration of

50mg/L, and dye concentration of 100mg/L. Although the highest dye removal efficiency was achieved after 120 min, considering cost-effectiveness, 45 min reaction time was appropriate for the removal efficiency of more than 89%. Therefore, the results demonstrated the usability of Fenton process as a promising method for the removal of Direct Red 81 dye from aqueous solutions.

ETHICAL ISSUES

Ethical issues such as plagiarism have been observed by authors.

CONFLICT OF INTEREST

There was no conflict of interest.

AUTHORS' CONTRIBUTION

The overall implementation of this study, including design, experiments, data analysis, and manuscript preparation, was the result of the corresponding author's efforts. All the authors made an extensive contribution to the review and finalization of this manuscript. All the authors also read and approved the final manuscript.

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