COD removal from disperse blue dye 79 in wastewater by using Ozone-Fenton process

A. E Mohammed 1, H H Hamed 1, W M Sh Alabdraba 2 and O M Ali 1
1Technical Institute/ Hawija, Northern Technical University, Iraq
2Environmental Engineering Department, Tikrit University, Iraq
Corresponding Author Email: awadesa76@gmail.com

Abstract. Nowadays, one of the major problems that can face humankind is the water pollution. In this regard, making this natural resource a fresh and unpolluted is a major social and economic concern. However, a great attention has been given for the removal of organic pollutants discharged within wastewater by using advanced oxidation processes (AOPs) based on generation hydroxyl radicals. Among these, Ozone-Fenton (O₃/H₂O₂/Fe²⁺) process used in this study as AOPs to remove chemical oxygen demand (COD) from Disperse Blue 79 (DB 79) dye in wastewater. Also, the effect of various experimental parameters such as ozone mass flow rate, mole ratio (mole H₂O₂/mole Fe²⁺), and reaction time on the COD removal efficiency of DB 79 were studied with constant initial concentration 60 mg/L of DB 79. Based on the results obtained in this study, the maximum COD removal efficiency was 85% achieved at optimum operating parameters are 288 mg/L h ozone mass flow rate, 33.53 mole ratio (150 mg/L H₂O₂, 20 mg/L Fe²⁺), and 60 min reaction time. All Ozone-Fenton experiments were performed using Jar test apparatus at room temperature (23℃ ± 2) and atmospheric pressure (1 atm). Finally, Ozone-Fenton offered an effective solution as AOPs for degrading pollutant especially DB 79 discharged within wastewater from various industrial processes.

Keywords: AOPs, Ozone-Fenton, DB79, COD removal.

1. Introduction

Many of chemical dyes present in different fields of daily life and used in variety of industrial processes (textile, food, pharmaceutical, pulp, paper manufacturing, leather treatment, dyeing, and printing) where, the presence of these dyes within water can be causing coloring, undesirable, and toxic [1]. The quantity and quality of wastewaters discharged from these industries are a major concern for public and governments [2]. Effluent dyes from textile industries and dyestuff manufacturing processes can be cause carcinogenic effects on microbial population [3]. Dyeing and finishing processes are the major pollution of textile wastewater. A wide range of organic complex structure from dyestuffs and chemicals are fed to these processes [4]. Science 1970s the using of dyes and especially disperse dyes such as Disperse Blue (DB 79) dye have been increased widely in the textile industry and various processes are nylon, acrylic fiber, and polyester [5]. According to statistics about 40,000 L of water and 700 kg of different chemicals are used to convert 1 ton of skins or hides into leather could generated about 32,000 L of wastewater [6]. Therefore, one of the most important aspects of dyes wastewater treatment before discharging into the environment are to remove theses pollutants and make it fresh and unpolluted.
Many of conventional treatment methods used for wastewater treatment such as biological treatment, reverse osmosis, carbon adsorption, air stripping, pure ozone oxidation [7]. The limitations of these conventional methods have been overcome by using the application of advanced oxidation processes (AOPs) [8], which defined firstly by Glaze [9], and then sited by other researchers. AOPs are powerful, effective and alternative methods used for treating pollutants that cannot be treating biologically that generate hydroxyl radicals, where these species are non-selective, strong oxidants that can degrade chemical organic compounds discharged within wastewater from variety of industrial dyeing processes [10]. AOPs are commonly and widely utilized for removing color and COD from industrial dyeing wastewater [11]–[13]. Recently, different combined AOPs have been used, and developed by several researchers as alternative methods for wastewater treatment to remove harmful organic pollutants and also, to reduce the contact time and economic cost [14]–[16]. Indeed, AOPs can induce decolorization and transformation of dissolved organic compounds to carbon dioxide mineral acids [17]. Ozonation is a type of AOPs and it is powerful, efficient, environment-friendly method used in wastewater treatment and has been employed for many decades and cited by researchers in review articles [18], [19]. Ozone is a strong oxidant with a reduction potential (E° = 2.07 V) capable to oxidize organic and inorganic compounds as shown in equation (1):

\[ \text{O}_3 + 2\text{H}^+ + 2e \leftrightarrow \text{O}_2 + \text{H}_2\text{O} \quad \text{E}^\circ = 2.07 \text{ V} \quad (1) \]

The generation of hydroxyl radicals can be accelerated by combining O₃, H₂O₂, O₃/UV, H₂O₂/UVO₃/H₂O₂ to treat textile wastewater [4]. The hydroxyl radicals generated in AOPs attack the organic molecules by abstracting hydrogen atom from the molecule [20]. Carey [21] described a common pathway for the degradation of organics by the hydroxyl radicals as follow:

\[ \text{OH}^- + \text{RH} \rightarrow \text{H}_2\text{O} + \text{R}^\cdot \quad (2) \]
\[ \text{R}^\cdot + \text{H}_2\text{O}_2 \rightarrow \text{ROH} + \text{OH}^- \quad (3) \]
\[ \text{R}^\cdot + \text{O}_2 \rightarrow \text{ROO}^\cdot \quad (4) \]
\[ \text{ROO}^\cdot + \text{RH} \rightarrow \text{ROOH} + \text{R}^\cdot \quad (5) \]

The AOP offers several advantages are the ability to treat organic compounds in wastewater with a very low organic load in part per billion (ppb). The major (main) advantage of AOP is the transformation of chemical organic compounds to simple compounds or to CO₂ and H₂O without formation sludge. Besides that AOP has a disadvantage is highly expensive cost [22].

Many of researchers used Ozone-Fenton in wastewater treatment such as Mashal, et al., [23] were studied the removal of COD from wastewater, they found that best removal achieved after reaction time about 30 min. Abu Amr and Abdul Aziz [24] treated textile wastewater, they found that maximum COD removal efficiency was 65% achieved at 90 min reaction time. Abu Amr and Abdul Aziz [25] were degraded COD from industrial wastewater, they found that COD removal efficiency reached up to 87% at optimum parameters (1700 mg/L H₂O₂, 2800 mg/L Fe²⁺, 80 mg/L O₃, pH=8.3). Ahmad et al., [26] were studied the COD removal from industrial oil refinery, they found that maximum COD removal efficiency was 69.87% achieved at optimum operating parameters are 90 min reaction time and 313 mg/L h of ozone.

The major target from this study was to remove COD from DB79 dye in wastewater by using Ozone-Fenton (O₃/H₂O₂/Fe²⁺) as AOPs and also examining the effect of key operational parameters such as ozone mass flow rate, mole ratio of hydrogen peroxide to ferrous ions (H₂O₂/Fe²⁺), and reaction time on COD removal efficiency. The effectiveness of the processes was measured by COD removal.

2. Experimental and Methodology

2.1. Preparation of wastewater.

The synthetic wastewater dye solution used in this study was prepared at constant initial dye concentration of 60 mg/L by dissolving 60 mg of (DB 79) dye into 1L of distilled water. The main characteristics of DB 79 used in this study were the same as that used in pervious literature Hussein et al., [27], as shown in Table 1:
Table 1. The main characteristics of DB 79 dye [27].

| Item                        | Structure Dye |
|-----------------------------|---------------|
| Linear Formula              | C_{23}H_{25}BrN_{6}O_{10} |
| Molecular Weight            | 625.38 g/mole  |
| Color Index Number          | 11344         |

2.2. Materials and Chemicals.
For Ozone-Fenton (O\textsubscript{3}/H\textsubscript{2}O\textsubscript{2}/Fe\textsuperscript{2+}) process, the materials and chemicals used in this study are hydrogen peroxide solution (H\textsubscript{2}O\textsubscript{2} 50% w/v), ferrous sulfate heptahydrate (FeSO\textsubscript{4}·7H\textsubscript{2}O), sulfuric acid (H\textsubscript{2}SO\textsubscript{4}, 98%), mercuric sulfate (HgSO\textsubscript{4}), sodium hydroxide (NaOH), potassium dichromate (K\textsubscript{2}Cr\textsubscript{2}O\textsubscript{7}), silver sulfate (AgSO\textsubscript{4}), ozone (O\textsubscript{3}), and distilled water. All chemical used were in high purity and analytical grade without purification.

2.3. Ozone-Fenton Experimental Procedure.
All Ozone-Fenton experiments were performed using Jar test apparatus which consists of four cylindrical glass beakers (2000 ml capacity for each beaker) with a magnetic stirrer operates at different operational mixing speeds ranged from 0 to 200 rpm and designed to achieve perfect, and efficient contact between the gas and liquid. Ozone generated at various flow rates (mg/L. h) by using ozone generator apparatus (OZOMAX, 1 VTTL, Kanada) was supplied and injected through a diffuser located at the bottom of the four beakers (bottom of each beaker). A pure air (free oil) generated by using air compressor device with a flow meter connected with the compressor to control the outlet air flow rate from the compressor. The compressor connected with ozone generator and Jar test via plastic pipes. A schematic diagram for experimental set up used in this study for Ozone-Fenton experiments is shown in figure 1.

Firstly, about 80 ml sample of wastewater with constant initial concentration 60 mg/L was added into each beaker of Jar test. Then, different doses of ozone, hydrogen peroxide, and ferrous ions were mixed with the sample solution into each beaker. The final volume of the solution into each beaker completed to 2000 ml by adding raw laboratory tab water. Before starting the reaction, the solution pH was adjusted to the required value (pH= 10 basic condition) by adding a few drops of NaOH and/or H\textsubscript{2}SO\textsubscript{4} and measured using pH-meter. The solution was agitated and mixed vigorously at the starting of reaction for a period 5 minutes at 5 rpm to achieve efficient mixing and more homogeneity. Finally, the reaction proceeded and continued during the whole reaction time (60 minute) and then stopped to settle completely. Moreover, the effect of various experimental parameters such as ozone mass flow rate, mole ratio (mole H\textsubscript{2}O\textsubscript{2}/mole Fe\textsuperscript{2+}), and reaction time on the COD removal efficiency of DB 79 were studied. In this study the experimental runs were carried out by changes in parameter, while keeping other parameters constant. The optimum parameters for each one was determined while keeping the others constant, i.e. for determining optimum ozone mass flow rate we add constant doses from each of Fe\textsuperscript{2+} and H\textsubscript{2}O\textsubscript{2} then, starts the operation and so on.
2.4. Analytical Methodology.
The samples were taken for measuring of COD (mg/L) at various periods time (20, 40, 60 min) during the reaction were measured using COD apparatus (COD VARIO, Lovibond, MD 200, Japan) according to the standard methods via a closed reflux titrimetric method [28]. About 2.5 ml of sample was mixed with (2.5 ml H\textsubscript{2}SO\textsubscript{4} + 1.5 ml K\textsubscript{2}Cr\textsubscript{2}O\textsubscript{7}) into a glass test tube and heated for 120 min at 150\degreeC via heater with an operation temperature (0-200\degreeC). After completion the heating operation, solution was cooled and taken for COD measuring. The COD removal efficiency was calculated by using equation (6).

\[
\% \text{COD} = \frac{\text{COD}_i - \text{COD}_t}{\text{COD}_i} \times 100
\]  
(6)

Where, \(\text{COD}_i\) is the initial COD (260 mg/L); \(\text{COD}_t\) is the COD measured at time t.

3. Results and Discussion

3.1. Effect of Ozone Combined with Hydrogen Peroxide and Ferrous Ions.
The effect of ozone mass flow rate combined with hydrogen peroxide and ferrous ions on the COD removal efficiency of DB79 in wastewater by using Ozone-Fenton process was studied, the results are shown in figures. 2(a & b). The results presented in figure 2a shows the effect of ozone mass flow rate on the COD removal efficiency with varying hydrogen peroxide while keeping other parameters constant (20 mg/L \(\text{Fe}^{2+}\), 60 min reaction time). Also, the results presented in figure 2b shows the effect of ozone mass flow rate on the COD removal efficiency with varying ferrous ions, keeping the other parameters constant (150 mg/L H\textsubscript{2}O\textsubscript{2}, 60 min reaction time). As can be seen in figure 2a, an increasing in ozone mass flow rate from 96 to 288 mg/L. h the COD removal efficiency also increased from 65 to 85\%, respectively with keeping the other parameters constant (150 mg/L H\textsubscript{2}O\textsubscript{2}, and 20 mg/L \(\text{Fe}^{2+}\)). Further increasing in ozone mass flow rate more than 288 mg/L. h the COD removal efficiency decreased. The same behavior of COD removal efficiency could be observed in figure 2a with varying other doses of hydrogen peroxide, but in lower efficiency. It is clear that the addition of hydrogen peroxide to the ozone within wastewater will enhance the decomposition of O\textsubscript{3} and this can be led to hydroxyl radical formation, as shown in equation (7).

\[
\text{O}_3 + \text{H}_2\text{O} \rightarrow 2\text{OH} + \text{O}_2
\]  
(7)

The mechanism oxidation with ozone that ozone (O\textsubscript{3}) molecular can reacts directly with organic pollutant in water or indirectly with ozone decomposed and this can lead to generate hydroxyl radicals, according to the following reaction. Ozone can react with pollutants present in wastewater with two different ways are called direct and indirect reaction. In the first one (e.g. direct reaction) ozone
molecules attacks and break the unsaturated bonds. While, indirect reaction hydroxyl radicals generated react with organic pollutant. Keep in mind and taking into account that indirect reaction occurred at basic condition (high pH media).

However, taking into account the decomposition rate of ozone strongly depended on pH, where better decomposition rate of ozone can take place at basic conditions (pH $\geq$ 9) because an increasing in pH can produce higher rate of hydroxyl radical production [29]. The hydroxide ions catalyze the decomposition of ozone to produce hydroxyl radicals which are highly reactive species, oxidizing, non-selective, and have a higher oxidation potential than that for ozone (2.80 V) [30].

![Figure 2](image)

**Figure 2:** Effect of ozone mass flow rate (O$_3$) on COD removal efficiency of DB79 with varying: (a) Hydrogen peroxide (H$_2$O$_2$) and constant (Fe$^{2+}$ 20 mg/L, 60 min reaction time) (b) Ferrous ions (Fe$^{2+}$) and constant (150 mg/L H$_2$O$_2$, 60 min reaction time).

### 3.2. Effect of Mole Ratio.

The effect of mole ratio of hydrogen peroxides to ferrous ions H$_2$O$_2$/Fe$^{2+}$, plays an important role in COD removal efficiency of DB 79 in wastewater by using Ozone-Fenton process. However, it is very important to determine the optimal dosage of hydrogen peroxide in the Ozone-Fenton process from a critical point due to the high cost of hydrogen peroxide. In this study the effect of various ranges of
mole ratio from 7 to 67 were considered with varying ozone mass flow rate on the COD removal efficiency as shown in figure 3. The results presented in figure 3 shows an increasing in mole ratio to 33.35 the COD removal efficiency increased to the highest value of 85% at 288 mg/L hr ozone mass flow rate. Further increasing in mole ratio more than this limit the COD removal efficiency sharply decreased. It can be explained that an increasing in mole ratio means an increasing in hydrogen peroxide more than the limited required and decreasing ferrous ions from the limited required this can be led to a scavenger which slow down the COD removal efficiency. The results obtained in figure 3 indicated that maximum COD removal efficiency achieved was 85% at 33.53 mole ratio corresponds to (150 mg/L H₂O₂, 20 mg/L Fe²⁺).

![Figure 3](image_url)

**Figure 3:** Effect of ozone mass flow rate (O₃) and mole ratio (H₂O₂/Fe²⁺) on COD removal efficiency of DB79.

### 3.3. Effect of reaction time.

The contact time plays an important role, and COD removal of DB79 with Ozone-Fenton process strongly dependent on it. Hence, the effect of reaction time on COD removal efficiency was studied in the range of 0 to 60 min separately with each parameter such as ozone mass flow rate, hydrogen peroxide, and ferrous ions. The effect of reaction time with ozone mass flow rate, hydrogen peroxide, and ferrous ions on COD removal efficiency has been studied as shown in figure 4a, figure 4b, and figure 4c, respectively. As can be seen in figure 4a, an increasing in reaction time from 20 to 60 min at optimum ozone mass flow rate 288 mg/L hr caused an increasing in COD removal efficiency from 64 to 85%, respectively keeping in mind the other parameters constant (150 H₂O₂, 20 Fe²⁺). Further increasing in reaction time more than 40 min the COD removal efficiency slightly increases and becomes more stable. It can be explained that more reaction time provides enough contact time between the reagents to complete the oxidation reaction. According to the results presented in figures 4 (a, b, & c) the COD removal efficiency was very efficient at the start of operation (start of reaction time) till reaches to a reaction time of 40 min, further increasing a slightly response occur of COD removal efficiency. However, the same behavior of COD removal efficiency against reaction time can be seen in figure 4b, and figure 4c. Based on results presented by many of researchers revealed that sufficient reaction time about 30-90 min required to enhance COD removal [25],[26].
Figure 4. Effect of reaction time on COD removal efficiency of DB79 with varying: (a) ozone mass flow rate (O$_3$) and constant (H$_2$O$_2$ 150 mg/L, Fe$^{2+}$ 20 mg/L) (b) Hydrogen peroxide (H$_2$O$_2$) and constant (288 mg/L. h O$_3$, 20 mg/L Fe$^{2+}$) (c) Ferrous ions (Fe$^{2+}$) and constant (288 mg/L. h O$_3$, 150 mg/L H$_2$O$_2$).
4. Conclusions
Advanced oxidation processes (AOPs) are a good, and efficient option for wastewater treatment compared with the conventional treatment methods. The main target in this study was to degrade COD from DB79 in wastewater by using Ozone-Fenton process ($O_3/H_2O_2/Fe^{2+}$) as AOPs. And, also to examine the effect of operating parameters on the COD removal efficiency. According to the results obtained in this study, Ozone-Fenton process was effective, and quick performed successfully in COD removal efficiency up to 85% was achieved at optimum operating parameters were determined and found to be 288 mg/L.h ozone mass flow rate, 33.53 mole ratio (150 mg/L $H_2O_2$, 20 mg/L $Fe^{2+}$), and 60 min reaction time. The presence of hydroxyl radicals scavengers slow down the COD degradation efficiency. The combination of Ozone-Fenton was found to be a proper, and alternative method used in degrading DB 79 successfully in wastewater. Moreover, a great attention should be taken into account in the future by researchers to fill some specific gaps such as pH effect, reaction intermediate, scale up parameters, and cost effectiveness.

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