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Performance evaluation of combined O_3/Fenton process on decolorization and COD removal of disperse blue 79 dye from aqueous solution

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ABSTRACT

Water plays a vital, essential and important role in our life fields. Nowadays, this natural resource became scare, making a fresh and availability is a major social and environmental concern. Dyes and synthetic dyes are the major industrial pollutants and water contaminants. The wastewater discharged contains various contaminants characterized by a high content of organic matter, additives, surfactants and dyes. Effluent streams which contain dyes within wastewater make it polluted, harmful, carcinogenic and environmentally problematic. However, combined advanced oxidation processes (AOPs) such as (O_3/H_2O_2/Fe^{2+}) were used and applied in this study as a novel wastewater treatment to remove color (decolorization) and chemical oxygen demand (COD) from Disperse Blue 79 (DB79) within synthetic wastewater solution. The aim of this study was to evaluate the performance efficiency of combined AOPs and examine the effect of various operational parameters such as ozone mass flow rate, hydrogen peroxide to ferrous ions (H_2O_2/Fe^{2+}) mole ratio and contact time on decolorization and COD removal efficiency. According to the results obtained revealed that 93% and 85% color and COD removal efficiency respectively achieved at optimum operating conditions are 288 mg/L h ozone mass flow rate, 33.53 mole ratio (H_2O_2/Fe^{2+}) and 60 min contact time. Overall it can be established that O_3/H_2O_2/Fe^{2+} process plays an important role in obtaining good results and had better performance in color and COD removal.

Keywords: Decolorization; COD; DB79; Advanced oxidation process; O_3/H_2O_2/Fe^{2+}

1. Introduction

One of the major concerns to water quality is related to the chemical contaminants detected in both municipal and industrial textile wastewater, where most of these pollutants (synthetic and natural organic chemicals) such as pharmaceuticals, pesticides, endocrine disruptor, personal care products, dyes and synthetic dyes, entered into the aquatic medium in various ways that can be transported and distributed in the water cycle [1,2]. Furthermore, textile industry is one of the most significant sectors of the global economic particularly in countries such as China, India, Malaysia, Italy, Turkey and Pakistan [3], besides that, one of the major sources of environmental pollution and dyes are among the prominent pollutants present within textile effluents [4]. Moreover, textile wastewater consists of a high variable mixture of various polluted substances, including dyes discharge to the environment [5] which can be characterized by a high content of dyestuffs, salts, suspended solid, high chemical oxygen demand (COD) and pH fluctuating. Dyes wastewater are the major environmentally obstacle for the growing of the textile industry besides the other minor issues like solid waste and resource management. Industrial wastewaters originating from synthetic dyes production and the application of these dyes can be polluted the surrounding ecosystem due

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to their highly toxicity and carcinogenic in nature [6,7]. Approximately, about 800,000 tons of synthetic dyes can be consumed in the textile industry in the whole world [8]. Besides that, large amounts of dyes produced annually, and applied in many industrial processes include food, leather, paper, pharmaceutical and textile industries. According to the statistics, more than 100,000 dyes commercially available with an estimated annual production about 700,000 tons, in which that 50% of these dyes are lost during the dyeing processes [9]. However, from 1970s the using of disperse dyes have been increasing widely, where DB79 is considered as one of the most colorants applicable in the textile industry and can be used in different processes such as nylon, acrylic fibers and polyester. The release of disperse dyes within wastewater into environment is a concern problem due to color change of natural waters and significantly high toxicity of these dyes. Therefore, the color problems of some textile wastewaters caused due to the residual dyes during the dyeing operations which need more efforts to study and investigate [10]. The treatment of various wastewater pollutants have been applied and treated successfully by using advanced oxidation processes (AOPs), such as textile wastewater [11], pharmaceutical wastewater, pulp and paper industrial processes [12], phenolic wastewater [13]. In such case the using of AOPs are an alternative and effective method commonly and widely utilized for the overall removal efficiency of these contaminants. AOPs have been commonly and widely used for the color removing from textile wastewater effluent. The mechanism of AOPs in degrading the organic pollutants based on the generation of highly reactive free radicals which are non-selective reactive species, and capable to oxidize contaminants into mineral end-products, yielding CO_2 and harmless substances [14]. However, the hydroxyl radical ($HO^{\bullet}$) generated by AOPs are able to oxidize and degrade the organic contaminants (RH), thus causing a chemical decomposition of these compounds as expressed by the following reactions [15]:

$$HO^{\bullet} + RH \rightarrow H_2O + R \quad (1)$$

$$R^{\bullet} + Fe^{3+} \rightarrow R^{+} + Fe^{2+} \quad (2)$$

$$R^{+} + H_2O \rightarrow ROH + H^{+} \quad (3)$$

AOPs show explicit advantages compared to the conventional treatment methods as in removing the non-biodegradable organic substances and there is no problem of residual sludge disposal [16]. Recently, many of the researchers have been studied decolorization, COD removal and the application of AOPs on various dyes within wastewater. Kadir and Yildiz [17] studied the decolorization of direct dye (Sirius Blue SBRR) in textile wastewater by using ozonation process, a maximum color removal efficiency was marked at basic conditions pH = 12. Abu Amr and Abdul Aziz [18] treated a stabilized leachate using ozone-Fenton method as AOPs, also they found that, the decolorization efficiency was decreased with increasing mole ratio more than 1. Wijannarong et al. [19] were applied AOPs for removing reactive dyes from industrial textile dyeing effluents, and found that more than 90% of removal color efficiency was achieved after 6 h contact time. Ahmad et al. [20] were decolorized and degraded the petroleum wastewaters by using AOPs, a maximum color, COD, and phenol removal efficiency about 85%, 77% and 75%, respectively was investigated using Ozone/Fenton method. Hussein et al. [21] was decolorized and degraded DB79 from aqueous solution using Fenton process as AOPs, a better results could obtained in color and COD removal of about 85% and 75%, respectively.

The main target of this present work was to evaluate the performance of ($O_3/H_2O_2/Fe^{2+}$) as AOPs on the decolorization and COD removal efficiency of DB79. Besides that, an operational parameter such as ozone mass flow rate, hydrogen peroxide to ferrous ions (H_2O_2/Fe^{2+}) mole ratio and contact time were examined to find optimum decolorization COD removal state.

2. Materials and methods

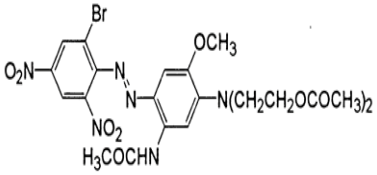
2.1. Materials and chemicals used

In this study, synthetic wastewater dyes solution was prepared at constant initial concentration 60 mg/L DB79 by dissolving 60 mg of DB 79 into 1 L of laboratory tab water. The main characteristics of DB79 dye and its structure are the same characteristics that used in previous literature [21] are shown in Table 1. The chemicals used are hydrogen peroxide (H_2O_2) (50% w/v), ferrous sulfate hepta hydrate ($FeSO_4{\cdot}7H_2O$), sulfuric acid (H_2SO_4), sodium hydroxide (NaOH) were used in a high purity and analytical grade available without further purification.

2.2. Experimental and methodology

All the experimental runs were performed using Jar test apparatus, which consists of four cylindrical glass beakers (2 L capacity for each beaker) with a magnetic stirrer operates with an operational mixing speed from 0–200 rpm, and designed to achieve good contacting and perfect mixing between the liquid and gas phase. A jar test photo used in this work is shown in Fig. 1. Ozone system consists of air

Table 1
DB79 Characteristics [21]

Structure	
Linear formula	$C_{23}H_{25}BrN_6O_{10}$
Molecular weight	625.38 g/mol
Color index number	11,344
pH	6.5–8.4
Color[a] (AU)	0.9
COD[b]	260 mg/L

[a]Absorbance unit measured at wavelength 565 nm.
[b]Initial concentration of COD.

compressor (free-oil) in order to generate pure air (free-oil) connected with ozone generator apparatus (OZOMAX, 1 VTTL, Canada) via plastic pipe to transport the air from the compressor to the ozone generator. A flow meter connecting with the compressor is in order to control the outlet air flow rate from the compressor. Jar test beakers connected with the ozone generator to transport the ozone generated via plastic pipe. The zone generated (mg/L h) was injected through diffusers located at the bottom of the four reactors (bottom of each beaker) at various flow rates.

In each experiment, the four cylindrical glass beakers jar were filled with 80 ml of the sample dye solution at 60 mg/L, and the other doses such as H_2O_2, O_3 and Fe^{2+} at required dosage added into the solution. The final volume of solution completed to 2 L (2,000 ml) by adding a few drops of raw laboratory tab water. Initially, before each experiment, the solution pH level was measured using pH meter and adjusted to the requirement pH value, basic condition (pH = 9) by adding few drops of NaOH. Then, the solution agitated and mixed at 50 rpm for 5 min in order to ensure perfect mixing and high homogeneity. For all experiments, the operation time of each experiment was 60 min. As the experiment progressed and continued for 60 min. Finally, 10 ml of sample was taken at different duration intervals (each 20 min) in order to examine the color and COD parameters. Color and COD measurements were measured in the same manner and apparatus described in literature [21] according to the standard procedures and by using UV/Vis spectrophotometer (Thermo Scientific, Genesys 20, German). The absorbance of each sample withdrawn at various time intervals was monitoring and measured at maximum absorption at the wavelength for the DB79 dye (λ_{max} = 565 nm) by using UV-spectrophotometer. The experimental runs were performed by changes in parameter, while keeping other parameters constant. In this work optimum condition alone for each parameter was estimated while other parameters were constant, that is, for estimating optimum ozone mass flow rate we add constant doses from each of H_2O_2, and Fe^{2+} then start the operation. The experiments, were carried out at room temperature 23 (∓ 2)°C and atmospheric pressure at (1 atm).

Fig. 1. Jar Test apparatus.

3. Results and discussions

3.1. Effect of mole ratio H_2O_2/Fe^{2+}

The initial concentrations of H_2O_2 and Fe^{2+} are very important parameters to optimize the estimation of the best H_2O_2/Fe^{2+} mole ratio in order to achieve maximum decolorization and COD removal efficiency of DB79 in lower cost. So, the selection of optimum H_2O_2 concentration for the decolorization efficiency of DB79 has greater importance in critical point of view because of the high cost of H_2O_2 concentration. In this work the effect of the mole ratio on the decolorization and COD removal efficiency of DB79 from wastewater was studied with varying ozone mass flow rate. Initial concentrations of H_2O_2 are (50, 100 and 150 mg/L) and Fe^{2+} are (10, 20 and 30 mg/L) were used in this work. So, the influence of mole ratio on the decolorization and COD removal efficiency was examined at various ranges from 7 to 67 mole ratio as shown in Fig. 2 and 3, respectively. As shown in Fig. 2, the increasing mole ratio from 7.45 to 33.53 at constant ozone mass flow rate 288 mg/L. h the decoloriation efficiency enhanced and increased to achieve the maximum value 93% of color removal. A significant enhancement of decolorization efficiency was noticed when mole ratio was increased till reaching 33.53 which equivalent to (150 mg/L of H_2O_2 and 20 mg/L of Fe^{2+}). Further, increasing the mole ratio more than this limit the behavior of decolorization efficiency decreased due to the activity of hydroxyl radicals in oxidizing the organic substances decreased. And, also an increasing mole ratio means decreasing in initial concentrations of Fe^{2+} less than the optimum value. The same behavior can be seen in Fig. 3 which presents the COD removal efficiency increases with an increasing the mole ratio till reaching to 85% at 33.35 mole ratio which corresponds to (150 mg/L of H_2O_2 and 20 mg/L of Fe^{2+}).

These results are in agreement with previous literature [21,22] in which the optimum mole ratio obtained was 33.53 (150 mg/L of H_2O_2 and 20 mg/L of Fe^{2+}) that achieved

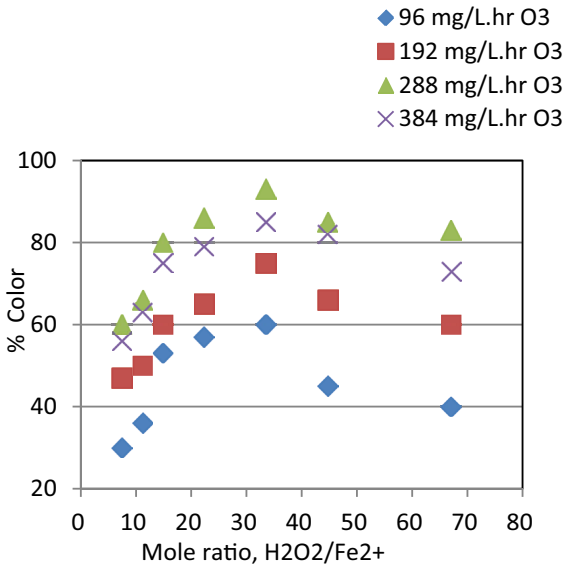


Fig. 2. Effect of mole ratio and ozone mass flow rate on decolorization efficiency at 60 min contact time.

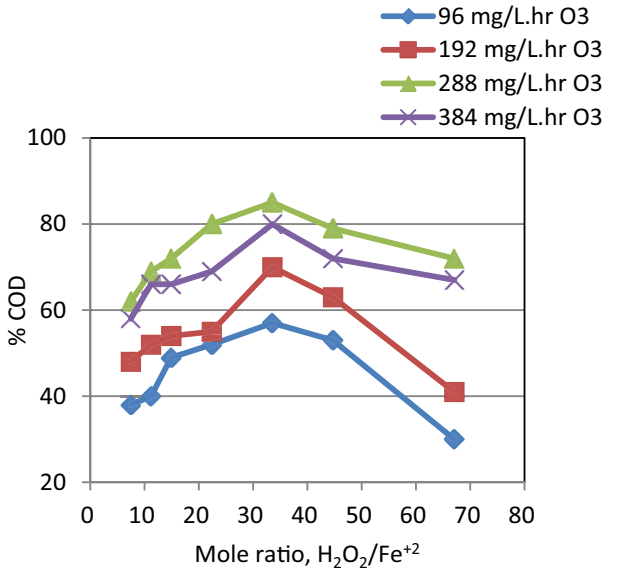


Fig. 3. Effect of mole ratio and ozone mass flow rate on COD removal efficiency at 60 min contact time.

maximum color and COD removal efficiency of DB79 from aqueous solutions. Other literatures such as Abu Amr and Abdul Aziz [18] are disagree with these results, where they found that optimum mole ratio was 1, further increasing more than this limit, the removal efficiency was decreased. The mole ratio was determined in this work was similar to 33.53 reported by Hussein et al. [21]. Besides that, optimum dosages of (H_2O_2 and Fe^{2+}) were determined in this study were lower than those reported by many of the works such as Hong et al. [23] found, that optimum H_2O_2 and Fe^{2+} dosages were 4,000 and 1,000 mg/L, respectively were applied in Fenton process for livestock wastewater treatment, achieved COD decreasing from 895 to 76 mg/L. Consequently, we can say optimal mole ratio is 33.53 equivalents to 150 mg/L H_2O_2 and 20 mg/L Fe^{2+}

3.2. Effect of ozone mass flow rate

To study the effect of ozone mass flow rate on the decolorization and COD removal efficiency of DB79 by $O_3/H_2O_2/Fe^{2+}$ as AOPs and to find optimum ozone mass flow rate dosage, a series of experiments were carried out at different doses of ozone mass flow rate are (96, 192, 288 and 384 mg/L h) as shown in Figs. 4a and b and 5a and b, respectively were performed at basic conditions (pH = 9). In this work, the effect of ozone mass flow rate on the decolorization efficiency was examined with varying the initial concentrations of H_2O_2 and Fe^{2+} in two parts. In the first part, ozone mass flow rate varied with varying the initial concentration of H_2O_2 (50, 100 and 150 mg/L), keeping both Fe^{2+} concentration and contact time are constant at 20 mg/L and 60 min, respectively as shown in Fig. 4a. While, in the second part, ozone mass flow rate was varied with varying the initial concentration of Fe^{2+} (10, 20 and 30 mg/L), keeping the other parameters constant such as H_2O_2 150 mg/L and 60 min contact time as shown in Fig. 4b. The effect of the same parameters was varied and examined

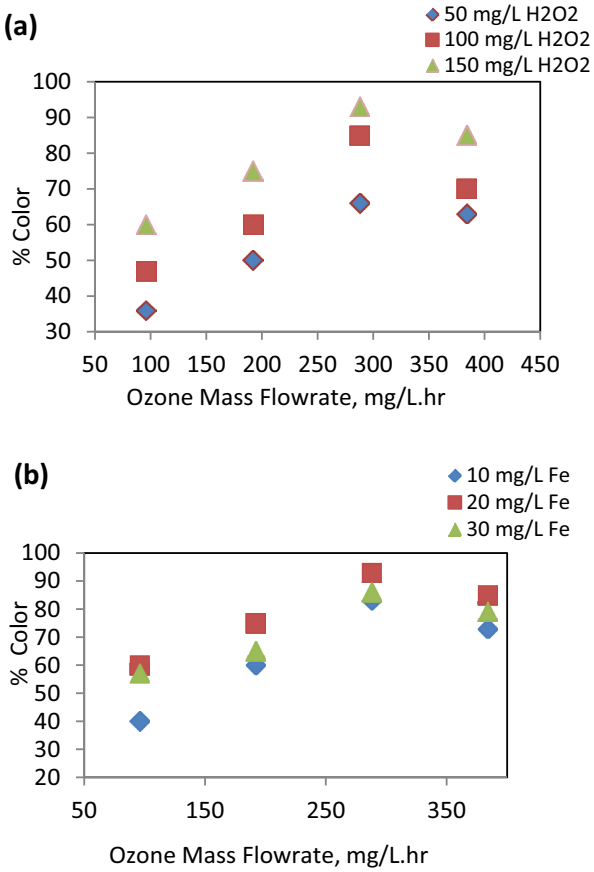


Fig. 4. Effect of ozone mass flow rate on decolorization efficiency at 60 min contact time with varying: (a) initial concentration of H_2O_2 and (b) initial concentration of Fe^{2+}.

on COD removal efficiency as shown in Figs. 5a and b. As shown in Fig. 4a, an increasing in ozone mass flow rate from 96 to 288 mg/L h while keeping the other parameters constant (20 mg/L Fe^{2+} and 60 min contacting time) could also led to an increase in decolorization efficiency from 60% to 93%, respectively. Further increasing in ozone mass flow rate more than 288 mg/L h can lead to a sharp decreasing in decolorization efficiency. The same behavior of the decolorization efficiency was shown in Fig. 4b, in which that the maximum removal efficiency achieved was 93% at optimum operational parameters are 288 mg/L h of ozone mass rate, 150 mg/L H_2O_2, 20 mg/L Fe^{2+} and 60 min contact time. The results obtained in Figs. 4a and b indicated that the decolorization efficiency increases with increasing ozone mass flow rate and this attributed to the high amounts of hydroxyl radicals produced, while further increasing in ozone production causes decreasing of oxidation potential of hydroxyl radicals. The high concentration of ozone can affect as a scavenger and react with the free radicals, and also react with the secondary reactions of decolorizations of ozone and causes a quench oxidation mechanism. The previous literatures have been shown that ozone mass flow rate could affects mainly on the hydroxyl radicals generated and the best conditions for hydroxyl radicals generated at basic conditions. At basic

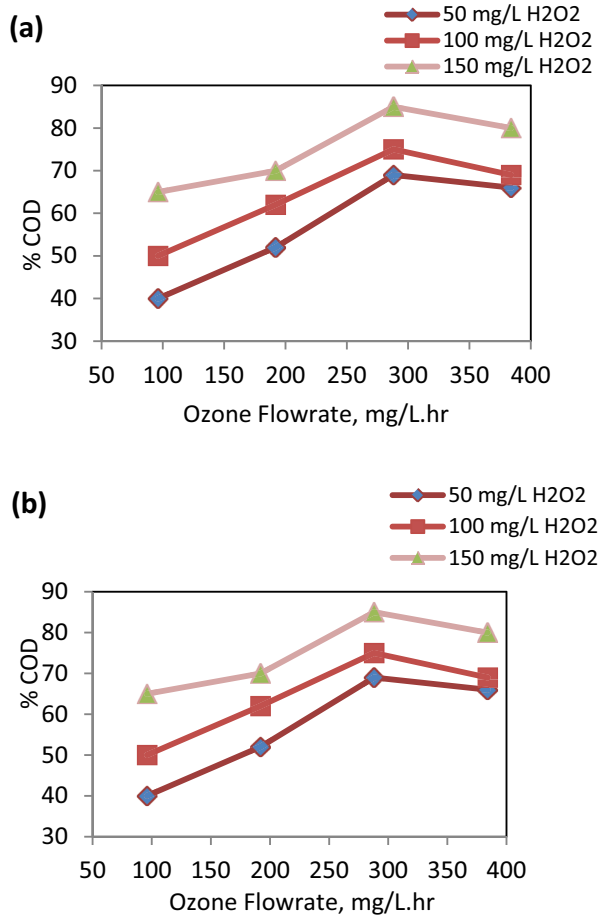


Fig. 5. Effect of ozone mass flow rate on COD removal efficiency with varying: (a) initial concentration of H_2O_2 and (b) initial concentration of Fe^{2+}.

condition, the effect of hydroxyl radicals beneficial besides that, hydroxide ions added can be catalyzed the decomposition of ozone in order to form non-selective and highly oxidizing hydroxyl radicals that have high oxidizing potential for ozone about (2.80 V) [24]. As expected, and according to the results obtained in Figs. 4a and b and 5a and b, indicated that, the $O_3/H_2O_2/Fe^{2+}$ process as AOPs efficient, and yielded a highly decolorization and COD removal of efficiency at optimum values of O_3, H_2O_2 and Fe^{2+} are 288 mg/L h, 150 mg/L and 20 mg/L, respectively.

3.3. Effect of contacting time

The contacting time between liquid and gas phase plays an important and essential role on the decolorization and COD removal efficiency of DB79. In this work, the effect of contact time ranged from 0 to 60 min on the decolorization and COD removal efficiency was examined as shown in Figs. 6a–c and 7a–c, respectively with varying ozone mass flow rate, initial concentrations of H_2O_2 and Fe^{2+}. The effect of contact time was examined in the following experiments, by varying contact time with each parameter while, keeping the other parameters constant (the optimum value for each

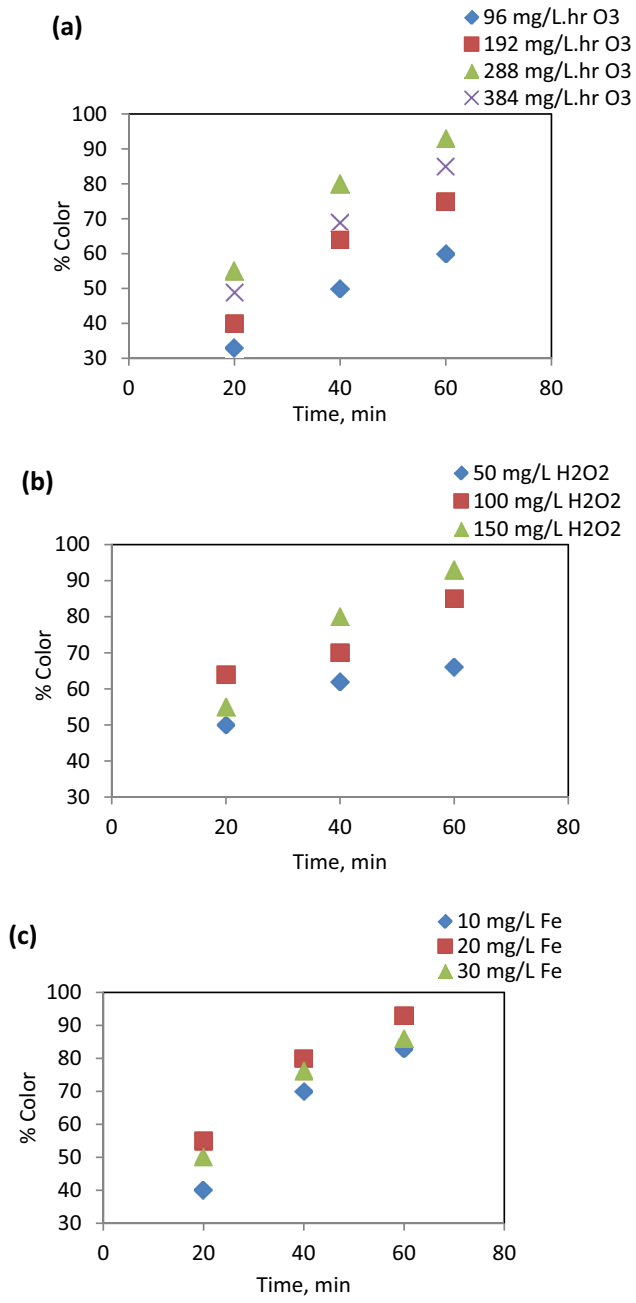


Fig. 6. Effect of contact time on decolorization with varying: (a) ozone mass flow rate, (b) initial concentration of H_2O_2 and (c) initial concentration of Fe^{2+}.

parameter according to the results obtained in Figs. 4a and b and 5a and b. As shown in Fig. 6a an increasing in contact time from 20 to 60 min, while keeping other parameters constant at (150 mg/L H_2O_2 and 20 mg/L Fe^{2+}) the decolorization efficiency also increased from 55% to 93%, respectively. The same behavior was shown in Figs. 6b and c. The decolorization efficiency was very efficient especially at the start period of treatment (till reach 40 min), after that the decolorization efficiency slightly changes and becomes stable. It can be explained that a long contact time means more advantage for removing color completely, and according to the results

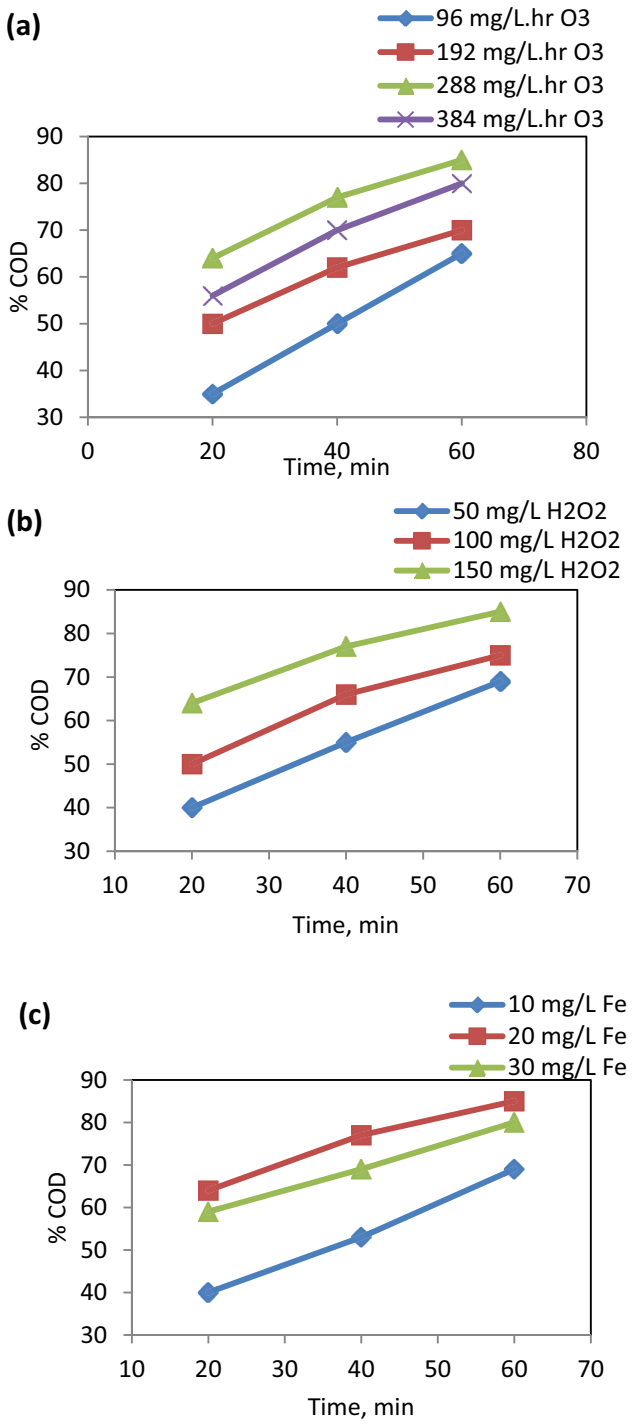


Fig. 7. Effect of contact time on COD removal efficiency with varying: (a) ozone mass flow rate, (b) initial concentration of H_2O_2 and (c) initial concentration of Fe^{2+}.

presented by a few researchers, a complete destruction of azo dye and reactive dyes have been investigated and recorded in 30–90 contact time. Regardless to the dosage applied in the processes contain ozone can be provided about 27%–30% TCOD removal after 30 min a contact time. On the other hand, the contact time also plays a significant role on COD removal efficiency of DB 79 as shown in Figs. 7a–c. According to the results obtained in Fig. 7a revealed that an increasing in contact time to 40 min while keeping the other parameters constant (288 mg/L h O_3, 150 mg/L H_2O_2, 20 mg/L Fe^{2+}) the behavior of COD removal efficiency could also increase to 77%, further increasing in contact time to 60 min with keeping the other parameters also constant the COD removal efficiency a slightly increased to the maximum value 85%. The same behavior can be seen by varying the contact time with initial concentrations of H_2O_2, and Fe $^{2+}$ in Figs. 7b and c respectively while keeping the other parameters constant.

4. Conclusions

$O_3/H_2O_2/Fe^{2+}$ as AOPs are good and efficient option for wastewater treatment and also, fast reaction rates and non-selective oxidation compared with the conventional methods. The main target from this study is to evaluate the color and COD removal of DB79 from wastewater by using AOPs. In this experimental study the color and COD removal efficiency using $O_3/H_2O_2/Fe^{2+}$ process in a semi-batch reactor were investigated. According to the results obtained, its indicated that the maximum decolorization and COD removal efficiency was 93% and 85%, respectively at optimum operating conditions are 288 mg/L h ozone mass flow rate, 33.53 mole ratio H_2O_2/Fe^{2+}(150 mg/L H_2O_2 and 20 mg/L Fe^{2+}) and 60 min contact time. The presence of hydroxyl radical's scavengers slow down the COD degradation efficiency. The combination of Ozone-Fenton was found to be an alternative method used in degrading DB 79 successfully in wastewater. Finally, it can be concluded that $O_3/H_2O_2/Fe^{2+}$ process plays an important role in obtaining good results and had better performance in color and COD removal of DB79 from wastewater.

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