DEGRADATION OF DISPERSE BLUE 79 DYE IN AQUEOUS SOLUTION USING FENTON (H_2O_2/Fe^{2+}) PROCESS

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ABSTRACT*: The release of variety dyes within wastewater into the environment is increasing and regarded one of the most issues in wastewater treating due to the toxicity and coloration of natural waters. Fenton method is an alternative and advanced oxidation processes used as a novel wastewater treatment process .In this study the degradation of Disperse Blue 79 (DB 79) dye at 60 mg/L in aqueous solution is investigated. The main focus of this work is to investigate the effect of different operational parameters such as amount of Fenton's reagent (H_2O_2, Fe^{2+}) doses, mole ratio of (H_2O_2/ Fe^{2+}), mixing speed, and the reaction time on the color, and chemical oxygen demand (COD) removal efficiency upon such process. Experimental results showed that, 85% color removal and 75% COD removal was achieved under optimum process conditions (33.53 mole ratio (150 mg/L H_2O_2, 20 mg/L Fe^{2+}), 50 rpm mixing speed, and 60 minute reaction time). Higher or complete degradation of DB 79 was observed within 60 minutes as a reaction time. Finally, Fenton process was very efficient and suitable for removing color, and COD from DB79 dye in aqueous solution.*

Keywords: Fenton, Disperse Blue 79 (DB 79), Color and COD removal

1. INTRODUCTION

Dyes and synthetic dyes are present in many fields of our life and the application of these dyes are continuously growing, and progressing in many of branches such as textile industry, leather tanning industry, palp, paper production, food technology, pharmaceutical industry and in hair colorings [1]. However, One of the most significant sectors of the global economy is the textile industry, particularly in countries such as India, China, Pakistan, Malaysia, [2]. The textile industry is one of the major sources of environmental pollution. Industrial wastewaters originating from synthetic dyes production and the application of these dyes have a major threat to the surrounding ecosystem due to their high toxicity and carcinogenic nature[3].

According to statistics it is estimated that over 100,000 types of dyes and synthetic dyes commercially are used and 700,000 tons annually of dyes manufactured in the world where 50% of these dyes are textile dyes and also 1-15% are lost during processes and discharged within wastewater[4]. Effluent streams that contain dyes within wastewater make it polluted, harmful, carcinogenic, and environmentally problematic [5], and hence highly colored and can cause human hazardous like the allergic reaction in eyes, irritation of skin [6,7]. Dyes effluent even at low concentrations can change the color of water[4].

Moreover, using dyes and especially disperse dyes have been increased significantly from the 1970s [8]. Disperse Blue 79 (DB 79) dye is one of the most colorants applicable in the textile industry and can be used in different processes such as nylon, polyester, and acrylic fibers[9]. The release of disperse dyes within wastewater into the environment is a concerning problem due to color change of natural waters and significantly high toxicity of these dyes [9]. Therefore, the color problems of some textile wastewaters caused due to the residual dyes during the dyeing processes which need more efforts to study and investigate[10,11].

A wide number of conventional treatment techniques have been used for treating wastewater effluents containing disperse dyes from industrial textile processes to fulfill the legislation requirements, among these techniques are gravitational separation, adsorption, coagulation floatation, filtration methods, biological methods, and thermal oxidation[12–16], where these treatment methods are not very efficient, useful in removing these dyes due to the complex structure of these aromatic dyes [17]**.** But the main drawbacks in using these conventional techniques in treating of dyes wastewater are these dyes very resistant to biodegradation methods, the highest water solubility, and poor adsorption ability on to the sludge, and hence these treatment techniques can offer low efficacy for degradation [18]. Therefore one of the main targets in the twentieth century was to improve simple, safe, economic, efficient and the most important factor not harmful to the environment [19]. Advanced oxidation process (AOPs) have been successfully applied for treating various of water pollutants such as textile wastewater[20], phenolic wastewater[21], pharmaceutical wastewater[22], pulp and paper industrial processes [22].

AOPs, first mentioned and stated by Glaze [23] and then cited by other researchers. AOPs are an alternative and effective method that generates hydroxyl radicals that use in treating wastewaters discharged from industrial dyeing textile wastewater industry [24-27] where these radicals are non-selective, strong oxidants that can degrade chemical organic compounds [28].

There are various types of AOPs such as Fenton, photo-Fenton, Fenton-like, UV/H2O2, O3, photocatalysis, sonolysis, wet and catalytic wet air oxidation where, these techniques are environmentally, and friendly processes based on a generation of hydroxyl radicals as major oxidants. Fenton method is one of the important processes of AOPs utilized in degradation of disperse dyes. However, it is very important to perform the Fenton process under the acidic condition to obtain high removal efficiency. Fenton's reagent in Fenton process is a mixture of hydrogen peroxide and ferrous ion (H_2O_2 & Fe^{2+}) which generates hydroxyl radicals according to the following reaction [29].

$$Fe^{+}+H_2O_2 \rightarrow Fe^{+++}+HO\cdot+{}^{-}OH \quad (1)$$

The main benefits of Fenton's process are easy operation and maintenance, besides that the hydroxyl radicals generated through this process are a viable source compared with other AOPs [30].

Recently, many literature reports have been addressed the application of Fenton method on the removal of organic compounds and color from a variety of industrial wastewaters. Keshmirizadeh and Farajikhajehghiasi [31]were studied the decolorization and degradation of Disperse Blue 56 and Basic Blue dyes by using Fenton process, they found more than 99% color removal and 88% COD removal were observed respectively at 10-100 mg/L initial dye solution and acidic condition (pH= 3). Hajjaji *et al* [32] have been addressed the application of Fenton method on the removal of aqueous acid orange by using clay/red mud. Yasar *et al*.[33] Showed the color removal of reactive azo dye (Blue CL-BR) were very efficient by using the Fenton process with bleach wastewater as a source of H_2O_2. Wali [34], studied the degradation of various commercial disperse dyes such as Disperse Yellow 23, Disperse red 167 and Disperse Blue 2BLN from aqueous solution by using the Fenton process, it was found that the removal efficiency was (84.66% color and 75.81% COD for Disperse Yellow 23), (77.19% color and 78.03% COD for Disperse red 167) and (79.63% color and 78.14% COD for Disperse Blue 2BLN) at optimal operating conditions (120 mg/L ferrous sulfate heptahydrate, 3 gm/L of H_2O_2, 160 min reaction time, and pH=3). Other researchers have studied combined Fenton processes (e.g.) UV/H_2O_2, UV-Fenton, and solar photo-Fenton for treating variety of textile dyes [18], [35]–[38].

The main objectives of this work are to investigate the removal color, and COD efficiency for Fenton process in order to remove color, and COD of DB 79 in aqueous solutions and also investigate the effect of key operational parameters such as (hydrogen peroxide, ferrous ions) doses, speed of mixing, and reaction time on Fenton process.

1- MATERIALS AND METHODS

2.1- Chemical used

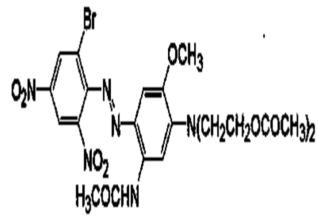
The Chemical materials used such as sulfuric acid (H_2SO_4), sodium hydroxide (NaOH), hydrogen peroxide (50% H_2O_2 w/v), ferrous sulfate heptahydrate($FeSO_4.7H_2O$), mercuric sulfate ($HgSO_4$), silver sulfate($AgSO_4$) and potassium dichromate($K_2Cr_2O_7$). All the chemicals used were of analytical grade and high purity without further purification

2.2- Preparation of sample Dye solution

A stock solution of synthetic DB 79 dye with an initial concentration (60 mg/l) used in this work was prepared by dissolving 60 mg of DB79 into 1 L of tap water. The major characteristics of DB 79 dye used in this present work are summarized and listed in **Tab.1**.

However, the total solution was stirred vigorously for five minutes at 50 rpm in order to ensure more homogeneity of the solution. It is very important to know before each run to adjust the solution pH to ensure best operation pH conditions (pH = 3) by adding few drops of sulfuric acid (H_2SO_4) and/or sodium hydroxide (NaOH) where pH value of the aqueous solution was monitored by using pH Meter[39].

Table.1: Characteristics of DB79 dye.

Item		
Linear Formula	$C_{23}H_{25}BrN_6O_{10}$	
Molecular Weight	625.38 gm/mole	
Color Index Number	11344	
pH	6.5-8.4	
Color[a] (AU)	0.9	
COD^b_i	260 mg/L	

[a]AU: absorbance unit measured at wavelength 565 nm

COD^b_i : initial COD of DB 79

[40]. In each run, the solution stirred and allowed to react and mixed for 60 min reaction time. Then, the mechanical stirrer was stopped and waiting for the solution for 30 minutes to settle completely. Finally, about 20 mL sample of the final settled solution was then taken at each run to estimate the color, and COD removal. Moreover, in order to evaluate the efficiency of Fenton method, different operational parameters were performed Fenton's reagents (H_2O_2 & Fe^{2+}) doses, molar ratio of hydrogen peroxide (H_2O_2) to ferrous (Fe^{2+}), mixing speed (0, 50, 100 rpm), and reaction time (0, 20, 40, 60 min). In all experiments, the solution dye was prepared using tap water and all the experimental runs were performed at room temperature (25 $\pm$ 2℃) and atmospheric pressure (1 atm).

2.3- Fenton method

Fenton method was carried out and performed using Jar test apparatus as shown in **Fig.**1 which consists of 4 cylindrical glass beakers (2000 mL capacity for each beaker) with mechanical stirrer 0-200 rpm operational speed. Firstly, 80 ml of sample dye solution was filled into each beaker and then tab water and Fenton's reagent (ferrous ions, Fe^{2+}, and hydrogen peroxide H_2O_2 30%) are added to complete the final solution volume to 2000 mL.

Figure.1: Jar Test apparatus

2.4- Analytical Methodologies

Color and COD measurements were obtained via a closed reflux titrimetric method and accordingly to standards methods[41]. Color measurements were measured using UV-spectrophotometer (Thermo Scientific, Genesys 20, Jerman). 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. COD measurements were determined by taking 2.5 ml of each sample at various time intervals and filled into a test tube containing (2.5 ml diluted sulfuric acid and 1.5 ml potassium di-chromates) and the samples were heated at 150°C for a period 120 min by using heater an operation temperature (0-150°C). After heating, the samples were performed to cool and then, taken to measure COD. All the sample of color and COD withdrawn at various time intervals (20, 40, 60 min) were performed and measured at room temperature (23-25°C). The apparatus used in analytical methodologies in this work is shown in **Fig.2.** The percentage color (%color) and COD removal (% COD) efficiency were estimated according to equation 2 and equation 3.

$$\% \text{ Color} = \frac{C_i - C_t}{C_i} * 100 \qquad (2)$$

$$\% \text{ COD} = \frac{COD_i - COD_t}{COD_i} * 100 \qquad (3)$$

Where, C_i, COD_i is the initial absorbance (0.9 mg/L) and COD(260 mg/L) respectively,

C_t, COD_t are the absorbance and COD at time t respectively.

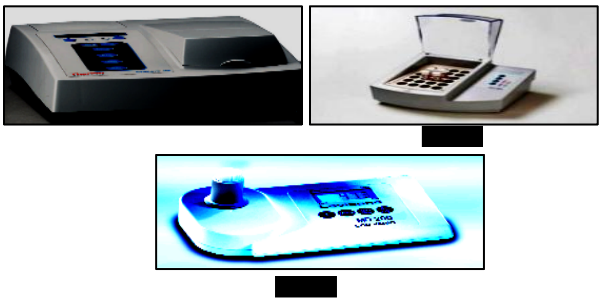


Figure. 2. Apparatuses used in analytical methodologies (a) UV-spectrophotometer (b) Heater (c) COD measurement.

3- RESULTS AND DISCUSSIONS

3.1- Effect of Hydrogen Peroxide (H_2O_2) dose

The selection of an optimal H_2O_2 dose for the degradation of DB 79 dye by using Fenton's process is very important due to the high cost of H_2O_2 and further excessive amounts doses of H_2O_2 cause side effects [42,43]. However, to understand the effect of addition H_2O_2 on the degradation of DB 79 dye aqueous solution with an initial concentration (60 mg/L), experiments were carried out at different doses of H_2O_2 (50, 100, 150 mg/L), at constant other parameters such as Fe $^{2+}$ dose, reaction time, were 20 mg/L, and 60 min respectively and with various three mixing speeds (0, 50, 100 rpm) as shown in Fig. 3(a) and (b). The results obtained in Fig. 3(a) and (b) show the removal efficiency of color and COD against H_2O_2 doses at various mixing speeds.

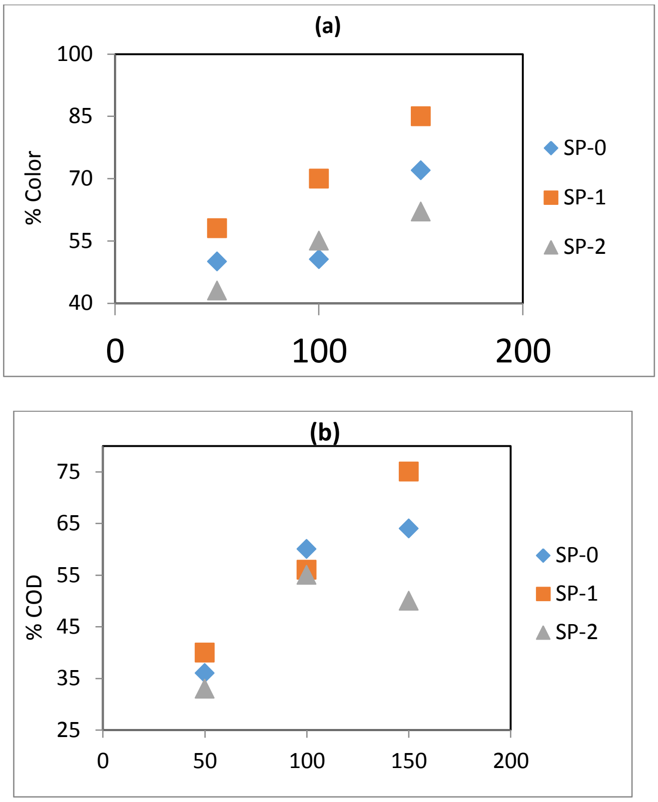


Fig. 3. Effect of H_2O_2 dose on (a) % color (b) % COD, of Disperse Blue 79 dye at various mixing speeds and constant (Fe $^{2+}$ = 20 mg/L, reaction time = 60 min).

It can be observed from Fig. 3(a) and (b) that with an increase in H_2O_2 dose from 50-150 mg/L there is an increasing in color removal from 50 to 72% and 36 to 64% COD at 0 rpm. Consequently, further increases in mixing speed to 100 rpm at the same increasing in H_2O_2 dose the color and COD removal can be enhanced and increased to the maximum removal from 58 to 85% color and 40-75% COD. The observed behavior can be attributed to the fact that with an increasing the initial H_2O_2 dose, there is also increasing of hydroxyl radicals generated. But, it is very important to take into account further amounts of H_2O_2 dose can act a scavenger for the hydroxyl radicals generated and this can lower the color and COD removal of the dye[43]. The same results were also obtained and reported by other researchers such as Alahiane et al. [43]. excessive amounts of H_2O_2 dose can act hydroxyl radical's scavenger reactions and become more dominant[44] according to reaction 4[45].

$$H_2O_2 + HO^{\cdot} \rightarrow H_2O^{-} + HO_2^{\cdot} \qquad (4)$$

According to the results obtained in this work, it can be proposed the optimal H_2O_2 dose is 150 mg/L in giving 85 and 75% color and COD removal respectively of the DB 79 dye solution.

3.2- Effect of ferrous ions (Fe $^{2+}$) dose

During Fenton process, another important and limiting factor influence on Fenton process is the number of ferrous ions where the effect of Fe $^{2+}$ dose on the color and COD removal efficiency was studied using Fenton process in the range of from 10-30 mg/L as shown in Fig. 4(a) and (b). It was found in Fig. 4(a) and (b) increasing the Fe $^{2+}$ dose from 10 to 20 mg/L can be resulted increasing removal efficiency from 67% to 85% color removal and 60% to 75% COD removal respectively, at constant other parameters such as 150 mg/L H_2O_2, 50 rpm, and 60 min reaction time. However, further increasing in Fe^{2+} dose more than 20 mg/L showed decreasing in color and COD removal efficiency to 74% and 52% respectively, due to the fact that excessive amounts of Fe^{2+} dose can act hydroxyl radical's scavenger reactions and become more dominant[44] according to reaction (5)[45].

$$Fe^{2+} + HO^{\cdot} \rightarrow OH^{-} + Fe^{3+} \quad (5)$$

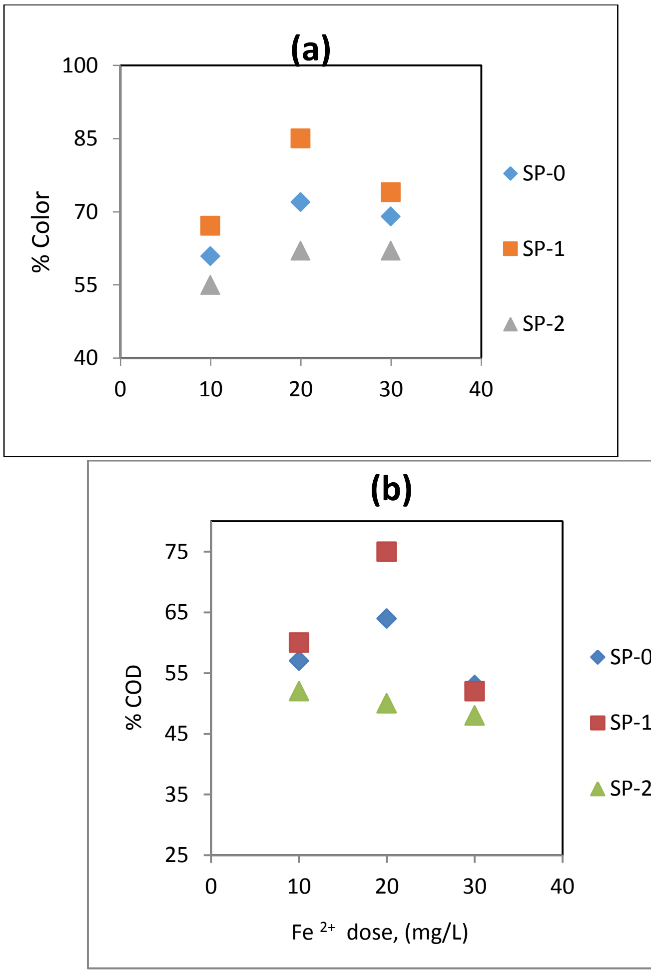


Fig. 4. Effect of Fe^{2+} dose on (a) % color (b) % COD, of Disperse Blue 79 dye at various mixing speeds and constant (H_2O_2 = 150 mg/L, reaction time = 60 min).

The results obtained in Fig. 4(a) and (b) showed that maximum removal efficiency obtained at optimum ferrous dose (20 mg/L), where in case of higher doses of ferrous ions the removal efficiency decreases and this may due to the increase of brown turbidity Fe^{3+} ions that hinder the absorption of light required for Fenton process, where the same behavior has been observed and cited by Tony[46].

3.3- Effect of (H_2O_2 / Fe^{2+}) mole ratio

It is very necessary to select the optimal H_2O_2 and Fe^{2+} doses, due to the cost issues of hydrogen peroxide and ferrous ions. However, several researchers [47], [48] attempted to find optimum H_2O_2 and Fe^{2+} doses. The effect of mole ratio on the color and COD removal in this work was investigated in the range from 7.45 to 67.07 at various mixing speeds as shown in Fig. 5(a) and (b). Results obtained demonstrate that at 7.45 mole ratio (50 mg/L H_2O_2, 30 Fe^{2+} mg/L) obtained 49% color removal and 35% COD removal at constant other parameters (50 rpm mixing speed, 60 min reaction). Sharply increasing in removal color and COD could be obtained with increasing mole ratio and continued to the maximum 85% color and 75% COD at 33.53 mole ratio (150 mg/L H_2O_2, 20 Fe^{2+} mg/L) at the same mixing speed. Further increasing in mole ratio more than33.53 could lead to sharply decreasing in removal color and COD efficiency due to excessive amounts of hydrogen peroxide H_2O_2 react with ferrous ion Fe^{2+} in aqueous solution under acidic conditions to generate hydroxyl radical.

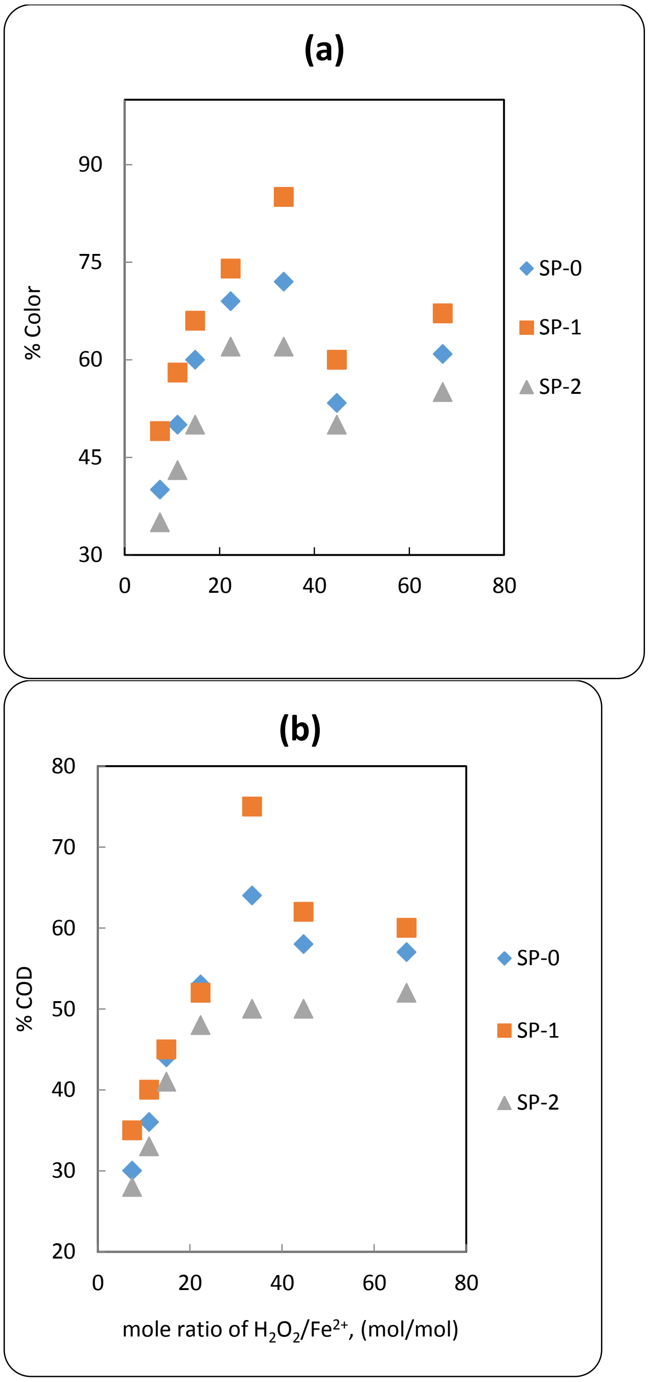


Fig. 5. Effect of mole ratio of H_2O_2/Fe^{2+} on (a) % color (b) % COD, of Disperse Blue 79 dye at various mixing speeds and constant (reaction time = 60 min).

3.4- Effect of Reaction Time

Contact time plays a more crucial role in the overall color and COD efficiency of Fenton process. Where the effect of reaction time on color and COD removal efficiency in Fenton process in the range of from 0 to 60 min was studied at optimum mole ratio 33.53 H_2O_2 / Fe^{2+} with various mixing speeds as shown in Fig. 6 (a) and (b). The results obtained in Fig. 6 (a) and (b) show increasing the reaction time from 20 to 60 min could lead to increasing the removal efficiency from 43% to 85% color and 43% to75% COD at 50 rpm. However, the removal efficiency increases with increasing the reaction time till reaching to the final reaction time 60 min.

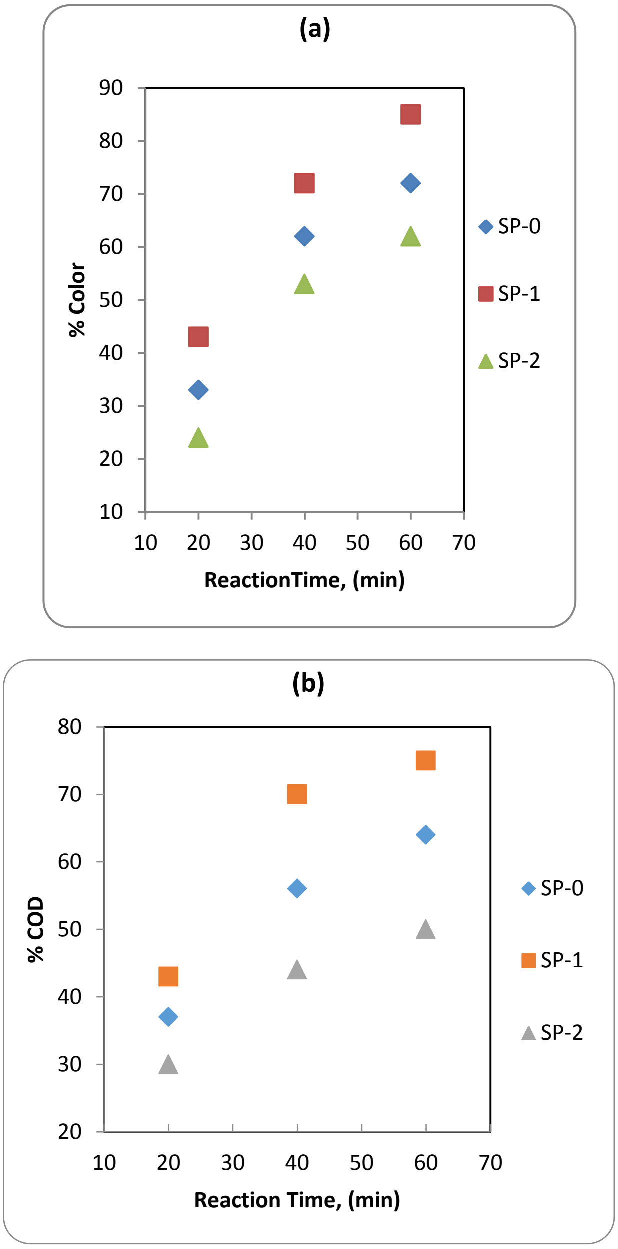


Fig. 6. Effect of reaction time on (a) % color (b) % COD, of Disperse Blue 79 dye at various mixing speeds and constant (H_2O_2 = 150 mg/L, Fe^{2+} = 20 mg/L).

Fig. 6 (a) and (b) indicate that the removal efficiency strongly depends on reaction time, where the color and COD removal efficiency sharply increased with increasing the reaction time (direct proportion) till reached to 40 min, furthermore than 40 min the removal efficiency enhanced slightly and be stable. It is observed from that the removal efficiency against reaction time increased with increasing the reaction time due to the high and enough contact time between Fenton's reagent and the dye in aqueous solution, and hence more time could perform to allow Fenton's reagent to consume completely with more contact time, thus optimum reaction time was 60 min. These results agree with Hyunhee [49].

3.5- Effect of Mixing Speed

The effect of mixing speed on the color and COD removal efficiency was examined by changing the mixing speed in the range of (0-100 rpm) while keeping other operation parameters constant (60 min reaction time, 33.53 mole ratio (150 mg/L H_2O_2, 20 Fe^{2+} mg/L)). Fig. 7 (a) and (b) shows that as mixing speed increased from 0 to 50 rpm, the removal efficiency also increased to 72% color removal, 64% COD removal, respectively. Where an increase in mixing speed up to 100 rpm, the removal efficiency decreasing to 62% color removal and 50% COD removal.

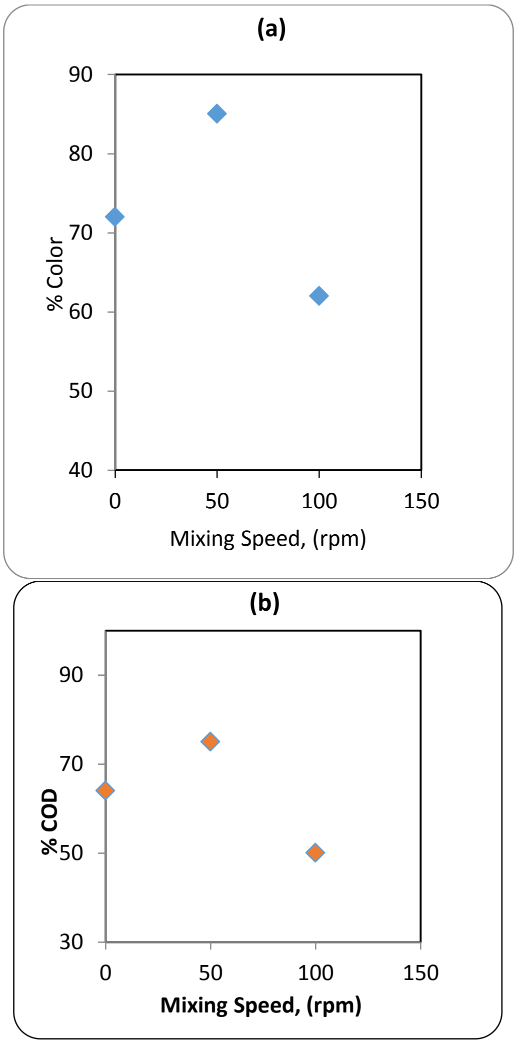


Fig. 7. Effect of mixing speed on (a) % color (b) % COD, of Disperse Blue 79 dye at constant (H_2O_2 = 150 mg/L, Fe^{2+} = 20 mg/L, reaction time=60 min).

It is clear from Fig. 7(a) and (b) the maximum removal color and COD efficiency could be obtained at optimal mixing speed 50 rpm due to at this operation speed a good homogeneity and distribution of Fenton's reagent to ensure efficient contact and reaction with the dye solution. Increasing mixing speed up to 100 rpm, the solution state could be transferred from homogeneity state to the dis-homogeneity (turbulence state), and hence there is no efficient contact time will be performed.

4- CONCLUSION

Fenton process was performed successfully via Jar Test in degradation and removing color and COD from Disperse Blue 79 dye. Based on the above results, the maximum removal color and COD efficiency was 85% and 75% respectively, at optimum operating conditions (33.53 mole ratio of H_2O_2/Fe^{2+} (150 mg/L H_2O_2, 20 Fe^{2+} mg/L), 50 rpm mixing speed, 60 min reaction time). It is concluded from this work the effect of mixing speed on Fenton Process was very significant and has a high effect on the removal color and COD efficiency. Hence, further researchers should be focused on the effect of mixing speed because there is no or fewer papers have focused on the effect of mixing speed on the Fenton process.

From these results, it can be concluded that Fenton process was the efficient method in degradation and color removal of Disperse Blue 79 dye when optimum operation conditions were used.

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