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# An optimization study of methylene blue dye treatment in synthetic textile wastewater by Fenton oxidation process

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### An optimization study of methylene blue dye treatment in synthetic textile wastewater by Fenton oxidation process Md. Burhan Kabir Suhan, Niaz Mahmud, Muhammad Hasanur Arefin Shahria, Md. Shahinoor Islam\*

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**Abstract:** Methylene blue (MB), an azo dye, is water-soluble and bio persistent because of its complex aromatic ring structure. Along with its carcinogenic, toxicogenic, and mutagenic nature, the MB dye can decrease the photosynthetic activity of aquatic plants. Therefore, the objective of the current study was to treat and optimize the treatment of MB dye in synthetic wastewater by the Fenton oxidation process and to determine the degradation's kinetics. To analyze the performance of the treatment, color, TDS, TSS, TS, turbidity, BOD and COD removals were assessed after the treatment of synthetic textile wastewater. The optimum dosing of FeSO<sub>4</sub>/H<sub>2</sub>O<sub>2</sub> was found 200 ppm/2.4 mL for the treatment and the corresponding reaction time for this optimum dose was 180 min. A noticeable reduction of color (>85%), COD (>85%) and turbidity (>70%) were observed from the study. The kinetic study demonstrated that COD, TDS and BOD degradation follows the second-order model satisfactorily. The rate constants were found  $0.09 \times 10^{-5} - 6 \times 10^{-5}$  Lmg<sup>-1</sup>min<sup>-1</sup>and half-lives were found at 3.2-30 min in second order model. Based on the performance of Fenton process, it can be concluded that Fenton oxidation process can be a better option for the treatment of textile wastewater. *Keywords: Fenton, methylene blue, ferrous sulfate, hydrogen peroxide, textile wastewater, kinetics* 

#### **1. Introduction**

In developing countries like Bangladesh, textile industries are severely polluting surface water by discharging tons of wastewater daily. This wastewater contains dyes, surfactants, and other organic and inorganic pollutants. These dyes are stable to light, heat, and conventional oxidizing agents and have persistent behaviour to aerobic biodegradation [1,2]. Therefore, it cannot be removed after applying conventional physicochemical and biological treatments. In addition, the Department of Environment (DoE) will soon implement a zero-discharge policy, which is a significant concern for the sustainable development of textile industries in Bangladesh. In this context, an effective treatment scheme for this effluent is urgently required before it is released into the surface water.

The conventional treatment methods applied coagulation-flocculation [3] and biological [4], treatments followed by sand filtration and adsorption [5,6] for the treatment of textile wastewater. However, it has been reported that, the conventional biological wastewater treatment process can hardly degrade azo dyes [7]. Physicochemical method has some constraints to the generation of large volumes of sludge, cost of chemical consumption and energy cost, etc. [8,9]. It is also inefficient due to bio-persistent and toxic nature of the dyes [10]. The adsorption process has a limitation of higher cost and adsorbent being contaminated in the recovery process [5,7]. Recently, it has been shown that advanced oxidation processes (AOPs) are promising in degrading dye containing wastewater. Among these methods, electrochemical [7], ozonation [11], persulfate degradation [12], Fenton [13] and nano-photocatalyst treatment have been applied efficiently [14-16]. These processes are very promising in destroying organic pollutants like textile dyes, naphthenic acids, cosmetics, and pharmaceutically active ingredients [10,15,17]. AOPs generate strong oxidizing hydroxyl radicals (•OH,  $E^0 = 2.8V$  versus NHE) capable of mineralizing organic pollutants to CO<sub>2</sub> and water [15].

Fenton oxidation process is one of the convenient AOPs treatments which can be used to degrade textile dyes, either independently or in combination with other conventional treatments. In this process, Fenton's reagent (a mixture of  $H_2O_2$  and  $Fe^{2+}$ ) is used where  $Fe^{2+}$  act as a catalyst, breaks down  $H_2O_2$  to generate •OH [18]. It is an effective process to degrade toxic and/or non-biodegradable compounds i.e., nitro-aromatics, aliphatic compounds, azo-dyes, phenols, etc., with the achievement of high reactions yields [19]. The primary mechanism of the Fenton process is explained in Eqn (1) and Eqn (2). Eqn (1) represents the generation of •OH, and Eqn (2) represents the catalytic formation of the Fe<sup>2+</sup> ion.

$$Fe^{2+} + H_2O_2 = Fe^{+3} + HO^{\bullet} + HO^{-} \dots \dots (1)$$
  
$$Fe^{+3} + H_2O_2 = Fe^{2+} + HO_2^{\bullet} + H^{+} \dots \dots (2)$$

The effectiveness of the Fenton process depends mainly on the generation of  $\cdot$ OH. Optimum dosage of the operational molar ratio of Fe<sup>2+</sup>/H<sub>2</sub>O<sub>2</sub> is essential to achieve the best output from Fenton process [20]. A higher dosage of Fenton reagent results in lower COD reduction and color removal efficiency due to the scavenging effect of  $\cdot$ OH with the reaction of excess Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> [21]. In addition, excess Fe<sup>2+</sup> can contribute to total dissolved solids (TDS) content of the effluent stream, which is an undesirable phenomenon [22].

 $H_2O_2$  concentration in treated wastewater is an important factor and contributes significantly to the removal of organics. Generally, increasing the  $H_2O_2$  concentration leads to increasing the degradation of the pollutants because more  $\cdot$ OH are produced, which degrade the color, COD and BOD [23].

pH of the treated wastewater is also a vital parameter of the degradation process. Initial pH can slower or faster the degradation rate of COD and phenol compounds [24]. At higher pH (generally above 4), oxidation potential of •OH

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decreases rapidly and the activity of Fenton reagent decreases for the existence of inactive iron oxo-hydroxides and the formation of ferric hydroxide precipitate. In addition, iron species can inhibit the reaction between  $Fe^{2+}$  and  $H_2O_2$  [25]. On the other hand, at low pH values (generally below 2.5), iron complex species  $[Fe(H_2O)_6]^{2+}$  forms, which reacts more slowly with  $H_2O_2$  than other species [26].

Methylene blue (MB) is a cationic dye because it forms a coloured cationic salt when dissolved in wastewater. Cationic dyes are significantly more harmful than anionic dyes. Since MB dye is extensively used in textile dyeing, paper, pharmaceutical, cosmetics, plastics, rubber, leather and food industries, an efficient treatment method is urgently required for degrading this dye [27,28].

In this study, a model was developed to investigate the optimum molar ratio, optimum dosage, and optimum reaction time required for Fenton based AOP's treatment of synthetic textile wastewater containing MB dye. The experiment explored both the advantages and disadvantages of Fenton based AOP's. A rate-based model has also been derived at the end of kinetic analysis, indicating the reaction order, rate constant and half-life of the reaction.

#### 2. Materials and Methods

#### 2.1. Materials

The materials used in this study are:

Analytical grade ferrous sulfate heptahydrate (FeSO<sub>4</sub>.7H<sub>2</sub>O), H<sub>2</sub>O<sub>2</sub> solution (30% w/w; density 1.07 g/cm<sup>3</sup>), sulfuric acid (98% purity, density 1.84 g/cm<sup>3</sup>), sodium hydroxide pellet, sodium carbonate and acetic acid (99%). All the chemicals mentioned above were purchased from Merck Company, Germany. Sodium chloride (99% w/w), detergents (SLS) and food-grade starch were purchased from a local laboratory supply chemical store. Textile grade MB dye (Chemical formula  $C_{16}H_{18}ClN_3S$ ) was collected from a local company Jamuna Denims Limited.

#### 2.2. Synthetic textile wastewater preparation

Synthetic textile wastewater was prepared according to the constituents shown in table 1 [29]. The room and wastewater temperatures were nearly 24 °C and 23 °C, respectively.

Table 1. Chemical constituents used for the	preparation of synthetic textile wastewater
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Materials	Concentration	Function
Starch	1000 mg/L	Sizing agent
Acetic acid	0.20 mL /L	Sizing agent
Dye (Methylene Blue)	251 mg/L	Coloring agent
Caustic Soda	512.5 mg/L	Hydrolysis
Sodium Carbonate	501.5 mg/L	Fixing agent
Sulfuric Acid	0.301 mL/L	pH control
Detergents	128.5 mg/L	Scouring agent
Sodium Chloride	3076.5 mg/L	Fixing agent

#### 2.3. Methods and analysis

The synthetic textile wastewater treatment was performed in a batch reactor. The schematic diagram of the experimental set-up used for this process is shown in Figure 1. The reactor was a 500 mL beaker made of quartz glass. Initial physicochemical parameters of the synthetic textile wastewater and experimental procedure of the solution preparation have been shown in Table 2 and Figure 2, respectively.

Color analysis and COD were determined via the spectrophotometer (HACH-DR/4000US) using a dichromate solution as the oxidant in a strongly acidic medium [30]. TDS was measured by Hanna-edge HI2030-01. Before calculating the BOD<sub>5</sub>, the wastewater was adjusted to neutral pH at seven by adding Na<sub>2</sub>CO<sub>3</sub>[31]. Other instruments used in experiments are weight machine (Vibra and XB-220A Precisa) and desiccator for TS analysis (GCA Precision Scientific Model 68351, heated vacuum desiccator).

	Table 2. Physicochemical	parameters of synthetic textile wastewater.
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Parameter	Unit	Quantity
Color	Pt-Co	2100
Turbidity	FAU	6200
TDS	gm/L	3.70
TS	gm/L	5.30
TSS	gm/L	1.89
BOD	mg/L	180
COD	mg/L	524
pН	-	10.3



Figure 1. Set up for the experiment. A- Reaction beaker, B- Wastewater solution, C- Magnetic stirrer, D- Electrode measuring pH, E- pH meter.

400 ml v	wastewater solution
	Adding H <sub>2</sub> SO <sub>4</sub>
	pH= 3.8
	Buffer solution of $pH = 4$ added
F	SeSO <sub>4</sub> added
	2 min at 1200 rpm for homogenity
]	H <sub>2</sub> O <sub>2</sub> added
	10 min at 1200 rpm
Sol	ution prepared

Figure 2. Steps of solution preparation for Fenton treatment.

#### 2.4. Kinetic study

MB dye removal kinetics has been performed for zero-order, first-order and second-order model with respect to degradation of TDS, COD and BOD with time. The integrated form of above kinetic models are presented by Eqns. 3-8 [7]

Zero order kinetics:

$$C - C_o = k_o t$$
 (3)  
 $t_{1/2} = \frac{C_o}{2k_o}$  (4)

First order kinetics:

$$\ln\left(\frac{C_0}{c}\right) = k_1 t \tag{5}$$
$$t_{1/2} = \frac{\ln 2}{k_1} \tag{6}$$

Second order kinetics:

$$\frac{1}{c} - \frac{1}{c_0} = k_2 t$$
(7)  
$$t_{1/2} = \frac{1}{k_2 C_0}$$
(8)

Where  $k_0 (mg.L^{-1}.min^{-1})$ ,  $k_1 (min^{-1})$  and  $k_2 (L.mg^{-1}.min^{-1})$ , represent the apparent kinetic rate constants of zero, first and second-order reaction kinetics, respectively. Co  $(mg.L^{-1})$  and C  $(mg.L^{-1})$  are the initial dye concentration and dye concentration at time t (min), respectively.

# Results and Discussions Optimum molar ratio of Fe<sup>2+</sup>/H<sub>2</sub>O<sub>2</sub>

The optimum molar ratio of  $Fe^{2+}/H_2O_2$  was evaluated by changing moles of  $H_2O_2$  where moles of  $Fe^{2+}$  remained constant. A total of 8 experiments have been conducted by varying the molar ratio of  $Fe^{2+}/H_2O_2$  from 1/1.5 to 1/21.4 and the optimum molar ratio has been determined from their degradation capability of color, COD and BOD. Figure 3 shows that when the molar ratio of  $Fe^{2+}/H_2O_2$  approached 1/14.5 the degradation capability of color, BOD and COD reached equilibrium. After that point, there was no significant changes in degradation with addition of  $H_2O_2$ . Thus, optimum molar ratio of  $Fe^{2+}/H_2O_2$  was 1/14.5 [32]. During each experiment concentration of  $FeSO_4$  remained constant at 250 ppm. Initially, the pH of the solution was maintained near 3.80. But the addition of 250 ppm  $FeSO_4$  reduces the pH to 3.05 due to its acidic behaviour in solution. On the other hand,  $H_2O_2$  addition has no impact on pH rise or drop.



Figure 3. Removal percentage of color, BOD and COD with respect to molar ratio of  $Fe^{2+}/H_2O_2$  after 20 hours keeping fixed [ $Fe^{2+}$ ] = 250 ppm (1.65 m.moles).

#### 3.2. Optimum dosing point determination while molar ratio of Fe<sup>2+</sup>/H<sub>2</sub>O<sub>2</sub> is 1/1.14

In order to find out the optimum dosing point a total of 10 experiments have been conducted by changing the dose of  $Fe^{2+}$  and  $H_2O_2$  while keeping the optimum molar ratio of  $Fe^{2+}/H_2O_2(1/14.5)$  constant.  $Fe^{2+}$  concentration varied from 10-1250 mg.L<sup>-1</sup> with corresponding  $H_2O_2$  dose. Table 3 represents the removal percentage of TDS, TS, color, turbidity and COD with the change of  $Fe^{2+}$  and  $H_2O_2$  concentration. pH of the solution decreased continuously with the addition of  $FeSO_4$  (from 3.8 to 2.42 when  $Fe^{2+}$  added from 10 mg.L<sup>-1</sup> to 1250 mg.L<sup>-1</sup>) because  $FeSO_4$  dissolution in water produces more H<sup>+</sup> hence the wastewater became more acidic. Except TS, in all cases the highest removal was obtained on 200 ppm  $Fe^{2+}$  concentration and 2.4 ml (30% w/v)  $H_2O_2$  dose.

The initial TDS, TS, color, turbidity and COD concentrations are shown in table 2. As the rise of  $Fe^{2+}$  concentration from 10 ppm, removal efficiencies of all the parameters increased continuously up to 200 ppm. After that, the removal efficiencies decreased significantly with the addition of  $Fe^{2+}$ . The highest removal of TDS, TS, color, turbidity, COD were found 43% (at 200 ppm  $Fe^{2+}$ ), 42% (at 150 ppm  $Fe^{2+}$ ), 88% (at 200 ppm  $Fe^{2+}$ ), 72% (at 200 ppm  $Fe^{2+}$ ) and 87% (at 200 ppm  $Fe^{2+}$ ), respectively. Therefore, the optimum dose of the  $Fe^{2+}$  was 200 ppm. After the optimum dosing point TDS removal percentage was decreased because of excessive iron accumulation and drop of pH of the treating solution. The drop of color removal percentage after optimum dose might be due to the excessive amount of  $FeSO_4$  that may impact the color measurement. Excessive iron accumulation, low pH and less reaction propagation or smaller organic formation in the unfavorable environment after a particular point might decrease the turbidity removal percentage after 200 ppm as shown in table 3. The reduction percentage of TS (total solid) was maximum at 150 ppm of MB dye concentration. The presence of unreacted FeSO<sub>4</sub> might be the reason of decreasing of removal percentage of TS at higher  $Fe^{2+}$  concentration. Again, excessive  $Fe^{2+}$  may lead to pH (optimum pH 4.0-3.0) lower than the optimum level, resulting in less reaction between organic compounds and reactive hydroxyl radicals. That's why, after a specific dose catalytic oxidation process was not effective.

FeSO <sub>4</sub>	Removal percentage (%)				
(ppm)	TDS	TS	Color	Turbidity	COD
10	7	12	19	39	28
50	6	21	43	56	45
100	16	33	62	60	53
150	30	42	85	68	81
200	43	41	88	72	87
250	28	37	68	70	83
350	16	35	38	69	86
500	3	19	31	63	85
750	2	13	29	60	79
1250	2	13	23	56	72

Table 3. Removal percentage of TDS, TS, color, turbidity, COD for a fixed molar ratio of Fe<sup>2+</sup>: H<sub>2</sub>O<sub>2</sub>=1:14.5.

#### 3.3. Effect of reaction time on optimum dosing

For 200 ppm FeSO<sub>4</sub> and 2.4 mL  $H_2O_2$  dose, the degradations of specific parameters (color, TDS, COD and BOD) were observed for 225 min, and the results have been illustrated in figure 4. From figure 4, it can be seen that color change was initially fast due to availability of the reactant. After 180 minutes, the color of the wastewater was nearly 250 Pt-Co and no effective removal was observed after that time (180 min). Similar results can be seen for other parameters except TDS. At 180 min of reaction time, COD value reached nearly 70 mg/L and became equilibrium in that value. Similar results were achieved in BOD degradation. However, TDS degradation didn't reach the equilibrium value within the reaction time of 180 min. The rate of TDS degradation was slow (figure 4), which might lead it to reach the equilibrium time slightly greater (225 min) than optimum color, COD and BOD degradation time. It is known that Fenton type oxidation continues for a longer period as long as  $Fe^{2+}$  and  $H_2O_2$  available within the reactor. Still, the reaction rate becomes very slow after a certain period. In this study, the aforementioned period was found 180 min for color, COD and BOD but 225 min for TDS removal.

#### 3.4 Kinetics analysis

Kinetic study has been performed following the zero-order, first-order and second-order models for TDS, COD and BOD degradation. Kinetic plots of them have been illustrated in figure 5 and the calculated parameters of the model have been shown in table 4. Comparing the regression coefficient ( $R^2$ ) shown in table 4 and figure 5, it can be seen that TDS, COD and BOD degradation follows second-order kinetic model. R<sup>2</sup> values were found at 0.99, 0.96 and 0.93 for TDS, COD, and BOD in the second-order model [33]. The rate constants (k<sub>2</sub>) were found 0.09×10<sup>-5</sup> Lmg<sup>-1</sup>min<sup>-1</sup>, 6×10<sup>-5</sup> Lmg<sup>-1</sup>min<sup>-1</sup> and 6×10<sup>-5</sup> Lmg<sup>-1</sup>min<sup>-1</sup> while half-lives were found 30 min, 3.2 min and 9.3 min for TDS, COD, BOD, respectively in the second-order model. Apart from the second order, the first-order kinetic model also fitted well (better than second order in COD degradation kinetics) where R<sup>2</sup> values were found 0.99, 0.94 and 0.79 for TDS, COD and BOD, respectively. Zero-order fitted well on TDS degradation only but very poor in other two. Half-lives of them have been calculated and the calculated data were placed in Table 4.



Figure 4. Degradation of Color, TDS, COD and BOD when  $Fe^{2+}/H_2O_2$  molar ratio = 1/1.14 and dosing 200 ppm/2.4ml

Analytical methods TDS COD			COD	BOD	
	Zero-order	k <sub>0</sub> (mg.L <sup>-1</sup> .min <sup>-1</sup> )	7.67	2.58	0.69
Ze Kinetic parameters Fi Sec		R <sup>2</sup>	0.98	0.65	0.57
		t <sub>1/2</sub> (min)	241	101	130
	First-order	k <sub>1</sub> ×10 <sup>-3</sup> (min <sup>-1</sup> )	2.60	10.50	6.20
		R <sup>2</sup>	0.99	0.94	0.79
		t <sub>1/2</sub> (min)	267	66	112
	Second-order	k <sub>2</sub> ×10 <sup>-5</sup> (L.mg <sup>-1</sup> min <sup>-1</sup> )	0.09	6.0	6.0
		<b>R</b> <sup>2</sup>	0.99	0.96	0.93
		t <sub>1/2</sub> (min)	30.0	3.2	9.3



Figure 5. Kinetic plots of zero-order, first-order and second-order model for TDS, COD and BOD degradation

#### 4. Conclusion

The optimum molar ratio of  $Fe^{2+}/H_2O_2$  and optimum time were found to be 1/14.5 (at optimum  $Fe^{2+}$  dose of 200 ppm) and 180 min, respectively, for the degradation of synthetic textile wastewater containing MB dye. The degradation reaction follows the second-order kinetic model. The process is highly efficient for color, COD and BOD removal, however, the reduction efficiency of TDS, TSS, TS was not satisfactory. The excessive iron accumulation and formation of small molecule from breaking large organic compounds can probably be a barrier in TDS, TSS, TS removal efficiency. Our results therefore indicate that the Fenton oxidation process can be successfully utilized to remove color and COD from MB dye containing textile wastewater.

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