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Optimization of Fenton Oxidation to Treat Textile Wastewater

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ABSTRACT

Studies were made to assess the performance and optimal condition of Fenton oxidation in textile wastewater. Three factors considered in this process which were concentration of hydrogen peroxide (H_2O_2), concentration of ferrous ion (Fe^{2+}) and reaction time. Central Composite Design (CCD) from Design of Expert was used to study the interaction of factors towards the removal of colour, which was chosen as response. This study were involved with three factors and five level. Range for each factors were selected at 8.00-16.00 mM for Fe^{2+}, 60-280 mM of H_2O_2 and 60-90 minutes. The optimal condition was taken by minimum concentration of Fe^{2+} and H_2O_2 and shortest reaction time to achieve the highest removal of colour. Result indicated that optimum conditions for the removal of colour were happened to be at 145.82 mM H_2O_2, 9.62 mM Fe^{2+} and 83.92 minute. The highest percentage removal of the colour were found to be at 98.18%.

Keywords: Colour Removal, Design of Experiment, Fenton Treatment, Optimization, Textile Wastewater.

1. INTRODUCTION

The textile industries consume high volume of water that causes the intense pollution through their effluent. The wet processing operations in textile industry involving the sizing, desizing, scouring, bleaching, mercerizing and dyeing processes (Pazdzior *et al.*, 2018). Most of the effluent released by the textile wastewater that contain colour is from the dyeing and washing operation which as much as 50% of the dye (Joshi *et al.*, 2004). The wastewater that contains many types of pollutants had formed from the whole processes of textile. Thus, pollutants by textile wastewater contributed to a large quantity of suspended solids, strong colour, and high temperature. The textile effluent also give effect on the environment, human health and aquatic life because they contained high concentrations of salts, nutrients, Chemical Oxygen Demand (COD) and toxic substance such as heavy metals (Ertugay *et al.*, 2017). Due to the variation of colours, high content of surfactants and other organic and inorganic compounds and defective biological recovery, textile wastewater is difficult to treat when using the classical physicochemical and biological methods. Therefore, in order to increase the efficiency of treatment methods with of high degree of pollutant removal and cost effective treatment, optimization was carried out.

Among the techniques that were studied were advanced oxidation processes, reverse osmosis, membrane filtration and coagulation, which proved to reduce pollutants concentrations in wastewater. However, Fenton treatment is much preferable due to inexpensive and easily accessible reagent. Fenton reagent is well known to produce radical agents by reacting Fe^{2+} and H_2O_2 (Ertugay *et al.*, 2017). These free radicals be involved in reacting as a combination of hydrogen peroxide and ferrous iron where scavenging activity will take place and oxidize toxic contaminants unbiased to form simpler products. Fenton reagent which using the reaction of hydrogen peroxide decomposition catalyzed by ferrous ions leads to the formation of hydroxyl radicals (Pazdzior *et al.*, 2018).

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$$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + OH^- + HO\cdot \qquad (1)$$

Due to the reaction between the $HO\cdot$ radical and the ferrous ion, the ferric and hydroxyl ions are also formed during the process of Fenton (Karthikeyan *et al*., 2012).

2. MATERIAL AND METHODS

2.1 Sample wastewater

The dye reactive black 5 (RB5) (N 150%, DyStar) was chosen as a dye wastewater sample and taken textile industry in Pasir Mas, Kelantan. The reactive black 5 was categorized as recalcitrant because of the existence of two Azo bonds (-N=N). The sample was observed for three days to observe any deviation values of pH, colour and absorbance value that may occur to the wastewater sto ensure it is in steady state. The sample was kept in room temperature and the data was recorded every day to see the changes.

2.2 Fenton's Experimental Procedure

Fenton oxidation process was conducted in room temperature. First, 100 ml of wastewater that contain the reactive black 5 was placed into 500 ml beaker. pH of the sample was measured and pH was lowered to pH 3 by using 0.5 M hydrochloric acid (95% to 98% v/v, HmbG Chemicals) solution. Later, the iron (II) sulphate ($FeSO_4.7H_2O$) (HmbG Chemicals) and hydrogen peroxide (35% v/v, Bendosen) were added in the RB5 solutions according to the desired dosage that was given by Design of Expert. The start time was recorded after hydrogen peroxide was added to the solution and the solution was stirred at 350 rpm. After the solution was stirred, the pH of the solution was measured and 0.1 M sodium hydroxide (NaOH) (pellet, HmbG Chemicals) solution was added until achieved pH 7 to terminated and stop the reaction. The solution was left to settle for two hours. The temperature was kept constant during the process which is at 30°C. After the solution was settled, the treated wastewater was filtered to separate the suspended particle. Lastly, the sample was measured for the wavelength using UV-Visible spectrophotometer, Lambda 25, Shimadzu, Japan.

2.3 Analytical Procedure

The UV-visible spectrophotometer is used to analyse the dye concentration of the Reactive Black 5 by measuring the absorbance of the untreated sample wastewater at the maximum wavelength. The maximum absorbance wavelength of Reactive Black 5 was observed at 597 nm. The percentage of the colour removal was calculated by using the following Equation 2:

$$Abs\ (\%) = \frac{\text{abs}\ (\lambda\ \text{max})\text{i} - \text{abs}\ (\lambda\text{max})\text{t}}{\text{abs}\ (\lambda\text{max})\text{i}} \qquad (2)$$

2.4 Parametric Study

The parametric study was done to study the effect of hydrogen peroxide, iron (II) sulphate and reaction time. Table 1 shows the range for parametric study Fe^{2+} , H_2O_2 and reaction time.

Table 1 Range of parametric study for iron (II) sulphate, hydrogen peroxide and reaction time

Parametric study	Iron (II) sulphate (mM)	Hydrogen peroxide (mM)	Reaction time (minute)
Effect of Iron (II) sulphate	3.0	100	30
	9.0	100	30
	15.0	100	30
Effect of hydrogen peroxide	12.0	170	30
	12.0	270	30
	12.0	370	30
Effect of reaction time	10.0	160	30
	10.0	160	60
	10.0	160	90

2.5 Design of Experiment

The range then was chosen based on parametric study to be studied for optimization. Table 2 shows the range for each variable in the experiments.

Table 2 The ranges and levels of values for three types of variables

Factors	Range and Levels				
	-2	-1	0	+1	+2
Concentration of Fe^{2+} [mM]	8.0	10.0	12.0	14.0	16.0
Concentration of [H_2O_2 [mM]	60	115	170	225	280
Reaction Time [min]	60	67.5	75	82.5	90

3. RESULTS AND DISCUSSION

3.1 Characteristics of Textile Wastewater

The characteristics of textile wastewater was recorded that are based on colour, pH, absorbance value and appearance. Table 3 summarized the observation obtained from the measurement.

Table 3 Characteristics of textile wastewater

Parameter	Observation
Colour	Black colour and more to dark blue due to the presence of RB5
Appearance	Wax appear on top of the wastewater surface Slippery when in contact
pH Value	11.40 – 12.15
Absorbance Value (nm)	0.0696 – 0.1075 (10 x dilution factor)

3.2 Parametric Study

The effect of ferrous ion concentration on colour removal was shown in the Figure 1. According to the figure, the percentage of colour removal was increased as the concentrations of ferrous ion was increased up to a critical ferrous ion concentration which is 10 mM. But when the excessive of ferrous ion, the percentage of the colour removal was decreased due to the hydroxyl scavenging effect of ferrous ion (Yuan *et al.*, 2018). While for the effect of hydrogen peroxide concentration to the colour removal of RB5 showed the percentage of colour removal increased with increase of H_2O_2 concentration up to the critical concentration. But when the concentration of H_2O_2 was further increase, a decrease of percentage of colour removal was observed, which may due to the excess of H_2O_2 dosage (Ertugay *et al.*, 2017).

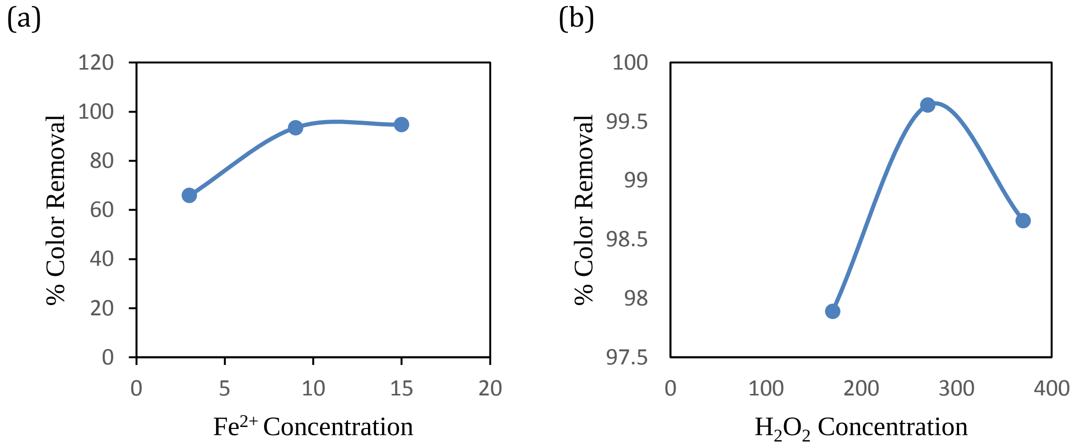

Figure 1. Effect of concentration of (a) ferrous ion and (b) hydrogen peroxide.

Based on the Figure 2, it presented the effect of reaction time towards colour removal. The percentage of colour removal increase as the reaction time was increased. The highest percentage of colour removal is at minute 60. After the 60 min, the percentage of colour removal start to decrease because most of the dye was fully oxidized by the hydroxyl radical. Further reaction time will not lead to significant reduction of color removal (Papadopoulus, 2007 #7).

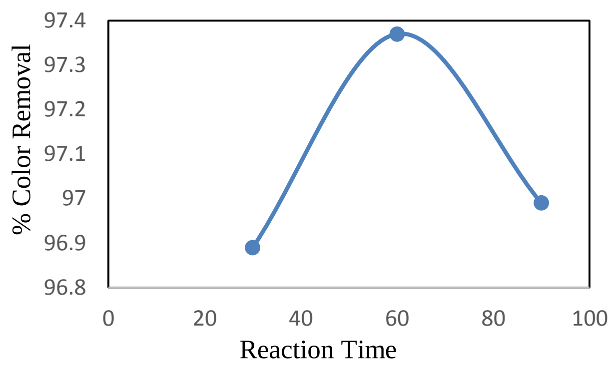

Figure 2. Effect of reaction time on colour removal.

3.3 Experimental Condition and Result of Central Composite Design (CCD)

For the optimization of the Fenton oxidation process, the Response Surface Methodology (RSM) with CCD (Central Composite Design) was used to analyse all responses (Lak *et al.*, 2018). Three parameters were selected to get the optimization which are the concentration of Fe^{2+}, concentration of H_2O_2 and the reaction time. Twenty experiments were conducted based on the range for each parameter that was selected by using the Central Composite Design (CCD) on Design of Expert software.

The concentration of Fe^{2+} are between 8.0 mM to 16.0 mM, while the concentration of H_2O_2 are between 60 mM to 280 mM and the reaction time are between 60 min to 90 min. The relationship between the three factors is listed in Table 4 together with the percentage removal of the colour, which was considered as the response in this design.

Table 4 Experimental data for Fenton process

Run	Factors			Response	
	Factor 1 $[Fe^{2+}]$ (mM)	Factor 2 H2O2 (mM)	Factor 3 Reaction Time (min)	Colour Removal (%)	
				Actual	Predicted
1	12.00	170.00	60.00	99.60	99.40
2	12.00	170.00	90.00	99.69	99.44
3	12.00	170.00	75.00	99.26	99.28
4	12.00	170.00	75.00	99.41	99.28
5	12.00	280.00	75.00	99.28	99.28
6	9.62	104.59	83.92	96.23	96.50
7	8.00	170.00	75.00	98.37	98.26
8	14.38	104.59	66.08	97.09	97.43
9	12.00	170.00	75.00	99.17	99.28
10	9.62	235.41	66.08	99.64	99.70
11	12.00	60.00	83.92	95.20	94.75
12	9.62	104.59	66.08	96.26	96.41
13	9.62	235.41	83.92	99.69	99.67
14	12.00	170.00	75.00	99.68	99.28
15	14.38	235.41	83.92	99.43	99.60
16	14.38	235.41	66.08	99.60	99.65
17	16.00	170.00	75.00	99.39	99.05
18	12.00	170.00	75.00	99.15	99.28
19	12.00	170.00	75.00	98.92	99.28
20	14.38	104.59	83.92	97.23	97.50

3.4 Adequacy of Fitting Model

The analysis of variance (ANOVA) were used to test the significance and adequacy of the constructed models. The ANOVA used the Fisher statistical test (F-test) to describe the distribution of experimental date based on the predicted results obtained from the model (Yuan *et al*., 2018). All the possible interactions and the significance of each variable that involved were measured. When the factors explain adequately the variation in the data about mean and estimated factor effects are reals, it shows that the F-value are greater from the unity (Azami *et al*., 2012).

Based on the result from Table 5, model F-value is 37.67 which implies that the model was significant. There is only 0.01% chance that a "Model F-Value" this large could occur due to noise. The p-value (Prob-F) of the selected model is <0.0001 which pointed out that the model terms are significant at 95% confidence level and the model was well fitted with the experimental data.. The model significance was acceptable when the p-value less than 0.5, while p-value more than 0.10 indicated that the model were not significant (Yuan *et al*., 2018).

Furthermore, the most important value that need to be considered is the p-value in lack of fit. The p-value in lack of fit must not be significant because to ensure that the model obtained from the CCD is well-fitted. So, the model must obtained the p-value of lack of fit that is higher than 0.05 to show that a model quality of fitness relative to pure error. Based on the result obtained, the p-value in lack of fit of the Fenton process is 0.1969 which indicated that the value is greater than 0.05. So it can be proved that the regression models identified in both of the processes are significant. The "Lack of Fit F-value" of 2.25 implies the Lack of Fit is not significant. There is only a 19.69% chance that a "Lack of Fit F-value" this large could occur due to noise.

Table 5 ANOVA for response surface quadratic model

Source	Sum of Square	Df	Mean Square	F Value	p-value Prob>F	
Model	35.99	9	4.00	36.76	<0.0001	Significant
A - Fe^{2+}	0.77	1	0.77	7.09	0.0238	
B – H_2O_2	24.82	1	24.82	228.15	<0.0001	
C–Reaction time	1.463E-003	1	1.463E-003	0.013	0.9100	
AB	0.57	1	0.57	5.21	0.0455	
AC	3.125E-004	1	3.125E-004	2.873E-003	0.9583	
BC	6.613E-003	1	6.613E-003	0.061	0.8102	
A^2	0.70	1	0.70	6.46	0.0293	
B^2	9.24	1	9.24	84.91	<0.0001	
C^2	0.036	1	0.036	0.33	0.5798	
Residual	1.09	10	0.11			
Lack of Fit	0.75	5	0.15	2.25	0.1969	not significant
Pure Error	0.33	5	0.067			
Cor Total	37.08	19				

The coefficients of determination R-squared controlled the fit of the models. The value of the R-squared must be close to one because it is the satisfactory of model on predicting the observation (Yuan *et al*., 2018).. Based on the ANOVA results in Table 6, the value of the R-squared obtained is 0.9707 which is 97.07% for the percentage colour removal by Fenton process. The suggested value for R-Squared must be at least 0.80 which confirms that the model is good predictability (Duc, 2014). The adjusted R-squared is 0.9443 which is 94.43% was obtained. The "Pred R-Squared" of 0.8326 is in reasonable agreement with the "Adj R-Squared" of 0.9443. The value of R-squared and adjusted R-squared obtained from the ANOVA are close to 1.0 which indicated that the value is very high and advocated a high correlation between the observed values and predicted values (Yuan *et al*., 2018). Therefore, the values of R^2 pointed out that the regression model gave an extremely good explanation and predictability for the relationship between the independent variable and response.

The Adequate Precision measures the signal to noise ratio by comparing the range of the predicted values at the design points to the average prediction errors. The ratio that must be obtained from the ANOVA must be greater than 4 because it is desirable. Based on the ANOVA result, the adequate precision obtained is 21.220 which indicates an adequate signal. This model can be used to navigate the design space. While the standard deviation (SD) and coefficient of variance (CV) must be smaller values to make sure that the experiment are valid and accurate. The value of the SD from the ANOVA is 0.33 and the value of CV is 0.33 which can be concluded that the value is small and the experiment is reliable.

Table 6 ANOVA for regression models

Types of Variance	Response for Fenton Process
R-Squared	0.9707
Adj R-Squared	0.9443
Pred R-Squared	0.8326
Adeq Precision	21.220
Std. Dev.	0.33
Mean	98.61
C.V. %	0.33
PRESS	6.21

The normal probability plot of residuals for the removal of colour in Fenton process was shown in Figure 3. Based on the graph obtained, the residuals fell on the straight line and the model adequately explains the experimental range studied. Based from the result achieved, it model can be assumed as adequate and reasonable.

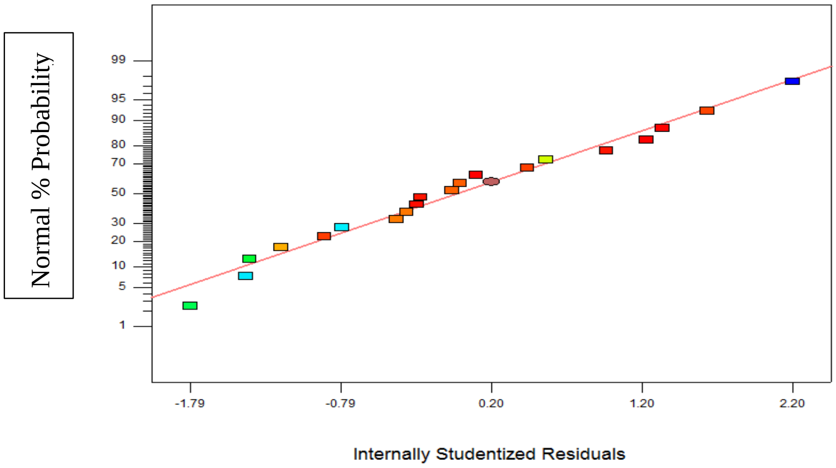

Figure 3. Normal probability plot of residuals for colour removal in Fenton process.

While Figure 4 shows the residuals versus predicted value for the removal of colour in Fenton process. Based on the plot, all the residuals data scattered randomly within the horizontal line of the ±3.00. The plot shows that no data are over than the horizontal line of ±3.00. So, the graph was proved that the value of residual was independent to the predicted value.

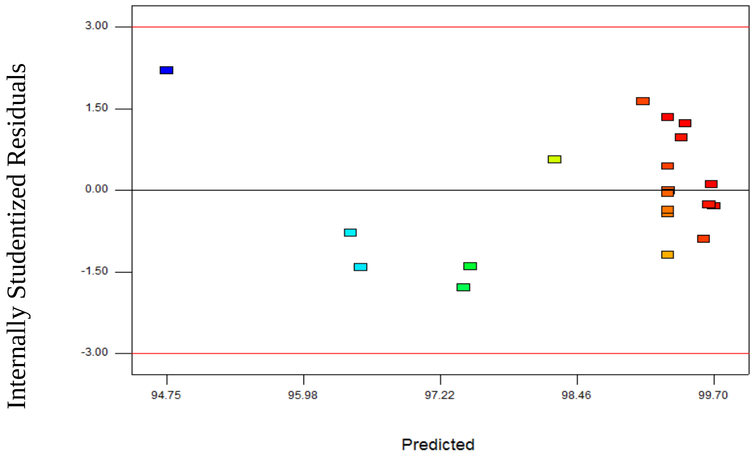

Figure 4. Residual versus predicted value for the colour removal in Fenton process.

The Figure 5 pointed out the predicted versus actual values for the removal of colour in Fenton process. The graph indicated the error between the predicted and actual that had been obtained from the experiments. The straight line on the graph represent the predicted values based from the model fitting technique. So it can be concluded that most of the data lies on the straight line which is the predicted line and the actual values is not much differ with the predicted value. The plot also proved that the model is reasonable and has high correlation between the actual result and predicted value (Lak *et al*., 2018).

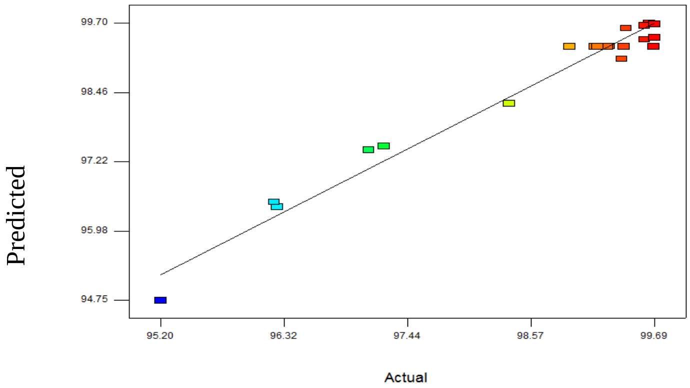

Figure 5. Predicted versus actual values for colour removal in Fenton process.

The empirical equations in terms of coded variables are as follows in Equation 3:

$$\text{Colour removal (\%)} = 99.28 + 0.24A + 1.35B + 0.010C - 0.27AB - 6.250E\text{-}003AC - 0.029BC - 0.22A^2 - 0.80B^2 + 0.050C^2 \quad (3)$$

The empirical equation in terms of actual factors are as follows in Equation 4:

$$\text{Colour removal (\%)} = 82.59113 + 1.34952\,(Fe^{2+}) + 0.10847\,(H2O2) - 0.0806082\,(\text{Reaction time}) - 1.71152E\text{-}003\,(Fe^{2+} * H2O2) - 2.94628E\text{-}004\,(Fe^{2+} * \text{Reaction time}) - 4.92832E\text{-}005\,(H2O2 * \text{Reaction time}) - 0.039023\,(Fe^{2+})^2 - 1.87138E\text{-}004\,(H2O2)^2 + 6.25044E\text{-}004\,(\text{Reaction time}) \quad (4)$$

4. CONCLUSION

As conclusion, the optimization of the Fenton oxidation to treat textile wastewater has been determining using Response Surface Method (RSM) based on the Central Composite Design (CCD) in this project. The effectiveness of the Fenton oxidation on the textile wastewater was studied and the parameters that had been set was observed in this project. The parameters that had been set for concentration of of Fe^{2+} are from 8.0 mM to 16.0 mM. While the concentration of H2O2 are from 60 mM to 280 mM and the reaction time between 60 min to 90 min. The optimal value for each parameter was obtained after further validation had been done. After the response was recorded, the optimum value for the concentration of Fe^{2+} is 9.62 mM, the optimum value for the concentration of H_2O_2 is 145.82 mM, and the optimum value for the reaction time is 83.92 min. The optimum value for each parameter contributed to the highest removal of the colour which is up to 98% where the concentration of Fe^{2+} and H_2O_2 was minimized.

By using RSM that based on the CCD, optimization approach, the optimum removal of the colour was obtained. The correlation coefficient value, R^2 obtained in this analysis was 0.9707 which indicated that 97.07%. Based on the R^2 value, the value pointed out a high degree of correlation between the experimental and the predicted values. The model F-value of 36.76 implies the model is significant. There is only a 0.01% chance that a “Model F-Value” this large could occur due to noise. While the Lack of Fit at p-value pointed out to be 0.1969 that indicated the value is not significant.

The range for each parameter that had been set give the removal of colour up to 99%. The highest removal of colour by using Fenton process is 99.69%. But the most optimum condition where the concentration of Fe^{2+} and H_2O_2 were minimized, the reaction time was in range and the percentage colour removal was maximized give the removal up to 98.19%.

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