



Application of Box–Behnken factorial design for parameters optimization of basic dye removal using nano-hematite photo-Fenton tool

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Abstract

A huge amount of water is consumed in the textile industry, and the result is the production of a large amount of wastewater. The treatment of such wastewater significantly reduces the pollution load. Oxidation by nano-Fenton reactions ($\text{Fe}^{3+}/\text{H}_2\text{O}_2$) is a reasonable and cost-efficient process for the remediation of harmful pollutants in wastewater. In the present study, nano-hematite was applied as a source of iron in Fenton's reagent for methylene blue dye removal from wastewater. The effects of different parameters, presence of nano-hematite, hydrogen peroxide concentrations and pH, were optimized using the response surface methodology technique. A Box–Behnken design was applied, and the response (dye removal) was maximized. A maximal dye removal (81.6%) was attained when wastewater was treated at pH 2.5 in the presence of nano-hematite and hydrogen peroxide in the amounts of 41 and 388 mg/L, respectively. The model is well fitted and described using the second-order polynomial equation. Moreover, the model validation showed a 97% fit between the theoretical and experimental ones.

Keywords Methylene blue dye · Photo-Fenton · Nano-hematite · Response surface methodology (RSM) · Statistical optimization

Introduction

Recently, one of the major problems facing industrialized societies is the pollution of the environment by hazardous chemicals, especially the aquatic environment. Such industries that cause water pollution are: paint, textile, dyeing, pharmaceutical industries, tannery and paper industry. The residual dyes from different sources contain a wide variety of organic pollutants that discharge into the natural water supplies or conventional wastewater treatment techniques. Textile industry is considered as one of the main causes of pollution problems worldwide that discharges severe dye-containing wastewaters. In general, the effluent wastewater

discharged from this dyeing industry is esthetically and environmentally unacceptable as it is a highly persistent of organic pollutants and has strong color and high pH value (Wang et al. 2007; Sathian et al. 2013). The volume of wastewater from the industries has increased and needs to be treated. Such wastewater could be reused in order to reduce the amount of discharged effluents into the environment once it has been treated.

Traditionally, textile wastewater management practices have been concerned with treating the effluents using various systems. Several physical, chemical and biological approaches are available for the treatment of dye effluents wastewater, for example coagulation–flocculation, filtration, biological treatment and chemical adsorption (Singer and Chen 1980; Mohapatra et al. 2010). However, these processes can only convert the contaminants from one phase to another one without destroying them. Moreover, their costs are very high for treating raw textile wastewater (Altinbas et al. 1995; Coloma 1998).

Recently, alternative advanced oxidation processes (AOPs), which include both homogeneous and heterogeneous photocatalysis, have been emerged as promising

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technologies. The main aim of those technologies is the mineralization of the largest number of dyes into biodegradable and harmless end products besides the color removal from such wastewater (Tang and An 1995; Andreozzi et al. 1990).

AOPs are a set of alternative systems that substitute the conventional methods; AOPs are proceeded along the creation of highly oxidative species specifically hydroxyl radicals ($\cdot\text{OH}$) which are accomplished of oxidizing the organics to such a level of harmless products, CO_2 and H_2O . The application of homogeneous catalysis in oxidizing different organics specially dyes concerning much attention. This is due to their high efficiency in the mineralization and oxidation such as $\text{Fe}^{n+}/\text{H}_2\text{O}_2$ (Fenton) and $\text{Fe}^{n+}/\text{H}_2\text{O}_2/\text{UV}$ (photo-Fenton) processes (Hsueh et al. 2005; Daneshvar and Khataee 2006). Compared to the other systems, the use of AOPs has the advantages that there is no sludge formation, considerably safe and its ease of operation. Besides, there is the advantage of short reaction time (Marechal et al. 1997).

The application of UV/Fenton has been reported extensively in the literature, though there is a scarcity of the literature published in the case of using the nano-Fenton catalyzed reactions. For example, Rahman et al. (2009) applied photo-Fenton's reagent for the oxidation of commercial textile dye wastewater named Malachite green. Such reagent was used for treating water containing diesel oil Tony et al. (2009) and synthetic wastewater contaminated by phenol Tony et al. (2017). The decolorization of methylene blue by Fenton's reagent is achieved by Liu et al. (2011). Furthermore, Tony et al. 2012 investigated the photo-Fenton's reagent for the mineralization of oil refinery effluents. Abou-Gamra (2014) applied the reagent for the treatment of red dye-contaminated wastewater. El Haddad et al. (2014) treated azo dye-polluted wastewater from textile industry using Fenton's reagent. In addition, Minella et al. (2014), Srodowska (2015) and Assadi and Eslami (2010) applied the reagent for the degradation of phenolic wastewater. Moreover, Khan et al. (2016) using magnetite + H_2O_2 + UV process as a source of Fenton's reagent investigated the photocatalytic degradation of methylene blue in wastewater.

To a very large extent in many studies, Fenton's reagent optimization parameters are still conducted on a trial-and-error finding, i.e., changing one factor at a time. However, this is an experimentation technique based on the variation of a single factor while fixing the all-remaining parameters at a certain set of conditions. Moreover, single-dimensional (factor) search is not only a time-consuming technique, but also the attained optimum conditions is not accurate due to the neglect of the interaction between the operating variables (Mason et al. 2003). To overcome this experimental problem, the response surface methodology (RSM) has been suggested to define the effects of individual parameters. RSM is referred to a set of mathematical and statistical techniques to establish an experimental design model.

Thus, investigating the consequences of various independent parameters on the response is conducted to locate the optimum conditions with a reduced number of experimental trials (Khuri and Cornell 1996). The experimental design used to establish the RSM model is based on the least squares method. The diagnostic checking tests specified by the analysis of variance (ANOVA) is used to check the adequacy of the proposed model. In addition, a graphical representation plots the response surface to locate the optimum and study the surface (Montgomery 1991). As illustrated in the literature, RSM was applied in many areas to optimize the process parameters (Tekindal et al. 2012; Duc 2014; Elboughdiri et al. 2015; Suarez-Escobar et al. 2016; Ramirez et al. 2005).

The objective of this current work is to locate the optimum conditions of Fenton's reagent amount and pH to attain the highest of methylene blue (MB) removal rate in wastewater. The optimization is carried out via Box–Behnken RSM experimental design. The interaction between Fe^{3+} , H_2O_2 and pH variables that affecting the dye removal rate is studied, and model describing the effect of those variables is explained.

Materials and methods

Materials

Methylene blue (MB) with a chemical formula of ($\text{C}_{16}\text{H}_{18}\text{N}_3\text{SCl}$) is selected as a model wastewater pollutant. Nano-powder, $\alpha\text{-Fe}_2\text{O}_3$, with spherical-shaped particles and size ranging from 6.1 to 18.3 nm is used as an iron source for the nano-photo-Fenton method. It was previously prepared in our laboratory using sol–gel technique. Moreover, H_2O_2 (30%) supplied by Sigma-Aldrich was used to initiate the Fenton's reagent. pH values were adjusted at the desired values using sulfuric acid and sodium hydroxide, and both are supplied by Alpha Chemicals.

Experimental methodology

A sketch of the photocatalytic processes is shown in Fig. 1. It consists of a glass recirculation tank (1.5 L) containing 500 mL of dye solution containing 10 ppm dye, which is subjected to stirring and connected to a tubular UV reactor for 90 min, according to the preliminary work. The solution is re-circulated through the reactor at a flow rate of about 25 mL/min by means of a peristaltic pump. The UV lamp is used as a light source. Reagents (including H_2O_2 and Fe^{3+}) are added according to the required concentrations, and pH is adjusted. The experiments for this nano-Fenton's process are carried out according to Box–Behnken with RSM to determine the dye removal efficiency under the optimum operational conditions.

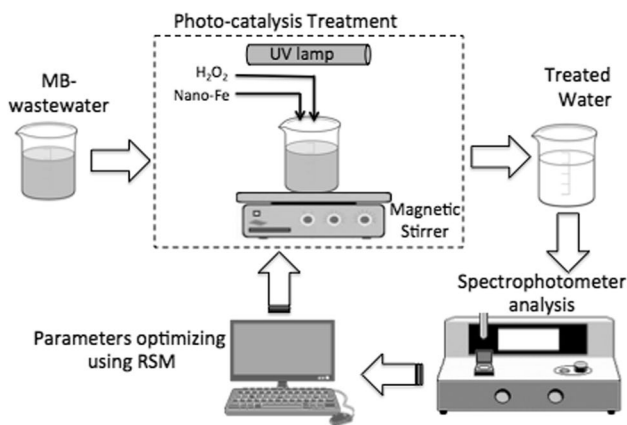


Fig. 1 Sketch of the experimental representation

Table 1 Range and levels of natural and corresponded coded variables for RSM

Variable	Symbols		Range and levels		
	Natural	Coded	-1	0	1
Fe ³⁺ (mg/L)	x ₁	X ₁	20	40	60
H ₂ O ₂ (mg/L)	x ₂	X ₂	200	400	600
pH	x ₃	X ₃	2.5	3.0	3.5

Analytical determinations

A Shimadzu spectrophotometer analyzer (UV-1601, Model TCC-240A, Japan) is used to measure MB dye concentration for each sample during the reaction time. The pH levels are monitored by using a digital pH meter. The dye removal performance is determined by the following formula:

Table 2 RSM for the three experimental variables in coded units and corresponding natural values

Experiment no.	Natural variable			Coded variable		
	Fe ³⁺ (mg/L)	H ₂ O ₂ (mg/L)	pH	X ₁	X ₂	X ₃
1	20	200	3.0	-1	-1	0
2	20	600	3.0	-1	1	0
3	60	200	3.0	1	-1	0
4	60	600	3.0	1	1	0
5	40	200	2.5	0	-1	-1
6	40	200	3.5	0	-1	1
7	40	600	2.5	0	1	-1
8	40	600	3.5	0	1	1
9	20	400	2.5	-1	0	-1
10	60	400	2.5	1	0	-1
11	20	400	3.5	-1	0	1
12	60	400	3.5	1	0	1
13	40	400	3.0	0	0	0
14	40	400	3.0	0	0	0
15	40	400	3.0	0	0	0

$$\gamma(\%) = \frac{(C_o - C_t)}{C_o} \times 100 \tag{1}$$

where γ is the percentage dye removal performance and C_o and C_t are dye concentrations in the aqueous solutions before and after treatment, respectively. The color removal performance was used as a response in a dye removal model.

Experimental design

Box–Behnken design applied in this study as the RSM tool including three variables, Fe³⁺ and H₂O₂ dosages and pH. The Box–Behnken is involving 15 runs. Table 1 presents the ranges and the levels of the experimental parameters in the present investigation. The detailed experimental design in the study is given in Table 2 as coded variables; in addition, the table represents the natural levels used in its original units of measurements.

The second-order polynomial equation model (Montgomery 1991) to predict the optimum value of three factors can be stated according to Eq. (2):

$$\gamma = \beta_0 + \sum \beta_i X_i + \sum \beta_{ij} X_i^2 + \sum \sum \beta_{ij} X_i X_j \tag{2}$$

where γ is the predicted response (phenol removal rate, %) used as a dependent variable, $i = 1, 2, 3$ and $j = 1, 2, 3$; β_0 , β_i , β_{ii} and β_{ij} are the model regression coefficient parameters; and X_i is the input controlling coded variable. In addition, the natural variables of the operating system (x_i)

were transferred to coded variables (X_i) according to Eq. (3) (Montgomery 1991) to simplify the model calculations.

$$X_i = \frac{(\text{value of operating variable } i) - (\text{its upper level} + \text{its lower level})/2}{(\text{its upper level} - \text{its lower level})/2} \quad (3)$$

Statistical Analysis System (SAS) (1990) was used for regression. In addition, in order to obtain the interaction between the parameters and their response, analysis of variances (ANOVA) was applied to graphically analyze the data. Square of the correlation coefficient, R^2 , is used as a tool to check the fitness of the polynomial model. Moreover, using the same program, the statistical significance of the model was evaluated using Fisher F -test. The F -test is used as the criterion to evaluate the statistical significance of the regression coefficients of the determined parameters [Eq. (4)]. The analysis of variance includes source (the source of the variation); DF (the degree of freedom); SS (the sums of squares of the dependent variables); MS (the mean squares) which are the sums of squares divided by the degree of freedom; Fisher F values; and probability p values (SAS 1990; Montgomery 1991).

$$F \text{ value} = \frac{\text{Between groups variance}}{\text{Pooled variance}} \quad (4)$$

The probability (p value) of the given statistical model determines the significance of the model to accepted or rejected. Generally, based on the p value the model is accepted with 95% confidence level. Three surfaces dimensional plot curves (using MATLAB 7.0 software) and their corresponding contour plots were attained based on the effects of the levels of the three variables (Fe^{3+} and H_2O_2 dosage and pH). These 3-D plots the simultaneous interaction of two variables on the response while keeping the third variable constant in the polynomial equation. Furthermore, based on the main variables in the 3-D surface and contour plots, the optimum region is determined. Finally, Mathematica software (V 5.2) was applied to locate the accurate optimum operating parameters.

Results and discussions

Optimization of Fenton's reagent parameters with RSM

RSM model

According to the experimental design illustrated in Table 2, experiments were conducted and the corresponding results are given in Table 3, which lists the theoretical and experimental dye removal (%). The second-order model is well

fitted to the theoretical data, the following experimental model given in terms of its coded values is achieved for

the dye removal (%), and it illustrates both the effects of the three operating variables and their interactions on the response:

$$\begin{aligned} \gamma = & 68.09 + 1.94X_1 - 2.94X_2 - 14.35X_3 \\ & - 13.97X_1^2 - 3.03X_1X_2 + 13.70X_1X_3 \\ & - 27.91X_2^2 - 5.10X_2X_3 - 6.36X_3^2 \end{aligned} \quad (5)$$

Generally, the model fitting is checked in order to confirm the adequacy of approximation of the model to the real application. However, it is necessary to check the model fitting approach, based on the investigated and optimized response surface which probably gives a poor result. Therefore, the investigative plots of the predicted against actual values give an adequate judgment of the model. Figure 2 illustrates the plot of the predicted values versus actual one for the MB dye removal. As illustrated in Fig. 2, a good agreement is attained between the predicted and the actual data for the MB dye removal (%) system; thus, the second-order regression model achieved is satisfied. The R^2 of the fitted model is 97%.

Effect of independent variables and their interaction

MATLAB software has been used to build the response surfaces by fixing one variable in the polynomial equation to

Table 3 Experimental and predicted achieved removal responses for RSM after 30 min of reaction time

Experiment no.	γ (%)	
	Experimental results	Predicted response
1	26.13	24.18
2	31.00	24.34
3	27.45	34.11
4	20.22	22.17
5	50.00	45.98
6	28.23	27.54
7	49.60	50.33
8	07.42	11.40
9	67.91	73.86
10	53.00	50.34
11	15.10	17.76
12	55.00	49.05
13	68.12	68.08
14	68.14	68.08
15	68.00	68.08

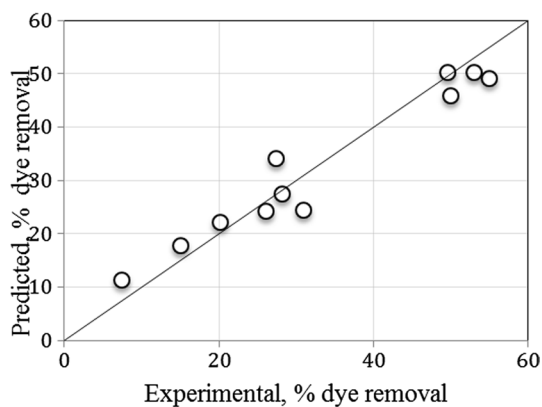


Fig. 2 Box-Behnken design plot for predicted versus actual values for dye removal

plot a 3-D representation of two independent variables with their response (represented in MB removal percentage after 90 min of reaction time).

Figures 3, 4, 5, 6, 7 and 8 illustrate two different representations of surface and contour plots of each two interaction parameters for the response attained with the experimentally adjusted equation. As illustrated in Figs. 3 and 4, for all iron concentrations, the H_2O_2 load has an optimum value in which MB removal rate reaches its maximum. However, when higher concentrations of hydrogen peroxide are applied, MB removal rate is decreased. In addition, a strong effect of H_2O_2 effect is obtained at a low iron loads as shown in Fig. 3.

In addition, Figs. 3 and 4 give data of different $\alpha-Fe_2O_3$ initial concentration and percentages of dye reduction. Adding a too high initial $\alpha-Fe_2O_3$ concentration results in lowering the detrimental effect because of a smaller excess

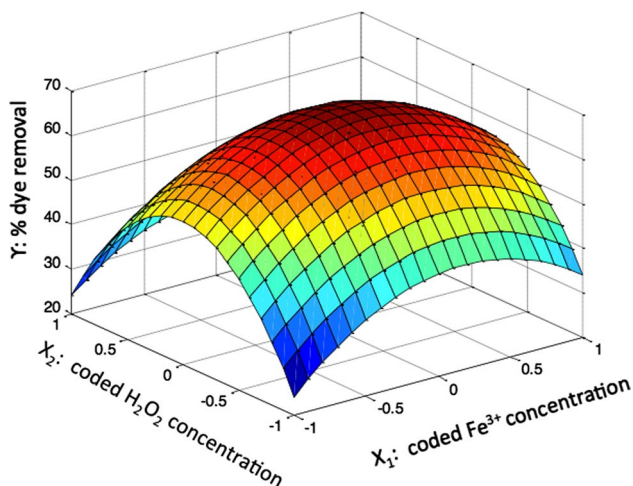


Fig. 3 Surface graph of dye removal showing interaction of Fe^{3+} dosage and H_2O_2

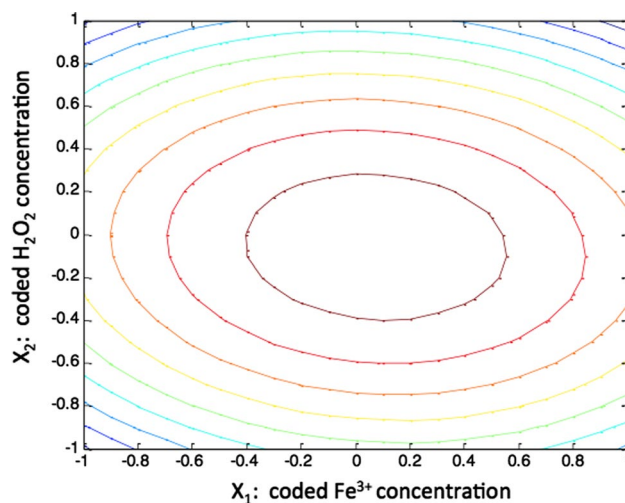


Fig. 4 Contour plot of dye removal showing interaction of Fe^{3+} dosage and H_2O_2

of H_2O_2 . In addition, when the iron salt increases, the dye removal rate is increased; however, after a certain limit, the increase in such reagent is unfavorable. The detrimental effect of high H_2O_2 loads could be described by the fact that instead of H_2O_2 producing the reactive species to mineralizing the dye, it reacts with the one of the reactive species (OH radicals), and therefore, the quantity of OH radicals reduced and the overall reaction rate thus reduced (Tony et al. 2009). Thus, the doses of the hematite and hydroxide should be in an optimal balance.

As shown in Fig. 4, it is possible to change the Fenton reagent ratios ($Fe^{3+}:H_2O_2$) while keeping the remaining experimental conditions at constant values. Moreover, it is possible to increase or decrease the dye reduction (Fig. 3) after 90 min of treatment under UV light irradiation.

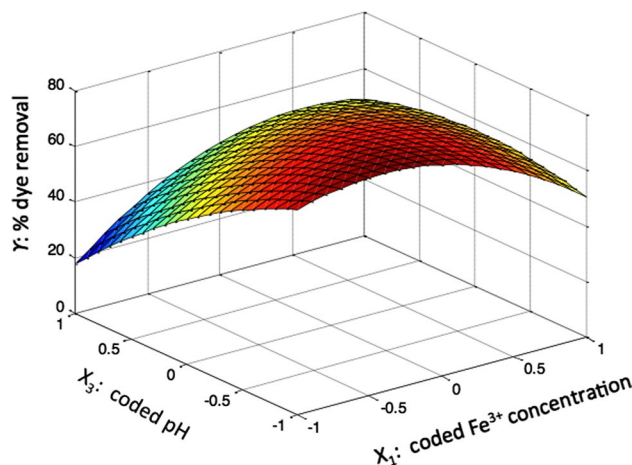


Fig. 5 Surface graph of dye removal showing interaction of Fe^{3+} dosage and pH value

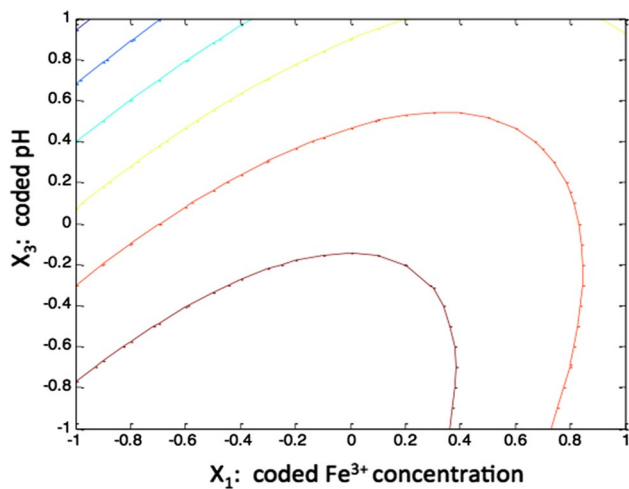


Fig. 6 Contour plot of dye removal showing interaction of Fe^{3+} dosage and pH

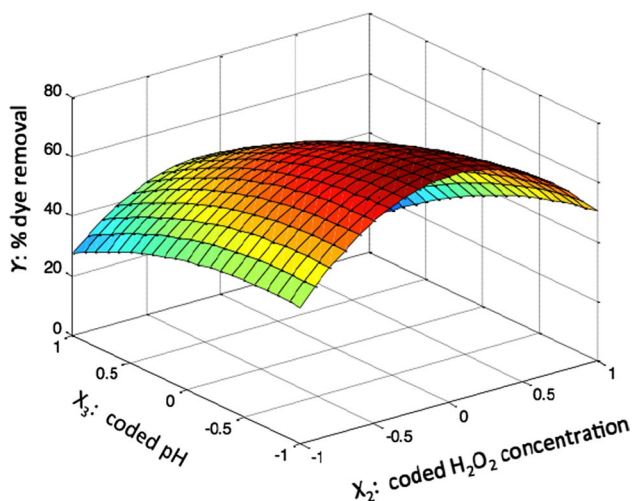


Fig. 7 Surface graph of dye removal showing interaction of H_2O_2 dosage and pH value

Figure 5 represents the effects of Fe^{3+} concentration on the color removal after 90 min of reaction. Usually, the effect of both pH and/or iron load may be positive or negative on the rate of MB removal. From Fig. 6, which illustrates the contour plot of the coded Fe^{3+} concentration and pH, the range of optimum value of the dye removal is related to both Fe^{3+} concentration and pH. Indeed, it is the same trend as previously illustrated in the literature; the optimum pH sharply affects the removal rate (Benatti et al. 2006; Torrades and García-Montaña 2014).

Thus, it could be concluded that any variable could affect the reaction rate positively or negatively, depending on the interaction effect of the other parameters. This validates the application of Box–Behnken tools for system optimization.

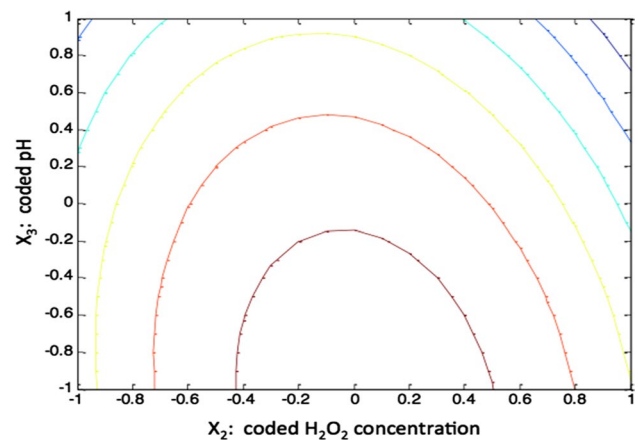


Fig. 8 Contour plot of dye removal showing interaction of H_2O_2 dosage and pH value

A similar performance is observed at a constant Fe^{3+} load, while varying the pH and H_2O_2 load (Figs. 7, 8). This could be illustrated by the fact that at some experimental conditions, very high H_2O_2 load causes a decrease in the overall MB dye removal rate, and this is due to the competition between these species for attacking the highly reactive OH radical species. Certainly, rather OH radicals are a non-selective particles; thus, it reacts with the organic matter present in wastewater as well as with other species (Freitas et al. 2014; Duc 2014; Elboughdiri et al. 2015).

It could be concluded from Figs. 5, 6, 7 and 8) that at optimum pH value the optimum concentration of both reagents (Fe^{3+} and H_2O_2) seems to affect positively the final performance.

ANOVA for response surface model

The effect of a certain factor is the change in response produced by the change in the level of that factor. When the effect of a factor depends on the level of another factor, the two factors are said to be interacting. In order to further assess the polynomial model (5) taking into account the interaction of factors, statistical analysis of variance (ANOVA) using SAS software was conducted and the statistical significance of the factors toward the response (γ) of the process was determined by Fisher's F -test (SAS 1990; Montgomery 1991; Torrades et al. 2003; Benatti et al. 2006).

Coefficient of determination, R^2 , was calculated to measure the degree of fit of the model. The R^2 value is expressed as the ratio of the explained variation to the total variation. It additionally provides a measurement of the proportion of the variability in the observed response variables and how it explains variables and their interactions (Haber and Runyon 1977). Moreover, the better the empirical model to predict the response accompanied by good fitting with the actual data

Table 4 ANOVA coefficient of regression and *t* checking

Variable	SD	<i>T</i>	<i>p</i> > <i>t</i>	Coefficient
X_1	2.314715	0.83844	0.440013	1.94075
X_2	2.314715	-1.27300	0.258995	-2.94663
X_3	2.314715	-6.19736	0.001596	-14.3451
X_1X_1	3.407168	-4.10112	0.009345	-13.9732
X_1X_2	3.273502	-0.92409	0.397846	-3.02500
X_1X_3	3.273502	4.18619	0.008603	13.70350
X_2X_2	3.407168	-8.19257	0.000441	-27.9135
X_2X_3	3.273502	-1.55896	0.179748	-5.10325
X_3X_3	3.407168	-1.86664	0.120934	-6.35996

R^2 , coefficient of determination, values were 0.97 for dye percent removal

when R^2 approaches unity. However, a low R^2 value indicates that there is a less relevance of the dependent parameters in the model to illustrate the performance variation (Little and Hills 1978; Mendenhall 1975; Joglekar and May 1987).

As indicated in Table 4, the response surface models established in this investigation, for estimating the MB dye removal rate of nano-hematite photo-Fenton’s reagent, were adequate. Student’s *t* test was used as a tool to establish the significance of the regression coefficients of the variables. In addition, the probability values (*p* values) were used as a measurement to validate the significance of the model. Table 4 shows the coefficients of the variables in the models and their corresponding R^2 . Besides, ANOVA, the high correlation coefficient value of R^2 of 0.97 indicates adequacy of the applied model and how it fits the experimental data; the model explains 97% of the response variability. The value of R^2 also confirms that the model has good predictability, for which at least $R^2 = 0.80$ is suggested (Montgomery 1991).

The analysis of variance (ANOVA) and coefficient of correlation (R^2) were applied to evaluate the fit the model adequacy. Table 5 represents ANOVA for the linear and quadratic model proposed to explain the response of the MB dye removal, based on significant terms of the variance analysis of the effects. ANOVA, with 97% reliability and a *p* value which is less than 0.05 (SAS 1990; Montgomery 1991), showed the significant factor for the removal of

Table 5 Analysis of variance (ANOVA) for the RSM model

Source	Degree of freedom (DF)	Sum of squares (SS)	Mean squares (MS)	<i>F</i> values	<i>p</i> > <i>F</i>
Model	9	6076.486	675.1651	15.75	0.004
Linear	3	1745.854	1745.854	40.73	0.701
Square	3	3747.178	3747.179	87.42	0.130
Interaction	3	891.9187	0891.919	20.81	0.586
Error	5	214.3162	042.8633		
Total	14	6290.802			

the dye with Fenton’s reagent to the quadratic model. The developed model showed the Fisher *F*-test of the regression of 15.75 and with a low probability of exceedance *p* value = 0.004, indicating that the model is highly statistically significant.

Determination of optimal removal conditions

Based on the experimental data and the developed model for simulating the decolorization performance, the optimum conditions for the maximum value of the decolorization efficiency can be determined by using Mathematica software. The criteria for three variables in correspondence with decolorization performance are shown in Table 6. The model developed in this study predicted that the optimum conditions for the highest dye removal performance were 41 and 388 mg/L for Fe^{3+} and H_2O_2 , respectively, and 2.5 pH and 81.5% for predicted dye removal.

Finally, a model describing photo-Fenton based on nanoparticles reagent for remediation of a synthetic prepared MB dye wastewaters was established based on Fenton reagent loads and pH, as related to the initial organic matter load in the wastewater. The model obtained was revealed to appropriate of the predicting for MB dye removal rate within the ranges of the investigated parameters.

Model validation

Under the optimum conditions given in Table 6, three replicates were conducted and the average dye removal efficiency was 80% that is very close to the predicted value

Table 6 Optimum values of the process parameters for maximum efficiency

Parameter	Optimum value
γ (dye removal, %)	81.6
Fe^{3+} (mg/l)	41
H_2O_2 (mg/l)	388
pH	2.5

Table 7 Predicted and experimental values for the responses at optimum conditions

Type of value	Dye removal (%)
Predicted	81.6
Experimental	80.0

of 81.6% (as shown in Fig. 7) for 30 min of reaction. The validity of the model for the dye removal performance of MB was confirmed by the high correlation concerning the experimental and predicted data.

This method provides savings in terms of time and the amount of material by limiting the amount of reagent used (Table 7).

Conclusion

Optimization of nano-photo-Fenton reagent with respect to the dye removal rate for the treatment of MB dye-contaminated wastewater effluents has been examined. Box–Behnken experimental design based on response surface methodology was used to locate the optimum operating variables to maximizing the dye removal efficiency. The Fenton dosages and pH are both important terms to attain higher dye removal rate. The model developed using RSM for dye removal can be applied for predicting MB dye removal rate within the ranges of the variables investigated. The optimum region for the photo-Fenton's process system is determined using RSM technique. The optimum values of the operating parameters are 41 and 388 mg/L for Fe^{3+} and H_2O_2 , respectively, and pH 2.5, where 80% of dye removal can be obtained.

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