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Research Article

Experimental Design of Photo-Fenton Reactions for the Treatment of Car Wash Wastewater Effluents by Response Surface Methodological Analysis

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Establishing a treatment process for practical and economic disposal of car wash wastewater has become an urgent environmental concern. Photo-Fenton's process as one of the advanced oxidation processes is a potentially useful oxidation process in treating such wastewater. Lab-scale experiments with UV source, coupled with Fenton's reagent, showed that hydrocarbon oil is degradable through such a process. The feasibility of photo-Fenton's process to treat wastewater from a car wash is investigated in the present study. A factorial design based on the response surface methodology was applied to optimize the photo-Fenton oxidation process conditions using chemical oxygen demand (COD) reduction as the target parameter to optimize. The reagent (Fe^{2+} and H_2O_2 concentration) and pH are used as the controlling factors to be optimized. Maximal COD reduction (91.7%) was achieved when wastewater samples were treated at pH 3.5 in the presence of hydrogen peroxide and iron in amounts of 403.9 and 48.4 mg/L, respectively.

1. Introduction

Car washing leads to disposal of large amounts of oily polluted water which results in potentially high levels of nutrients, metals, and hydrocarbons flowing into storm drains. The composition of pollutants found in car wash wastewater varies according to the way of washing, mechanical car washing or artificial high-pressure water washing, and the size and type of vehicle (e.g., small car, truck, commercial van, etc.). In some cases, car wash wastewater may also contain heavy metals [1–3].

Considering the large volume of wastewater generated from the car washing process, wastewater treatment coupled with recycling may possibly be an essential water quality measure. For instance, in the US, commercial car wash facilities either recycle or treat their wash water prior to discharge to the sanitary sewer system, so most storm water

impacts from car washing are from residential car wash systems that discharge polluted wash water into the storm drain system [1]. Some countries, for example, Switzerland, Germany, and The Netherlands, no longer allow outdoor car washing away from car washing stations [4].

In Egypt, as well as in many countries worldwide, car wash activities within petrol stations and outdoor car washing are among those activities that pose an environmental threat to the main freshwater source, the river Nile, which is already subjected to untreated wastewater [5]. Consequently, there is a growing need for research particularly on the application of innovative technologies in the treatment of such kind of wastewater.

The development of novel treatment methods encompasses investigations of advanced oxidation processes (AOPs), which are characterized by the production of the hydroxyl radical ($\cdot\text{OH}$) as a primary oxidant [6]. Examples of

TABLE 1: Properties of chemicals used in the study¹.

Compound	Molecular weight	Formula	Manufacturer	Purity
Iron chloride tetrahydrate	198.8	FeCl ₂ ·4H ₂ O	Sigma-Aldrich	98.0%
Hydrogen peroxide	134.01	H ₂ O ₂	Sigma-Aldrich	30 wt%
Sulfuric acid	98.08	H ₂ SO ₄	Sigma-Aldrich	97.0%

¹Hydrogen peroxide solution with a stabilizer (dipicolinic acid (approximately 40 mg/L)).

AOPs include the use of hydrogen peroxide with ultraviolet light (H₂O₂/UV) to treat hazardous compounds [7], Fenton and photo-Fenton reagent (H₂O₂/Fe²⁺) [8–10], semiconductor photocatalysis [11], and the sonolysis process using ultrasonic irradiation [12]. Among various AOPs, the Fenton reagent is one of the most effective methods for treating various industrial effluents including wastewater [13, 14] and oily wastewater [15]. Previous work by the authors has involved the application of Fenton and the photo-Fenton reagents for the treatment of water polluted with diesel oil emulsion [10, 16]. Although Fenton reagent has been reported extensively in the literature, there is a scarcity of publications focusing on its use for the treatment of car wash wastewater.

The main aim of the present study is to explore the possibility of treating car washing water to an acceptable level that can be recycled and reused for the same application. The study is of both national and international importance as it targets two global water issues: water conservation and water pollution. The study outlines the application of the photo-Fenton process to the mineralization of car wash wastewater. The effect of the reaction operating conditions is investigated and the factors that control the Fenton reaction process (Fe²⁺, H₂O₂, and pH) are optimized. Furthermore, the experimental design of the study applies a well-established [17–19] statistical-based technique, commonly known as RSM (response surface methodology) [20], to explore optimum range of values of Fe²⁺, H₂O₂, and pH for the maximum COD removal.

Factors to control the Fenton reaction process are the amounts of Fe²⁺ and H₂O₂ and the working pH. Optimizing such parameters plays a key role towards the achievement of the Fenton reaction. The experimental design using a statistical-based technique, commonly known as RSM (response surface methodology) [20], has been increasingly applied in many fields including wastewater treatment to study the optimization of the treatment process.

2. Materials and Methods

2.1. Car Wash Wastewater. Wastewater samples were collected from a car washing wastewater tank at a petrol station in the south of Egypt. The principal properties of the wastewater are 82 mg-COD/L, turbidity of 28.1 NTU, pH 8.2, and suspended solids of 55 mg/L.

2.2. Experimental Materials. Fe²⁺ in Fenton's reagent (Fe²⁺/H₂O₂) is prepared by making a solution from Fe²⁺ salt. H₂O₂ was obtained in liquid (30% of H₂O₂, wt) from

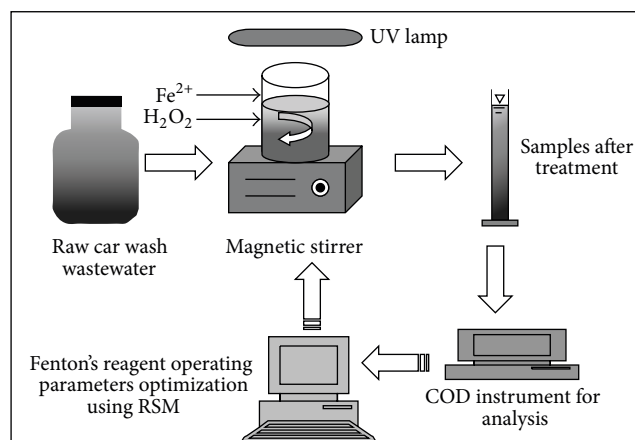


FIGURE 1: Schematic diagram of a lab-scale photo-Fenton test.

a commercial supplier. Sulfuric acid is used for adjusting the pH of the wastewater samples during treatment. Properties of chemicals used in this study are listed in Table 1.

2.3. Methodology. All photochemical experiments were carried out in a batch mode laboratory scale unit using a 250 mL beaker. Initially, the pH value of 100 mL of the car wash wastewater samples was adjusted at the desired values with sulfuric acid before being subjected to oxidation. Then, ferrous ions solution and hydrogen peroxide were added to produce hydroxyl radicals. Subsequently, the mixture was subjected to magnetic stirring and UV radiation (254 nm wavelength), as illustrated in Figure 1. Samples were taken at regular time intervals in the discontinuous experiments and analyzed.

2.4. Analytical Determinations. The COD measurements were performed using HACH analyser (model HACH DR-2400). Turbidity was undertaken using a HACH 2100N IS Turbidity meter (ISO method 7027). The pH of the wastewater was adjusted using a digital pH-meter (model PHM62 Radiometer).

2.5. Experimental Design. The Fenton oxidation process was optimized by applying the response surface methodology [20]. COD removal, defined by (1), of the effluents was used as the variable to be optimized. The amounts of H₂O₂, Fe²⁺, and pH were chosen as the control factors to be optimized.

TABLE 2: Range and levels of natural and corresponding coded variables for RSM.

Variable	Symbols		Range and levels		
	Natural	Coded	-1	0	1
Fe ²⁺ (mg/L)	δ_1	x_1	30	40	50
H ₂ O ₂ (mg/L)	δ_2	x_2	350	400	450
pH	δ_3	x_3	3.5	6	8.5

TABLE 3: RSM for the three experimental variables in coded units and corresponding natural values.

Experiment number	Natural variable			Coded variable		
	Fe ²⁺ (mg/L)	H ₂ O ₂ (mg/L)	pH	x_1	x_2	x_3
1	30	350	6	-1	-1	0
2	30	450	6	-1	1	0
3	50	350	6	1	-1	0
4	50	450	6	1	1	0
5	40	350	3.5	0	-1	-1
6	40	350	8.5	0	-1	1
7	40	450	3.5	0	1	-1
8	40	450	8.5	0	1	1
9	30	400	3.5	-1	0	-1
10	50	400	3.5	1	0	-1
11	30	400	8.5	-1	0	1
12	50	400	8.5	1	0	1
13	40	400	6	0	0	0
14	40	400	6	0	0	0
15	40	400	6	0	0	0

The initial design involved 15 tests, based on a three-level Box-Behnken factorial design [20]:

$$\eta (\%) = \frac{\text{COD}_o - \text{COD}}{\text{COD}_o} \times 100, \quad (1)$$

where η is the percentage of COD removal; COD_o measured COD in supernatant before oxidation (mg-O₂/L); and COD is the COD value after the treatment.

The first step in the RSM is to find a suitable approximation for the true functional relationship between the response (η) and the set of independent variables. The following response function was used to correlate the dependent and independent variables in the response surface:

$$\eta = \beta_o + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_i^2 + \sum_i \sum_{j=i+1}^3 \beta_{ij} X_i X_j, \quad (2)$$

where η is the predicted response; $i = 1, 2, 3$ and $j = 1, 2, 3$; β_o is the constant coefficient (intercept); β_i are the linear coefficients; β_{ij} are the cross product coefficients; and X_i is the input controlling coded variable. In addition, the natural variables of the operating system (ξ_i) were transferred to coded variables (X_i) according to (3) [20] to simplify the model calculations. The results of COD removal and the turbidity were analysed through the statistical analysis

software package of SAS Institute, Inc., [17] by performing the analysis of variance (ANOVA) and fitted with a second-order polynomial model:

$$x_i = \frac{(\delta_i) - (\text{its upper level} + \text{its lower level}) / 2}{(\text{its upper level} - \text{its lower level}) / 2}. \quad (3)$$

The combined effect of the three independent variables, that is, Fe²⁺ concentration, H₂O₂ concentration, and initial pH, is represented as δ_1 , δ_2 , and δ_3 , respectively. The range of the experimental variables investigated in the study and the time of reaction (1 hr) were chosen according to preliminary tests. Therefore, each variable ranged between -1 and 1, as the lower and upper levels, respectively. These ranges and levels are presented in Table 2. Fifteen runs were required for a complete set of the experimental designs.

3. Results and Discussions

3.1. Model Fitting. The three-level experiments were carried out according to the Box-Behnken design and the experimental plan is shown in Table 3 as coded and natural levels. The data shows the results of the photo-Fenton experiments as an average of three duplicate experimental results at each

TABLE 4: Experimental and predicted achieved removal responses for RSM.

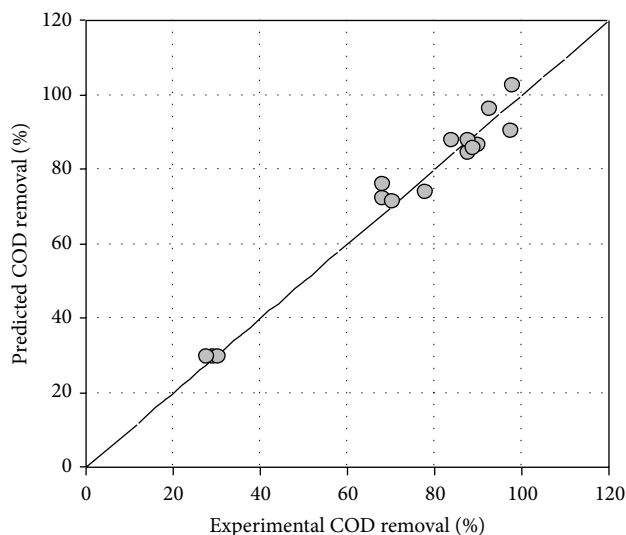
Experiment number	η (%)	
	Experimental results	Predicted response
1	68	72
2	88	84
3	93	96
4	90	86
5	98	90
6	71	71
7	88	88
8	68	76
9	84	88
10	98	102
11	78	74
12	89	85
13	29	29
14	31	29
15	28	29

operating condition. The following is the second-order fitting polynomial equation of coded factors:

$$\begin{aligned} \eta(\%) = & 29.30 + 6.48X_1 + 0.60X_2 - 7.70X_3 + 30.86X_1^2 \\ & - 5.50X_1X_2 - 0.75X_1X_3 + 24.63X_2^2 + 1.85X_2X_3 \\ & + 27.18X_3^2. \end{aligned} \quad (4)$$

The values of COD of the car washes wastewater as the responses obtained from the experiments and the predicted values are shown in Table 4 and plotted in Figure 2. A satisfactory agreement between the experimental and predicted data is achieved (Table 4). This is confirmed in Figure 2 which shows a regression coefficient R^2 value of 0.97 (the model being rejected if the R^2 value is less than 0.8 [20]). Thus, it is reasonable to state that the polynomial model (2) is a reliable tool to describe the Fenton reaction behaviour in car washing wastewater treatment.

3.2. Statistical Analysis. 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 (4) taking into account the interaction of factors, statistical analysis of variance (ANOVA) using SAS software was conducted and the statistical significance of the factors towards the response (η) of the process was determined by Fisher's F -test (F -value is the ratio of mean square of regression to the mean square of the error) [17, 20]. Student's t -test was used to determine the significance of the regression coefficients of the parameters. The probability values (P values) were used as a tool to check the significance of the model. In general, if the significance probability value ($P > F$) is small (below 0.05) and the

FIGURE 2: Predicted versus experimental data for COD removal (%) ($R^2 = 0.97$).

P value is lower than 0.01, the model is acceptable [17]. ANOVA of the tested model (Tables 5 and 6) indicated that the model is significant since the F -model is 19.94 and has a low probability value ($P > F = 0.002105$).

The response (COD removal, %) surfaces of two-dimensional contour plots and three-dimensional curves, generated by MATLAB 7.0, notably illustrate the relations between two interacting factors with the response (η), while the third factor was kept constant at zero. Figure 3 shows the response under the variable concentrations of Fe^{2+} and H_2O_2 . It demonstrates a considerable enhancement of COD removal (%) when the H_2O_2 concentration was increased. However, at higher concentrations of H_2O_2 the reduction rate was negatively affected. This trend (the decline of % COD removal with H_2O_2 concentrations higher than the optimum) is more evident when the iron concentration is low. Thus, an increase in the concentration of this reagent does not grant a continuing improvement to the COD removal efficiency of the treated wastewater. Similarly, the reduction percentage of COD demonstrated an increase with increasing Fe^{2+} concentration to a certain point after which it became slower. This indicates that there is an optimal dosage for both Fe^{2+} and H_2O_2 concentrations. Similarly, the 3D surface and the corresponding contour plot in Figure 4 show that the combination of Fe^{2+} concentration and pH has a significant effect on COD removal. The detrimental effect of higher H_2O_2 concentration is probably due to both autodecomposition of H_2O_2 into oxygen and water and the recombination of OH radicals [21]. If either H_2O_2 or Fe^{2+} is not present in optimal dosage, it will scavenge OH radicals and reduce their available amount in solution [19]. Figure 5 demonstrates that the increase in pH with the increase in the H_2O_2 concentration enhanced the rate of COD removal in a certain zone, beyond which less reduction of COD is observed. Therefore, optimising the sensitive parameters

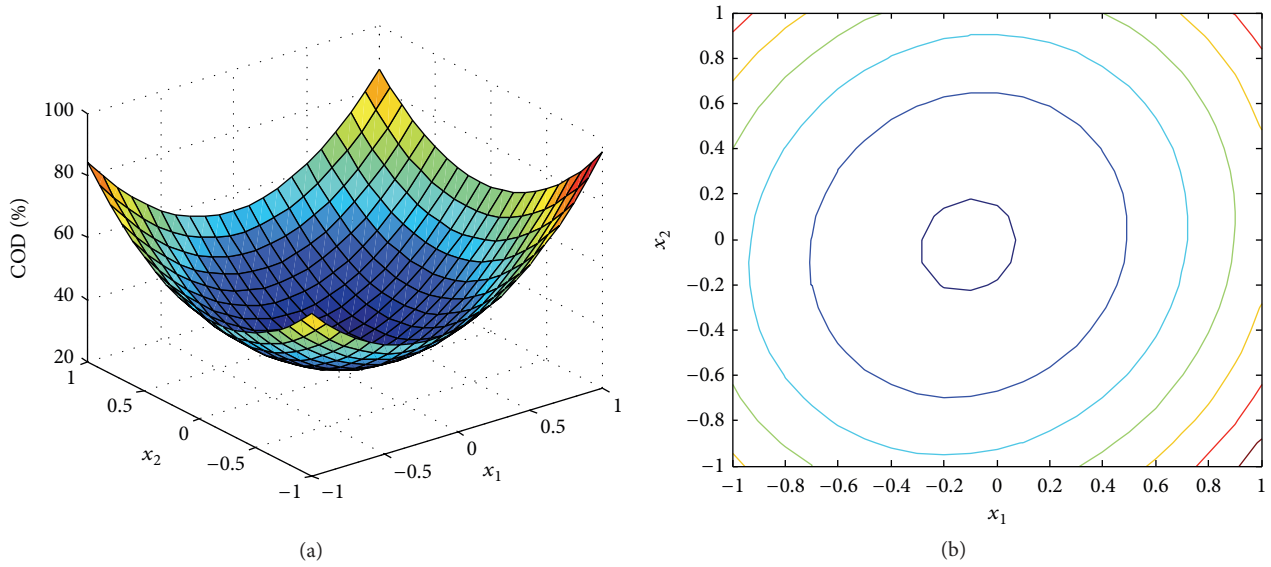


FIGURE 3: 3D surface and contour plot of response surface curve for COD removal showing interaction between (a) Fe^{2+} and (b) H_2O_2 .

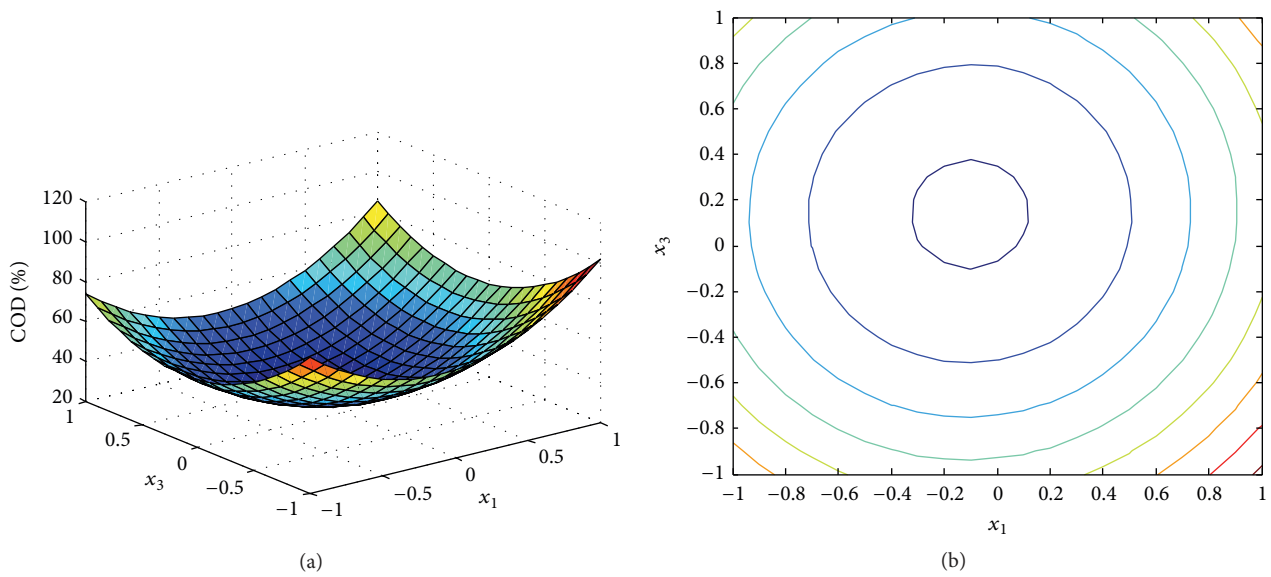


FIGURE 4: 3D surface and contour plot of response surface curve for COD removal showing interaction between Fe^{2+} and pH.

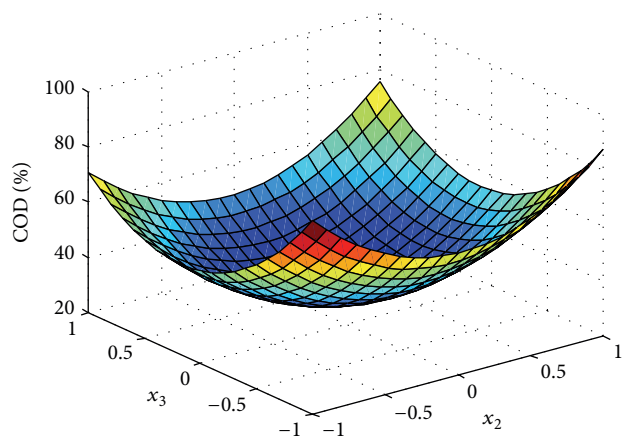
TABLE 5: ANOVA coefficient of regression and t checking¹.

Variable	Standard deviation	T	$P > t$	Coefficient
X_1	2.405956	2.691238	0.043235	6.475
X_2	2.405956	0.249381	0.812987	0.6
X_3	2.405956	-3.20039	0.023985	-7.7
X_1X_1	3.541472	8.704009	0.000331	30.825
X_1X_2	3.402536	-1.61644	0.166922	-5.5
X_1X_3	3.402536	-0.22042	0.834259	-0.75
X_2X_2	3.541472	6.953324	0.000945	24.625
X_2X_3	3.402536	0.543712	0.609995	1.85
X_3X_3	3.541472	7.673364	0.000599	27.175

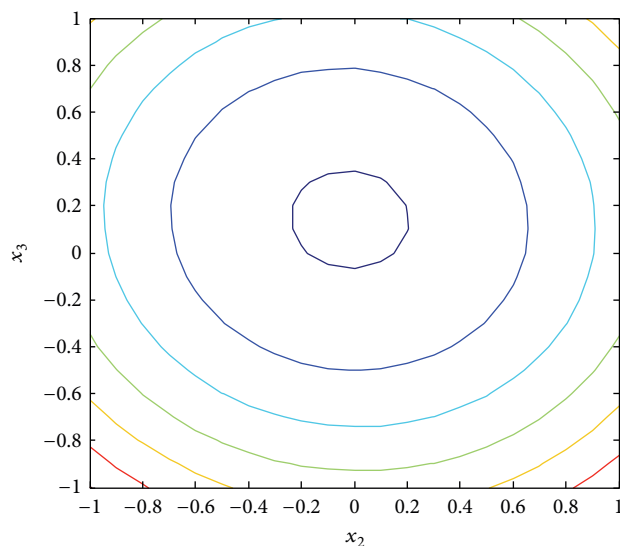
¹ R^2 : coefficient of determination; values were 0.97 for COD percent removal.

TABLE 6: Analysis of variance (ANOVA) for the RSM model.

Source	Degree of freedom (df)	Sum of squares (SS)	Mean squares (MS)	F statistics	P > F
Model	9	8309.248	923.2498	19.93673	0.002105
Linear	3	812.605	812.605	17.547452	0.880207
Square	3	3631.609	3631.609	78.42124	1.001512
Interaction	3	4979.369	4979.369	107.52485	0.611539
Error	5	231.545	46.309		
Total	14	8540.793			



(a)



(b)

FIGURE 5: 3D surface and contour plot of response surface curve for COD removal showing interaction between H_2O_2 and pH.

(Fe^{2+} , H_2O_2 concentrations, and pH) was conducted to achieve the highest COD removal for the system.

3.3. Optimization Analysis. Using the method of experimental factorial design and response surface analysis, the optimal conditions for COD removal percentage by photo-Fenton's reagent can be determined. Optimum values of the selected

TABLE 7: Optimum values of the process parameters for maximum efficiency.

Parameter	Optimum value
η (COD reduction rate, %)	91.7
Fe^{2+} (mg/L)	48.4
H_2O_2 (mg/L)	403.9
pH	3.5

variables can be achieved by solving the regression equation (using MATHEMATICA software (V 5.2)). The optimum values of the test variables in-coded were as follows: Fe^{2+} dosage, $x_1 = 48.4$ mg/L, H_2O_2 dosage, $x_2 = 403.9$ mg/L, and pH, $x_3 = 3.5$, while the predicted response was 91.7%. According to the relation between δ_i and x_i , the natural values of the test variables are shown in Table 7. This finding is in agreement with the previous observation of Tony et al. [16] and Kositzki et al. [22] for the treatment of wastewater.

The optimal molar ratio $\text{H}_2\text{O}_2 : \text{Fe}^{2+}$ in the present study is 12 : 1; hence, the hydrogen peroxide is in excess. This optimal molar ratio compares well with the molar ratio of 11 : 1 given by Tang and Huang [23] for 2,4-dichlorophenol degradation.

Increasing H_2O_2 concentration results in the generation of additional reaction intermediates ($\cdot\text{OH}$) radicals which enhances the degradation process. However, at higher peroxide concentrations, the excess hydrogen peroxide can act as an $\cdot\text{OH}$ scavenger, forming $\text{HO}_2\cdot$, which is also a free radical produced in situ from the H_2O_2 but is a less reactive oxidizing agent and therefore has a longer life time than the $\cdot\text{OH}$ and the result is a reduction in the overall reaction rate [24, 25]. Moreover, iron concentrations above the optimal value result in reduced process performance because more species of iron ions are produced rather than the more useful $\cdot\text{OH}$ radicals. This finding is in agreement with the previous observation of Kositzki et al. [22].

The recommended pH value in this investigation of pH 3.5 is well in agreement with the suggested value of 3.0 by Fongsatitkul et al. [26] in the treatment of wastewater from textile industry. These findings clearly suggest that the optimal ratio of the reagent concentration and the pH value vary in accordance with the type of the substance to be treated.

3.4. Verification of the Results. In order to validate the efficiency of the model, three additional experiments using

TABLE 8: Predicted and experimental values for the responses at optimum conditions.

Type of value	COD reduction, %
Predicted	91.7
Experimental	93.4

these optimum operating conditions were conducted. The duplicate experiments yielded an average COD removal percentage of 97%. The predicted COD reduction efficiencies (%) via (2) are jointly shown in Table 8. A good agreement of the data between the experimental and the predicted can be obtained with regression coefficient R^2 value of 0.97 (plotting is shown in Table 2). Thus, it is reasonable to believe that the polynomial model (2) is a reliable model to describe the Fenton reaction behaviour in the wastewater treatment.

4. Conclusion

Results from the present study have demonstrated the effectiveness of the application of photo-Fenton reagent ($\text{Fe}^{2+}/\text{H}_2\text{O}_2$) in the treatment of wastewater delivered from car wash centres. The response surface methodology for optimising such process parameters was applied. This experimental design methodology was shown to be a valuable tool in optimizing the process, which could be satisfied with the minimum number of experiments. The three statistical variables, Fe^{2+} , H_2O_2 concentrations, and pH, showed optimal values, giving maximum percentage COD reduction that reached 97% in treating such car washing water used in the study. The optimal molar ratio of $\text{H}_2\text{O}_2 : \text{Fe}^{2+}$ was found to be 12:1 and the optimum pH was 3.5. These findings are comparable to the literature. This demonstrates the usefulness and effectiveness of the Fenton reagent as an advanced technique for the treatment process.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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