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Degradation of aniline from aqueous solution by Fenton process: modeling and optimization

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ABSTRACT

In the present study, the influences of different parameters on aniline removal by Fenton oxidation using response surface methodology (RSM) were investigated. The central composite design (CCD) was applied in designing the experiments to observe the effects of important operating variables (initial aniline concentration, Fe^{2+} dose, H_2O_2 dose and reaction time). The process performance was evaluated with the analysis of variance. A quadratic model describes the experimental data. The experimental value and predicted value were in good agreement with $R^2 = 0.98$ and Adj-$R^2 = 0.92$, which state the correctness of the model. The optimum aniline removal of 72.57% was achieved at 1000 mg/L of aniline concentration, 1100 mg/L of H_2O_2, 70 mg/L of Fe^{2+} and 40 min of reaction time.

Keywords: Aniline; Fenton process; Modeling; Wastewater; Optimization

1. Introduction

Aniline is a key intermediate commonly used for the manufacture of polyurethanes, dyes, rubber additives, agricultural chemicals, pesticides and pharmaceuticals [1,2]. On the other hand, aniline and its derivatives are hazardous to the human health and the environment [3]. Aniline is highly toxic and may be fatal if swallowed, inhaled or absorbed through the skin [4]. According to blacklist that has been announced by United States Environmental Protection Agency (U.S.EPA) aniline introduced as one of the 129 priority control pollutants [5]. Based on USEPA's recommendation, the maximum allowable aniline concentration in water is 5 mg/L [6]. Therefore to achieve the aforementioned standard different alternatives have been applied to treat aniline from aqueous solution such as physical adsorption [7] electrochemical oxidation [8], ozonation [9], ultrasonic degradation [10] and biodegradation [11,12]. Nevertheless, these methods have the problem of high cost, poor efficiency, complicated maintenance, incomplete degradation and transform aniline into poisonous intermediates which cause adverse effect to water environment [13–15]. Advanced oxidation processes including Fenton, photo-Fenton, ozonation, electrochemical oxidation and photo catalysis have been widely proposed for treating of the various pollutants in waste waters [16]. Among them, Fenton process is preferred mostly because it does not require additional equipment such as ozone systems, ultraviolet lamps and expensive reagents [17]. Therefore, it is essential to develop an efficient method to degrade aniline from water and wastewater. Fenton oxidation because of its high oxidation potential, fast reaction kinetics, and relative lack of selectivity toward most organics is capable of removing the most of pollutants [18–20]. Other advantages of Fenton's process are the simplicity of equipment, mild operating conditions (atmospheric pressure and, room temperature), rapid oxidation rate, relatively low cost, and ease of operation and maintenance [3,21–23]. The Fenton oxidation includes a catalytic reaction between hydrogen peroxide (H_2O_2) and ferrous ion (Fe^{2+}) to produce hydroxyl radicals [19]. Hydroxyl radicals are very strong oxidants

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that rapidly decompose almost any organic pollutants [24]. According to other studies that used Fenton oxidation, this process is affected by H_2O_2 (oxidant), Fe^{2+} (catalyst), and pollutant. In addition other factors such as reaction time, pH and temperature are important parameters [25]. Consequently, optimizing the parameter has a significant effect on selecting process by operator of sewage treatment sectors. Studies for modeling and optimization of Fenton process to aniline degradation are rare and most of researches have been performed at low initial aniline concentration. The main aim of the current study was to investigate the performance of Fenton process on high concentration of aniline degradation in terms of process optimization and modeling by means of a response surface methodology. The worth of the model was monitored by the corresponding analysis of variance Prob > F and the coefficient of determination R^2. Central composite design (CCD) was offered for the optimization of the process with four effective variables (reaction time, initial aniline concentration, Fe^{2+} and H_2O_2 concentration).

2. Material and methods

2.1. Chemicals and artificial wastewater

Aniline ($C_6H_5NH_2$, 98%), obtained from Merck company (Germany), was dissolved with deionized water to the desired concentrations. $FeSO_4{\cdot}7H_2O$ (99%) (Merck company, Germany) was used for the formation of catalyst (Fe^{2+}) in Fenton reaction. H_2O_2 (30% w/w) (Merck company, Germany) was added in the mixture for preceding the reaction. The solution's initial pH was adjusted to pH 3 by sodium hydroxide (NaOH, 1 M) and sulfuric acid (H_2SO_4, 0.5 M).

2.2. Experimental set-up and performance

30 experiments for aniline degradation were carried out at 25°C in batch reactors (250 mL Pyrex glass) with working volume of 100 mL (Fig. 1). The flaxes were filled to capacity of 100 mL by aniline solution with different concentration (200, 600, 1000, 1400 and 1800 mg/L). Then pH value of the solutions was adjusted at 3 using a Jenway 3040 brand pH-meter. Hydrogen peroxide were added to the solutions at arranged concentration of 500, 700, 900, 1100 and 1300 mg/L. Fe^{2+} as catalysts at concentrations of 30, 50, 90, 70 and 190 mg/L were tested in this study. The samples were agitated by a shaker (ISH-OS4LD) at 150 rpm agitation speed in different reaction time (20, 30, 40 50 and 60 min). After accomplishment of each experiment supernatant was analyzed for measuring aniline degradation efficiency.

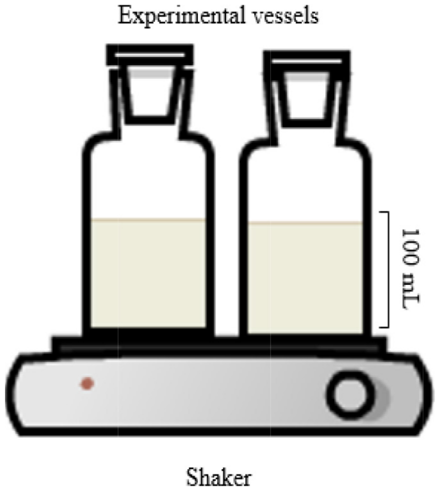

Fig. 1. Experimental set-up.

2.3. Experimental design and data analysis

The Design Expert (version 8.0.0) software was applied for the optimization of the process. Four numerical variables; initial aniline concentration (A), H_2O_2 dose (B), Fe^{2+} dose (C) and reaction time (D) were selected to analyze and optimize the process. The range and levels of the variables in coded and actual units are given in Table 1. The results were analyzed using response surface methodology (RSM) through CCD. In order to analyze the process, aniline removal efficiency as the process response was studied. The CCD is the standard RSM, which allows the use of second-degree polynomial in the estimation of relationships between the independent and dependent variables. The behavior of the obtained data in the CCD model can be described through Eq. (1) [26–28].

$$Y = \beta_{\circ} + \sum\nolimits_{i=1}^{k} \beta_i x_i + \sum\nolimits_{i=1}^{k} \beta_{ii} x_i^2 + \sum\nolimits_{i<j}^{k} \sum \beta_{ij} x_i x_j + e \tag{1}$$

where i represents linear coefficient; j stands for the quadratic coefficient; β is regression coefficient; x represents independent variables; k is the number of studied and optimized factors in the experiment; and e is the random error. The data obtained were analyzed using analysis of variance (ANOVA).

2.4. Analytical methods

The samples were analyzed after centrifuging at 5000 rpm for 10 min and filtering through a 0.22 mm membrane filter. The aniline removal efficiency (E) was determined by the following equations as given below:

$$E(\%) = \left(\frac{(C_0 - C_1)}{C_0}\right) \times 100 \tag{2}$$

Table 1
Experimental range and levels of the independent variables

Variable	Unit	Range and levels				
		–2	–1	0	1	2
A: Initial aniline concentration (An)	(mg/L)	200	600	1000	1400	1800
B: H_2O_2 concentration	(mg/L)	500	700	900	1100	1300
C: Fe^{2+} concentration	(mg/L)	30	50	90	70	190
D: Reaction time (t)	(min)	20	30	40	50	60

where C_0 is the initial aniline concentration and C_1 is the final aniline concentration, respectively. The concentration of aniline in solution was detected by spectrophotometer (Jenwey 6053) by measuring the adsorption intensity at 282 nm (maximum absorption wavelength).

3. Results and discussion

3.1. Statistical analysis and mathematical modeling

The CCD with four factors in five levels as well as the results of aniline removal efficiency for Fenton process is presented in Table 2. The data of Fenton process were fitted to a quadratic model. Adequacy of the model was checked by the coefficients of regression (R-Squared), adjusted R-Squared, coefficient of variation (CV), F- value, p-value and Lack of Fit F-value. The quadratic model results for aniline removal are summarized in Table 3. R^2 and the *Adj-R^2* are significant parameters in the model. They show adequate variation of quadratic model to the experimental data. In this research, R^2 and *Adj-R^2* values were 0.96 and 0.92, respectively. High values of R^2, and *Adj-R^2* (close to one) indicate the model validation [29]. The model F-value of 25.88 suggests that the model is significant. Adeq precision measures the signal to noise ratio that a ratio greater than 4 is desirable [30,31]. The adeq precision ratio in this study was 16.53, which indicates an adequate signal. Standard deviation and mean were found to be 3.81 and 53.53, respectively. The CV represents error between experimental and predicted data. It cannot be more than ten for a proper model. In the current research, the CV value was 7.12. P-value < 0.05 shows significance of the model terms while P-value > 0.05 expresses that model terms are not significant [32]. In this study A, B, C, D and AC were significant model terms. Based on the polynomial quadratic model [Eq. (3)], the predicted response (Y) for aniline removal was acquired in terms of coded factors as below:

$$Y = +65.80 - 4.32A + 6.13B - 4.54C - 3.40D + 0.12AB + 3.21AC + 1.17AD - 1.62BC + 1.04BD + 1.26CD - 11.72A^2 + 15.28B^2 - 10.96C^2 - 14.92D^2 \quad (3)$$

Table 2
Experimental conditions and results

Run no.	Variables				Response
	A: An (mg/L)	B: H_2O_2 (mg/L)	C: Fe^{2+} (mg/L)	D: t (min)	Removal (%)
1	1400	900	70	40	63.5
2	200	1300	30	60	60.5
3	1000	1100	70	40	73
4	1800	1300	110	60	40
5	1000	900	90	40	55.8
6	200	500	110	60	30
7	1000	900	50	40	71
8	1800	1300	30	20	51
9	1800	500	30	20	35
10	200	1300	110	20	42.7
11	1000	900	70	40	67
12	1800	500	30	60	31
13	1000	900	70	40	66
14	1000	900	70	50	61
15	600	900	70	40	63
16	1800	1300	110	20	47
17	200	1300	30	20	67.2
18	1000	700	70	40	67
19	200	1300	110	60	45.4
20	1000	900	70	40	63
21	1800	1300	30	60	43
22	1800	500	110	60	33
23	1000	900	70	40	65
24	1000	900	70	40	63
25	1000	900	70	40	68
26	1800	500	110	20	32
27	200	500	30	60	37.5
28	200	500	110	20	44
29	200	500	30	20	56.2
30	1000	900	70	30	63.9

3.2. Effect of initial aniline concentration

The effects of independent variables on aniline removal were determined by three dimensional response surface and two dimensional contour plots graph. The initial concentrations of the aniline are essential to evaluate the extent of chemical degradation efficiency. According to Figs. 2a and 3a aniline decomposition efficiency increased when initial aniline concentration increased from 200 mg/L to 1000 mg/L, however at the aniline concentration values higher than 1000 mg/L the removal efficiency decreased. The results of Azizi et al. [33] and Anotaia et al. [23] studies confirm this trend. This is due to that when the initial concentration of aniline is increased but the generation of $^{\bullet}OH$ is not increased correspondingly, so a relative lower $^{\bullet}OH$ concentration resulted in the decrease of degradation efficiency of aniline [33]. From the Fig. 2a 67% aniline removal was observed at initial aniline concentration of 1000 mg/L and reaction time of 40 min when the Fe^{2+} and H_2O_2 dose were

Table 3
Quadratic model ANOVA results responses

Variable	Aniline removal
Standard deviation	3.81
(CV %)	7.12
Mean	53.53
R-Squared	0.96
Adj R- Squared	0.92
Adequate precision	16.53
F-value	25.88
P-value	< 0.0001
Probability for lack of fit	0.053

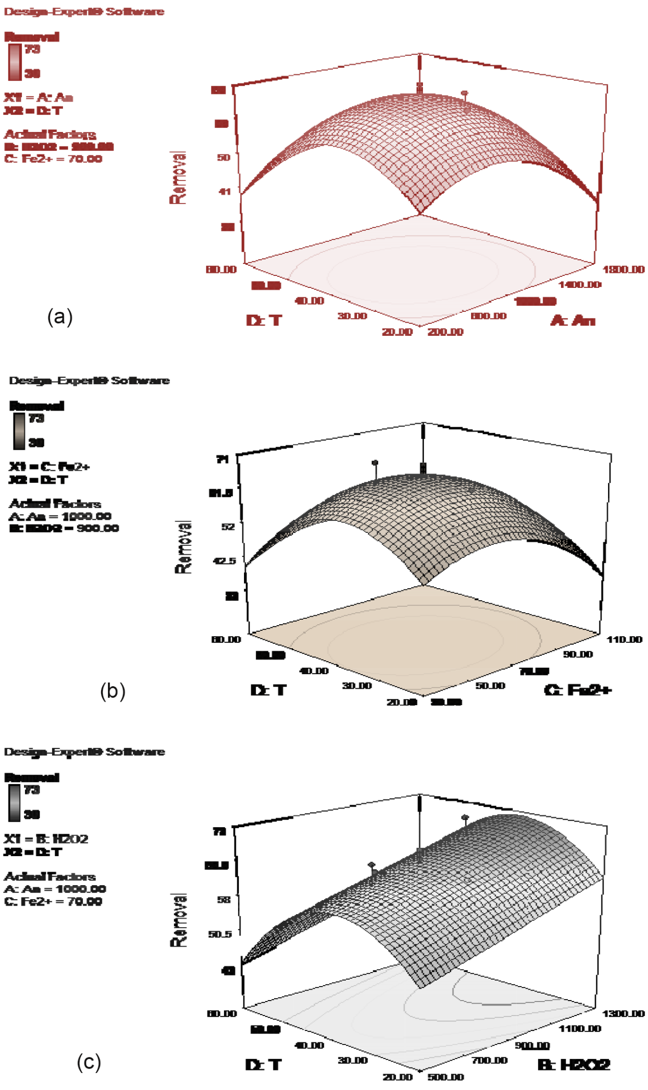

Fig. 2. Three-dimensional response surface of aniline removal in term of in depended variables: (a) initial aniline and reaction time, (b) Fe^{2+} and reaction time and (c) H_2O_2 and reaction time.

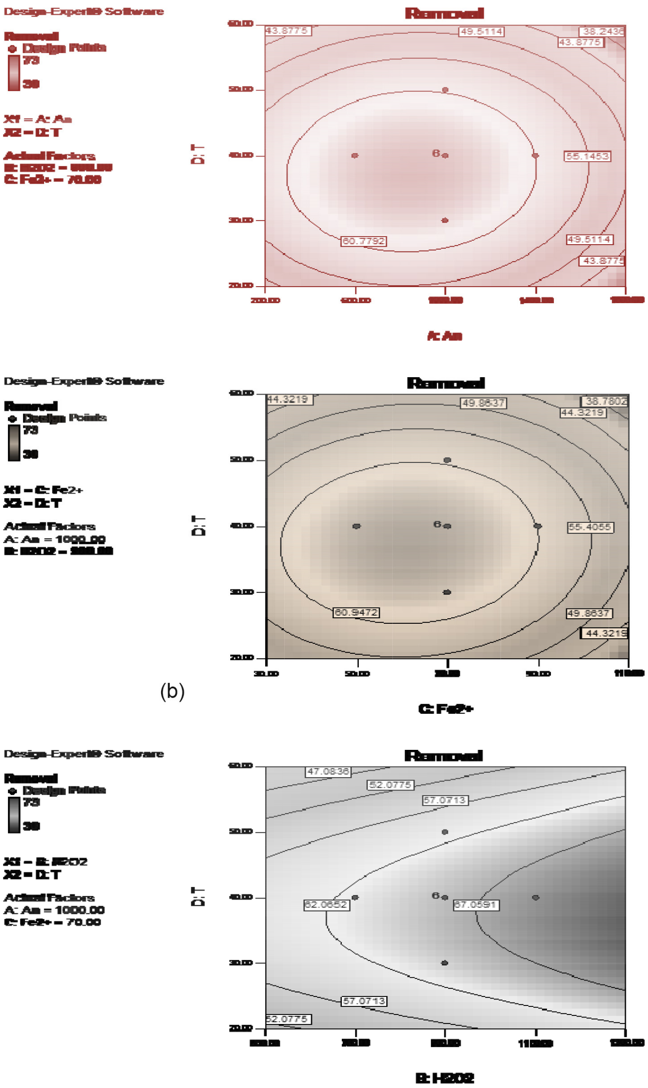

Fig. 3. Two-dimensional contour plots of aniline removal in term of in depended variables: (a) initial aniline and reaction time, (b) Fe^{2+} and reaction time and (c) H_2O_2 and reaction time.

70 mg/L and 1000 mg/L respectively. In several studies have been observed that Fenton based treatment processes are limited to effluent containing high organic material. The reason associated with this is energy consumption and for the effluent treatment process, it is proportional to the amount of organics and to the number of electrons involved in the conversion of organics into CO_2 [34–36].

3.3. Effect of reaction time

As shown in Figs. 2 and 3a–c aniline removal was significantly influenced by the reaction time as when reaction time increased from 20 to 40 min, degradation of aniline remarkably increased, the optimal reaction time was observed at 40 min with aniline removal efficiency of 73% (Fig. 2c). This can be explained that Fenton's reaction could be accelerated by raising the time reaction, which improved the generation rate of hydroxyl radicals and, therefore, enhanced the degradation of the organic matter [37,38]. Mousavi et al. [39] investigated the effect of reaction time on degradation of Rhodamine B by Fenton oxidation. Confirmed that the reaction time had a significant effect on the process, as the reaction time increased from 20 to 80 min the removal efficiency increased from 35% to 55% in which other parameter where constant. The results of current study indicated that a further increase of reaction time (> 40 min) did not show more effects on the process efficiency. Several studies have reported that during Fenton oxidation of organic matter, increasing reaction time more than a specific limit cannot lead to improve degradation efficiency [40–42].

3.4. Effect of Fe^{2+} concentration

According to Figs. 2b and 3b the concentration of Fe^{2+} and the reaction time are important parameters for removing aniline and aniline removal increased with increasing both Fe^{2+} dose and reaction time. The maximum aniline removal was observed 71% at Fe^{2+} dose of 70 mg/L, while it decreased at Fe^{2+} concentrations more than 70 mg/L. In the excess amounts of Fe^{2+}, it can react with hydroxyl radical. Then, the hydroxyl radical in the system will decrease and the organic degradation will also reduce [43]. It has been confirmed that the H_2O_2/Fe^{2+} ratio is an important parameter in Fenton oxidation due to its direct effect on the quantum yield of °OH generation [44]. The results of Masomboon et al. [22]'s study indicated that 62% of 2, 6-dimethyl aniline removal was achieved at 84 mg/L Fe^{2+}. Another study was performed by Azami et al. [45] in which Fenton process was employed to azo dye, methyl red removal from a wastewater. The results indicated that by increasing Fe^{2+}, an increasing trend was observed in the dye removal efficiency. Result confirms dye removal dropped by increasing concentration of Fe^{2+} more than specific limit that the maximum dye removal was observed at Fe^{2+}concentration of 0.3 mM. The results of studies that have been carried out by Jafari et al. [42] and Saini et al. [46] are consistent with the results of this study.

3.5. Effect of H_2O_2 concentration

Figs. 2c and 3c illustrate the removal efficiency of aniline depend on H_2O_2 dosage. Results confirm that by increasing the concentration of H_2O_2 concentration, the aniline removal increased because of more available hydroxyl radicals (•OH) [47]. The maximum aniline removal was 73% when the concentration H_2O_2 was 100 mg/L . The higher the concentration of hydrogen peroxide, the more aniline degradation, since there are more available •OH to react with the organics in the solution [22]. For the Fenton process, the oxidation of aniline was not inhibited by increasing the concentration of hydrogen peroxide up to 1100 mg/L. The same results were also found in other research [48,49]. The reason might be that the amount of hydrogen peroxide added in this study has not yet reached the degree of inhibition when using Fenton process [50]. Babuponnusami et al. [36] represented Fenton oxidation of phenol depending on H_2O_2 concentration, that with increasing of H_2O_2 degradation efficiency of phenol improved and the maximum phenol removal was 57% at 800 mg/L of H_2O_2. Previous researches have reported that stepwise addition of H_2O_2 is more effective for degradation of organic matter [42,51].

3.6. Optimization

Graphical optimization produces an overlay plot of the contour graphs to display the area of feasible response values in the factor space. The graphical optimization results allow visual inspection to choose the optimum operating conditions. Fig. 4 shows the graphical optimization, which display the area of feasible response values (shaded portion) in the factors space. The optimum region was identified based on aniline removal, with criteria >60%. From Fig. 4, the optimum region (72.57% removal) is covered by reaction time 40 min, Fe^{2+} 70 mg/L, initial aniline 1000 mg/L and H_2O_2 1100 mg/L.

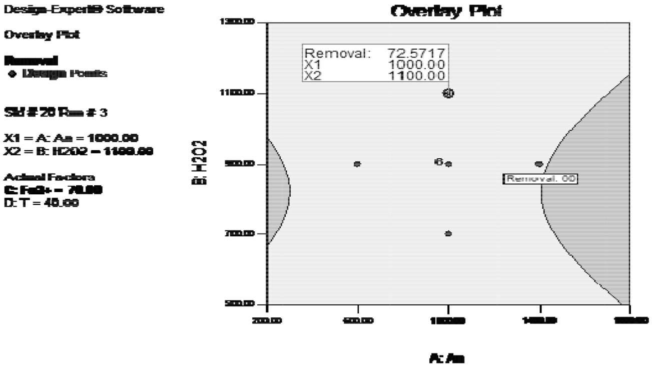

Fig. 4. Overlay plots for the optimal region; aniline removal as a response.

4. Conclusion

Fenton process was successfully applied to aniline removal from aqueous solution with high concentration. CCD of RSM was used for modeling and optimization of the process and better understand the effect of independent variables such as initial aniline concentration, H_2O_2 and Fe^{2+} concentration and reaction time on the performance of the process. The results indicated that independent parameters play an important role on Fenton oxidation of aniline. Through RSM analysis method, the predicted optimum condition for Fenton oxidation to aniline degradation was determined as 1000 mg/L of initial aniline concentration, 70 mg/L of Fe^{2+} concentration, 1100 mg/L of H_2O_2 concentration, and 40 min of reaction time, with 72.57% of aniline removal efficiency at pH of 3.

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