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Optimization of nitrobenzene wastewater treatment with O_3/H_2O_2 in a rotating packed bed using response surface methodology

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ABSTRACT

Effects on the removal efficiency of nitrobenzene treated by O_3/H_2O_2 in a rotating packed bed (RPB) were optimized with response surface methodology (RSM). Interaction effects between influence of H_2O_2 concentration, gas-phase ozone concentration, liquid flow rate, and high-gravity factor on the removal efficiency of nitrobenzene wastewater treatment with O_3/H_2O_2 in a RPB were investigated. The results indicate that the influence priority on nitrobenzene removal is high-gravity factor, gas-phase ozone concentration, H_2O_2 concentration, and liquid flow rate. Significant interaction effects of H_2O_2 concentration and gas-phase ozone concentration, gas-phase ozone concentration, and high-gravity factor were observed. The optimum treatment conditions after optimizing were H_2O_2 concentration 5.7 mmol L^{-1}, gas-phase ozone concentration 50 mg L^{-1}, liquid flow rate 125 L h^{-1}, and high-gravity factor 100. Under the optimal reaction conditions, the actual removal efficiency of nitrobenzene could reach 76.1% fast in a short-treatment time of 10 min. And the theoretical value was 78.2%. The deviation between the experiment test result and the predicted value of RSM-fitting equation was 2.68%, which indicate that the RSM-fitting equation could be used to predict the removal efficiency of nitrobenzene treated by O_3/H_2O_2 in a RPB and optimize the treatment conditions.

Keywords: Nitrobenzene; Oxidation; Degradation; Response surface methodology (RSM); High gravity

1. Introduction

As an important organic industrial intermediate, nitrobenzene (NB) is widely used in petrifaction, fuel, material, medicine and other industries, and millions of tons of nitrobenzene (NB)-containing wastewater are discharged into the environment all over the world every year [1,2]. Owing to its high toxicity to human body and biorefractory property, NB has been listed as a precedence-controlled pollutant in many countries [3,4]. Treatment of NB-containing wastewater before discharged attracts more and more concerns nowadays [5].

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Among the great deal of treatment methods, ozonation seems to be a promising process because of its strong oxidability (redox potential 2.07 V), nonselectivity and will not generate secondary pollution [6]. With the catalysis of H_2O_2, hydroxyl radical ·OH with much stronger oxidability (redox potential 2.8 V) could be generated in O_3/H_2O_2 system [7–9]. However, as the mass transfer of ozone into water is limited by the resistance of liquid phase, interphase mass transfer of ozone cannot be improved by conventional bubble apparatus significantly [10]. Therefore, field-scale application of ozonation has been greatly restrained. Recent decades, rotating packed bed (RPB) has been proved to be a efficient device in the intensification of mass transfer and reaction process by rotating to simulate an high-gravity field [11,12]. In RPB, due to the greatly gas–liquid contact areas improving and the rapidly interface updating formed by the shearing of the high speed rotator, gas–liquid phase resistance can be decreased dramatically, as a result, the interphase mass transfer of ozone can be significantly intensified [13]. With the process intensification of RPB in the interphase mass transfer of ozone and catalysis of H_2O_2, advanced oxidation method of O_3/H_2O_2 system is a promising alternative method for NB-containing wastewater treatment [14].

In most of the published references, methods for assessment and optimization of the treatment method of NB-containing wastewater are mainly single-factor test and orthogonal test [10,15]. Both of these two methods take single-factor test as the basis to evaluate the effect of each variable one by one, and only one factor can be optimized and examined each time. Unintentionally, interactions between different factors are ignored. Response surface methodology (RSM) is a comprehensive experiment design and an optimized method for mathematical modeling, which has been widely applied in many fields [16,17]. RSM can analyze all the numerical values of experimental points under continuity condition to regress and fit the function relationship between factors and results in the same time. Then, the interactions between different factors can be taken into account for the assessment and optimization of the treatment process [18,19].

In this paper, combination RPB-O_3/H_2O_2 process was established to degrade NB-containing wastewater. The objectives of this study were as follows: (1) to investigate the interactions between different factors on treatment efficiency of RPB-O_3/H_2O_2 process on NB degradation by RSM; (2) to optimize the treatment conditions of RPB-O_3/H_2O_2 process on NB degradation by RSM; and (3) to establish an RSM mathematical model for the assessment of RPB-O_3/H_2O_2 process on NB degradation.

2. Materials and methods

2.1. Materials

NB(AR, Tianjin Kemiou Chemical Reagent Co. Ltd, China), hydrogen peroxide H_2O_2 30% (AR, Tianjin Tianli Chemical Reagents Ltd, China), potassium dichromate (AR, Shanghai Pujiang Chemical Factory, China), ammonium ferrous sulfate (AR, Chengdu Chemical Reagents Factory, China), potassium iodide (AR, Tianjin Fuchen Chemical Reagents Factory, China), sodium thiosulfate (AR, Tianjin Beichen District Founder Chemical Agent Plant, China), oxygen and deionized water.

Simulated NB-containing wastewater was prepared by dissolving certain amount NB into deionized (DI) water. The initial pH was 7.5 and the initial NB mass concentration was 200 mg/L.

2.2. Experimental methods

Experimental RPB device was self-made cross-flow RPB. Inner diameter, external diameter and axial height of the RPB rotator were 40, 75, and 75 mm, respectively. The packing was stainless-steel wire

Fig. 1. Structure of the experiment RPB.

gauze packing. The structure of the setup shows in Fig. 1.

Fig. 2 shows the diagram of the degradation of NB-containing wastewater in RPB-O_3/H_2O_2 process. Ozone gas with certain gas-phase concentration was generated by Oxygen 1 passing through the ozonator 2, then flowed from the bottom of RPB 4 through gas flowmeter 3, and then went up through wire gauze packing. NB-containing wastewater was pumped from liquid storage tank 8 to the RPB center; then went through wire gauze packing from the inside to the outside in radial direction. NB-containing wastewater and ozone crosscontacted in the RPB to complete mass transfer and oxidation reaction. The treated wastewater then flowed into liquid storage tank 8 for circulation. Unreacted ozone gas went to tail gas treating tank 9. Hydrogen peroxide (H_2O_2) was added ahead into the wastewater in a desired concentration.

Optimizing experiment by RSM was conducted basing on the original data obtained from the single-factor experiment. According to our previous work [20], gas flow rate and initial pH were fixed at 75 L/h and 10.5, respectively. Main factors optimized included H_2O_2 concentration C_A, gas-phase ozone concentration C_B, liquid flow rate L, and high-gravity factor β [21]. Four-factor and three-level experiment was designed using Box–Benhnken design (BBD) method [22], the experimental factors and level were shown in Table 1.

2.3. Analytical methods

NB content in wastewater was detected by HPLC (Dionex, Ultimate 3000) with a C_{18} reversed-phase column (250 mm × 4.6 mm, 5 μm). The UV-detected wavelength was 262 nm, mobile phase was methanol/water (70/30), flow rate was 0.9 ml/min, column temperature was 20℃ and sample volume was 20 μL. The removal efficiency of NB was defined as the following equation:

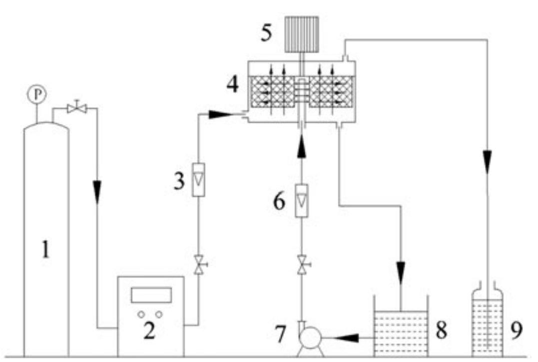

Fig. 2. Experiment process flow diagram.
Notes: (1) Oxygen bottle, (2) Ozone generator, (3) Gas flowmeter, (4) Rotating packed bed, (5) Electromotor, (6) Liquid flowmeter, (7) Liquid pump, (8) Tank, and (9)Tail gas treating unit).

Table 1
Factors and levels of RSM

Factor		Range and level		
		−1	0	1
A	C_A (mmol/L)	2.9	4.9	6.9
B	C_B (mg/L)	30	40	50
C	L (L/h)	100	120	140
D	β	60	80	100

$$\eta = \frac{C_0 - C_t}{C_0} \times 100\% \tag{1}$$

where C_0 and C_t were NB concentration in the wastewater after and before treatment.

COD for the wastewater was measured according to water quality determination of the chemical oxygen demand dichromate method (GB/T 11914-89, China). Gas-phase ozone concentration was measured by ozone detector (Honghai, GT901).

3. Results and discussion

3.1. Establishment of quadratic regression model of RSM

According to BBD method, RSM experiment of 29 points was designed. Zero experiment was repeated five times to estimate the error. The experiment results are shown in Table 2.

A module of Ansys 15.0 as design exploration software was applied to analyze the experiment data in Table 2. To establish the quadratic regression equation of RSM, a second-order empirical model as following was used for mathematical equation fitting:

$$\begin{aligned} Y = {} & 63.22 + 1.70A + 7.84B + 1.47C + 7.99D \\ & + 1.70AB + 0.100AC + 0.25AD - 1.18BC \\ & - 1.75BD - 0.025CD - 4.34A^2 - 0.38B^2 \\ & - 3.12C^2 + 0.47D^2 \end{aligned} \tag{2}$$

After eliminating the nonsignificant terms, quadratic regression equation of RSM was simplified as the following equation:

$$\begin{aligned} Y = {} & 63.27 + 1.70A + 7.84B + 1.47C + 7.99D \\ & + 1.70AB - 1.75BD - 4.36A^2 - 3.13C^2 \end{aligned} \tag{3}$$

Table 2
Results of the RSM experiment

	Coded variable				
Run	*A*	*B*	*C*	*D*	NB removal efficiency *Y* (%)
1	0	1	1	0	68.3
2	1	0	0	−1	54.1
3	−1	0	0	1	63.8
4	1	1	0	0	70.6
5	0	0	−1	−1	51.2
6	0	0	0	0	64.7
7	0	0	1	−1	53.7
8	0	−1	0	1	65.4
9	0	0	0	0	62.4
10	0	1	0	1	76.4
11	0	−1	0	−1	45.4
12	0	−1	−1	0	48.5
13	0	1	−1	0	67.9
14	1	0	0	1	68.2
15	−1	0	−1	0	52.1
16	0	0	−1	1	69.1
17	0	0	0	0	61.7
18	−1	0	0	−1	50.7
19	−1	−1	0	0	51.4
20	−1	0	1	0	55.5
21	−1	1	0	0	63.5
22	1	0	−1	0	54.5
23	0	0	1	1	71.5
24	0	−1	1	0	53.6
25	0	0	0	0	63.2
26	1	0	1	0	58.3
27	1	−1	0	0	51.7
28	0	0	0	0	64.1
29	0	1	0	−1	63.4

where Y was the response value (NB removal efficiency), A was H_2O_2 concentration, B was gas-phase ozone concentration, C was liquid flow rate and D was high-gravity factor.

3.2. Variance analysis and significance test

Variance analysis and significance testing was made for regression the RSM equation. The results after manually optimizing and eliminating nonsignificant terms are shown in Table 3. It can be seen from Table 3 that H_2O_2 concentration, gas-phase ozone concentration, liquid flow rate, and high-gravity factor exhibited significant influence on NB removal. As known from F-value inspection that the contribution sequence of the influence factors is high-gravity factor > gas-phase ozone concentration > H_2O_2 concentration > liquid flow rate. The interaction is significant between H_2O_2 concentration and gas-phase ozone concentration, as well as gas-phase ozone concentration and high-gravity factor. The selected second-order model is highly significant ($p < 0.0001$) within the rest range. Fig. 3 shows the comparison of predicted value and the actual value for the regression model. The predicted value and the actual value of NB removal in the mathematical model have excellent relevance with a relevance coefficient of $R^2 = 0.9790$, which means that over 97.90% data variability in the model can be explained with the model. The lack of fit items for the model are not significant ($p = 0.4243 > 0.05$); and the coefficient of variable is small (C.V. = 2.29 < 10%).

3.3. Effects of factors in the model on response value

In order to investigate the effects of four factors on NB removal, 3D diagram and contour map of response surface were drawn based on quadratic regression model, as shown in Figs. 4–9. Interactions between different factors of the model on NB removal can be seen visually.

As shown in Fig. 4–6, H_2O_2 concentration has significant effect on NB removal. In the presence of H_2O_2, ozone can be catalyzed to generate hydroxyl radical ·OH to enhance the ozonation efficiency. Table 3 and Fig. 4 indicate that interaction between gas-phase ozone concentration and high-gravity factor is significant. When gas-phase ozone concentration was 40 mg/L and H_2O_2 concentration increased to 6.9 mmol/L, NB removal efficiency increased firstly and then decreased. It is known from oxidation mechanism that hydroxyl radical ·OH plays a major role in oxidative degradation of organic matters [23]. As presented in formulas (4)–(5), the decomposed intermediate product of H_2O_2,HO_2^- can react with ozone to promote ·OH generation thus to improve NB removal. However, when H_2O_2 concentration was higher than a certain value, excess H_2O_2 would react with ·OH as Formula (6), therefore, ·OH concentration decreased and then NB removal efficiency decreased. The appropriate H_2O_2 concentration increased along with the increase in gas-phase ozone concentration and high-gravity factor. The reason may be that the increase in gas-phase ozone concentration and high-gravity factor made ozone water concentration higher in per unit volume, resulted more H_2O_2 to react with ozone to generate hydroxyl radical ·OH; therefore, the optimum H_2O_2 concentration increased.

$$H_2O_2 \leftrightarrow H^+ + HO_2^- \quad (4)$$

$$O_3 + HO_2^- \rightarrow {}^{\cdot}OH + O_2 + O_2^- \quad (5)$$

$$H_2O_2 + {}^{\cdot}OH \rightarrow H_2O + {}^{\cdot}HO_2 \quad (6)$$

Table 3
The variance analysis

Source	SS	DF	MS	F	p-value	Significance
Model	1,764.83	8	126.68	60.77	<0.0001	**
A	34.68	1	34.68	16.64	0.0011	**
B	737.90	1	737.90	353.98	<0.0001	**
C	25.81	1	25.81	12.38	0.0034	**
D	766.40	1	766.40	367.66	<0.0001	**
AB	11.56	1	11.56	5.55	0.0336	*
BD	12.25	1	12.25	5.88	0.0295	*
A^2	122.36	1	122.36	58.70	<0.0001	**
C^2	63.07	1	63.07	30.26	<0.0001	**
Residual	37.91	20	1.90			
Lack of fit	31.96	16	2.00	1.34	0.4243	Insignificant
Pure error	5.95	4	1.49			
Cor total	1,802.74	28				

Notes: It is not significant when $p \leq 0.05$, indicated with *; it is highly significant when $p \leq 0.01$, indicated with ** and it is no significant when $p > 0.05$; $R^2 = 0.9790$; $R^2_{\text{adi}} = 0.9706$; $R^2_{\text{pred}} = 0.9577$; Adeq precision = 41.287.

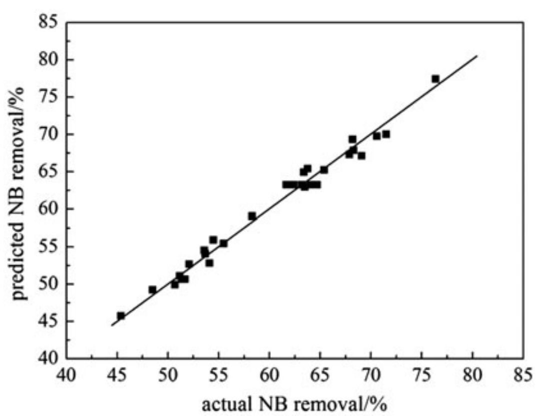

Fig. 3. Comparison of predicted value and actual value for NB removal.

Figs. 4, 7 and 8 show NB removal efficiency varied with the increase of gas-phase ozone concentration. It can be seen that gas-phase ozone concentration has significant influence on the treatment process, this mainly because the mass transfer driving force for ozone transfer from gas phase into liquid phase increased with the increase of gas-phase ozone concentration. However, ozone utilization rate decreased with the gas-phase ozone concentration increasing. With the optimum situation of other treatment conditions, NB removal within 10-min treatment increased from 62.4% to 67.8% when gas-phase concentration increased from 40 to 50 mg/L. NB removal efficiency increased by 5.4%, but the utilization rate of ozone decreased by almost 10%. Therefore, take into account of the economic perspective, higher gas-phase ozone concentration is not economic.

The smaller gas–liquid ratio represents stronger treatment capability and lower cost in wastewater treatment process. The effect of liquid flow rate on NB removal efficiency was investigated under the condition of lower gas flow rate 75 L/h combined with the structural feature of the RPB. It can be seen from Figs. 4, 7 and 9 that NB removal efficiency presented tendency of increased firstly and then decreased with the increase in liquid flow rate. Degree of wetting of the packing surface increased with the increase in liquid flow rate, which amplified the effective gas–liquid contact area, then the mass transfer of ozone enhanced. The continuous increasing of liquid flow rate resulted in decreasing of ozone concentration in per unit solution volume and shorter the gas–liquid contact time [24], which were negative for ozone mass transfer thus NB removal efficiency decreased.

As shown in Figs. 6, 8 and 9, NB removal efficiency increased with the increase of high-gravity factor. With an increasing high-gravity factor, the shear force imposed by the rotating packing on the wastewater also increased to reduce the liquid film thickness continuously, resulting in an increased gas/liquid contact area to intensify ozone mass transfer rate from gas phase to liquid phase. It can be seen from Table 3 and Fig. 8 that the interaction is significant between gas-phase ozone concentration and high-gravity factor. When the high-gravity factor was fixed, the updating speed of gas–liquid interface then kept unchanged, the improvement of ozone

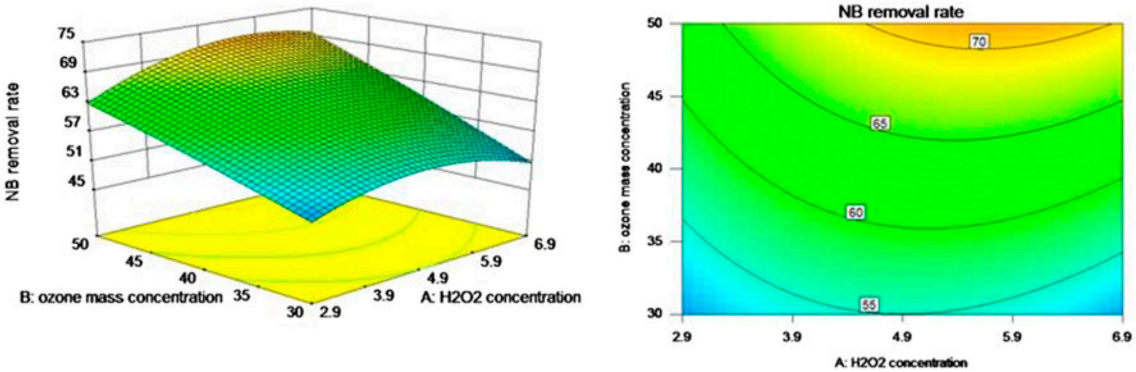

Fig. 4. Effect of H_2O_2 concentration *A* and ozone mass concentration *B* on nitrobenzene removal efficiency (liquid flow rate 120 L/h, high-gravity factor 80).

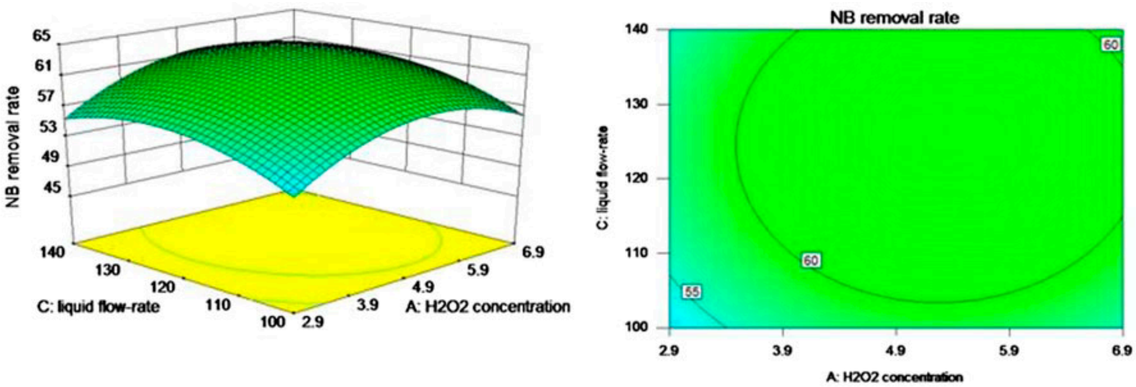

Fig. 5. Effect of H_2O_2 concentration *A* and liquid flow rate *C* on nitrobenzene removal efficiency (ozone mass concentration 40 mg/L, high-gravity factor 80).

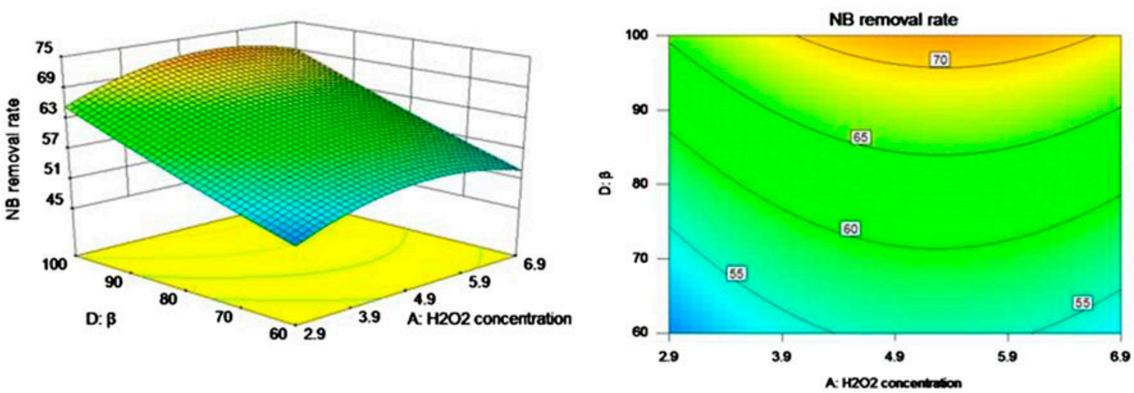

Fig. 6. Effect of H_2O_2 concentration *A* and high-gravity factor *β D* on nitrobenzene removal efficiency (ozone mass concentration 40 mg/L, liquid flow rate 120 L/h).

concentration in gas phase can amplify the mass transfer driving force and speed up ozone dissolution. As known from surface renewal theory that when the ozone concentration in gas phase was fixed, the appropriate improvement of high-gravity factor can speed up gas–liquid-phase interface updating rate and improve the ozone dissolution rate, and then, the NB degradation improved [25].

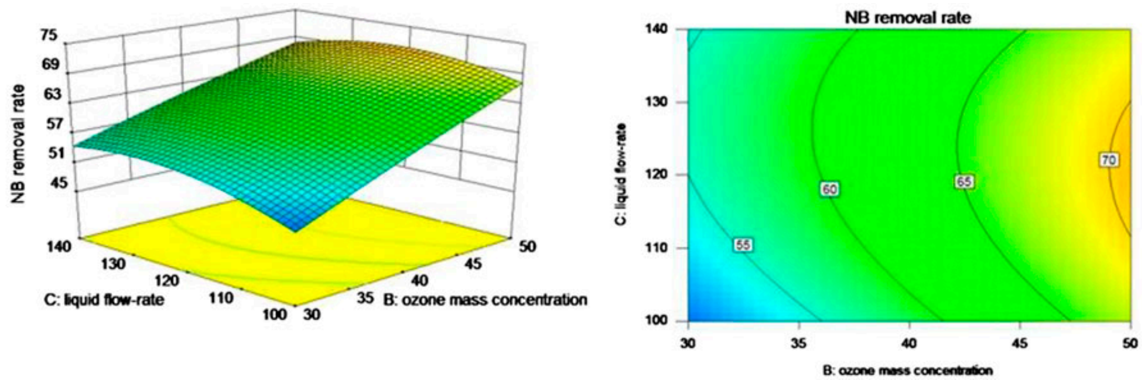

Fig. 7. Effect of ozone mass concentration *B* and liquid flow rate *C* on nitrobenzene removal efficiency (H_2O_2 concentration 4.9 mmol/L, high-gravity factor 80).

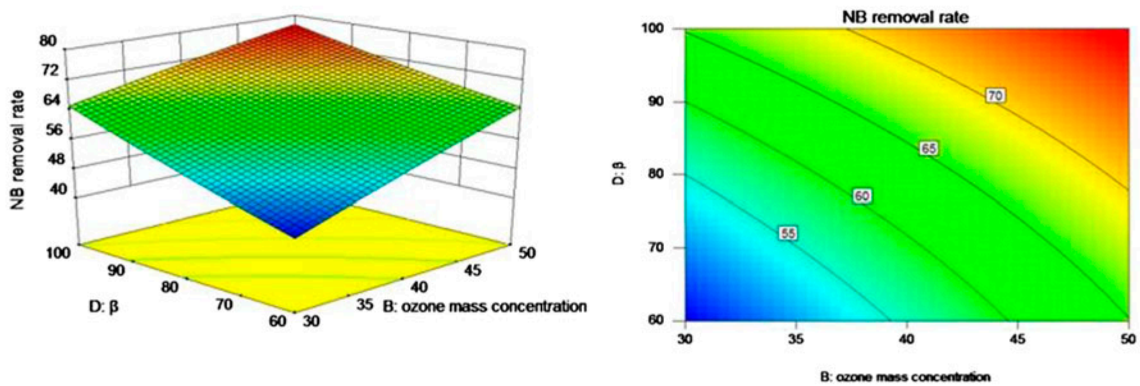

Fig. 8. Effect of ozone mass concentration *B* and high-gravity factor *β D* on nitrobenzene removal efficiency (H_2O_2 concentration 4.9 mmol/L, liquid flowrate 120 L/h).

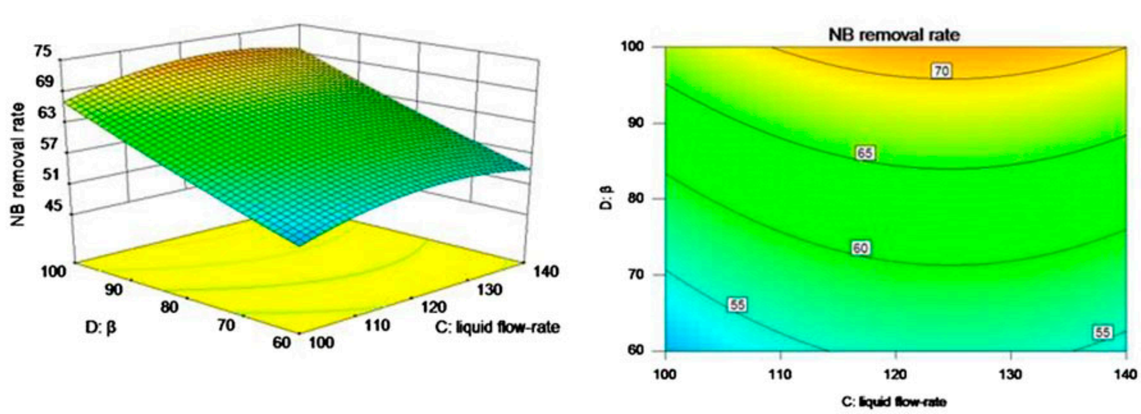

Fig. 9. Effect of liquid flowrate *C* and high-gravity factor *β D* on nitrobenzene removal efficiency (H_2O_2 concentration 4.9 mmol/L, ozone mass concentration 40 mg/L).

3.4. Validation of the optimum treatment conditions

Optimum treatment conditions obtained with the help of Ansys 15.0 were presented as the following: H_2O_2 concentration of 5.68 mmol/L, gas-phase ozone concentration of 50 mg/L, liquid flow of 124.72 L/h, and high-gravity factor of 100. Then, the theoretical NB removal efficiency was 78.2%. Considering the feasibility of the experiment, the treatment conditions were optimized as follows: H_2O_2 concentration of 5.7 mmol/L, gas-phase ozone concentration of 50 mg/L, liquid flow of 125 L/h, and the high-gravity factor of 100.

The model was verified under the above optimum conditions, the actually measured NB removal efficiency was 76.1% with 2.68% relative deviation from the predicted value. Therefore, the mathematical fitting equation of RPB-O_3/H_2O_2 process received from RSM for optimizing the treatment conditions of NB removal efficiency is accurate and reliable.

4. Conclusion

According to variance analysis, mathematical model of RPB-O_3/H_2O_2 process on NB degradation established by RSM was appropriate. The influence sequence for experiment factors was high-gravity factor > gas-phase ozone concentration > H_2O_2 concentration > liquid flow rate. The interaction was significant between H_2O_2 concentration and gas-phase ozone concentration, as well as gas-phase ozone concentration and high-gravity factor. Optimum treatment conditions received by RSM analysis were: H_2O_2 concentration of 5.7 mmol/L, gas-phase ozone concentration of 50 mg/L, liquid flow of 125 L/h, and high-gravity factor of 100. The theoretical NB removal efficiency and the experimental removal efficiency at 10 min treatment were 78.2% and 76.1%, respectively. Relative deviation of test result from predicted value was verified to be 2.68% indicates that the mathematical model obtained from RSM is feasible for the condition optimization of RPB-O_3/H_2O_2 process on NB degradation.

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