



Optimizing ammonia volatilization by air stripping from aquatic solutions using response surface methodology (RSM)

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ABSTRACT

In the present work, ammonia removal by air stripping process is optimized using response surface methodology based on central composite design. The removal of ammonia was studied based on the obtained model. To determine the effect of the operating parameter on the rate of reaction, the kinetic was investigated. The most important parameters which can affect ammonia stripping such as ammonia concentration, pH, and temperature were optimized. The optimum ammonia concentration, pH, and temperature of air stripping process were obtained as 1,440 mg/L, 10.7, and 36 °C, respectively. Under optimal conditions, effects of different flow rates were evaluated and the best volatilization was obtained using 3 L/min air flow rate. Maximum removal percentage was acquired as 84.11% at the end of 12 h of operating time. Analysis of Variance exhibited a reasonable correlation coefficient between the predicted and experimental values ($R^2 = 0.97$). Experimental results showed that the first-order kinetics with $R^2 = 0.99$ more fitted the removal data. According to the obtained results, correlation coefficient, volatilization rate, and half-life were obtained as 0.987, 0.1395/h, and 4.97 h, respectively.

Keywords: Ammonia; Air stripping; Kinetic; Modeling; RSM

1. Introduction

High concentrations of ammonia have been reported in various industrial wastewaters such as coke plant, tannery, textile, landfill leachate, and fertilizers [1,2]. Although ammonia nitrogen is a very essential element for living organisms, it can contribute to the accelerated eutrophication of lakes and rivers, dissolved oxygen depletion, and fish toxicity in receiving water bodies [2,3]. The commonly used

processes for nitrogen removal are biological process, chemical treatment, ion exchange, and ammonia volatilization by air stripping [4]. Ammonia volatilization is the transfer of NH_3 from liquid phase to the atmosphere across a water–air interface [5]. Ammoniacal nitrogen can be removed from liquid bulks by converting NH_4^+ into NH_3 through elevating pH and followed by desorption of NH_3 gas [6]. Ammonia stripping has been successfully employed by many authors and modeled in detail [5–10]. Air stripping is a popular technique which is used to remove volatile

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organic chemicals and oxidize contaminants such as iron and manganese. It could improve taste and odor from water sources [11]. Various gasses such as carbon monoxide and hydrogen sulfide can be removed with air stripping. The process could improve the rate of gas evaporation by increasing the contact surface area between air and water [12]. It has offered several advantages such as ability of stripping the volatile compounds, low cost, and effective technique for ammonia removal at high concentration [13,14]. Air stripping has various disadvantages like removal dependency on the Henry's law coefficient, excess air-flow, providing proper air and water balance, scaling, and biological growth [12]. A successful air stripping performance depends on various factors that can be asserted as follows: (i) characteristics of volatile material such as partial pressure, Henry's constant, and gas transfer resistance; (ii) solution and ambient air temperature; (iii) turbulence in gaseous and liquid phases; (iv) area-to-volume ratio; and (v) stripping time [12–15].

Various statistical experimental design techniques have been proposed in the optimization of experimental studies. Response surface methodology (RSM) was described by Box and Wilson as an experimental approach to identify the optimum conditions for a multivariable system using minimum experimental samples [16]. It has been used to make an experimental design in different experimental studies [17–19]. The aim of the present study is to investigate the optimization of the most important ammonia stripping parameters using RSM. Among the important ammonia stripping parameters, temperature and pH are more effective and their strong influence depends on the type of used salt (NH_4Cl or NH_4CO_3). In this case, the ammonium chloride was used and the interaction between the two parameters was investigated using RSM. For the optimization, the main parameters were considered and under the optimized conditions the ammonia kinetic was evaluated.

2. Experimental

2.1. Material

All the chemicals used in this work were of analytical reagent grade and were used without further purification. An aqueous stock solution of ammonia (from NH_4Cl salt) was prepared in deionized distilled water. Different concentrations of ammonia were obtained by diluting the stock solution. The pH was adjusted to the desirable value with 1 mol/L of HCl and NaOH.

2.2. Experimental setup

To optimize the main parameters of ammonia, volatilization was employed from a diffuser air stripper system. A glass column (with the effective volume of about 1,000 mL) was used for air stripping experiments (30 cm height \times 8 cm internal diameter). Flow rate was maintained at approximately 2 L/min and the operating parameters including ammonia, nitrate, nitrite, pH, oxidation reduction potential (ORP), and temperature were evaluated. The aeration time was considered as 4 h for all the experiments.

2.3. Experimental design and mathematical modeling

The coefficients of the response functions for different dependent variables were determined in correlation to the experimental results with the response functions using a design-expert regression program. Central composite design (CCD) was used to introduce this model as a specific design. CCD of the main parameters (x_1 : ammonia concentration 50–2,000 mg/L, x_2 : pH 7–12, and x_3 : wastewater temperature 24–40°C) is shown in Table 1. The three operating variables were considered at three levels, namely low (–1), central (0), and high (1). According to the proposed design by the software (Design-Expert 7.0, trial version), 20 experiments were conducted which included three repetitions to get a good estimate of the experimental error. Repetition experiments were carried out after the main experiments followed by the order of runs, as shown in Table 2. In order to carry out the comprehensive analysis of the air stripping process, six dependent parameters were either directly measured or calculated as response. These parameters were considered as important parameters that can affect the performance of air stripping. To determine the air volatilization efficiency, residual ammonia was determined. Nitrite/nitrate contents were analyzed as the intermediate byproduct that may be raised. To evaluate the redox condition, ORP was considered. After conducting the experiments, the coefficients of the polynomial model were calculated using the following:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i=1}^k \sum_{j=1}^k \beta_{ij} x_i x_j + \varepsilon \quad (1)$$

where Y : the considered response, $\beta_0, \beta_i, \beta_{ii}, \beta_{ij}$: the coefficients of regression, ε : the error value of the system, and x_j : coded variables. The results were analyzed using analysis of variance (ANOVA) by Design-Expert. Three-dimensional (3D) plots and their

Table 1
Experimental range and levels of variables

Variable	Unit	Symbol	Low axial (-2) - α	Low factorial (-1)	Center point (0)	High factorial (+1)	High axial (+2) + α
Ammonia Concentration	mg/L	x_1	50	537.5	1,025	1,512.5	2,000
pH	–	x_2	7	8.25	9.5	10.75	12
Temperature	°C	x_3	24	28	32	36	40

Table 2
CCD consisting of 20 experiments for the study of three experimental factors in coded units along with observed values

Run	x_1	x_2	x_3	Ammonia removal Eff. %	
				Actual	Predicted
1	1	1	1	40.67	41.62
2	-1	1	-1	25.34	25.86
3	0	0	0	29.8	29.99
4	1	-1	-1	10.94	10.80
5	-1	-1	-1	13.35	11.96
6	0	0	0	30.21	29.99
7	0	-2	0	2.71	2.03
8	1	1	-1	35.61	31.74
9	0	0	2	37.98	36.20
10	-2	0	0	7.2	5.85
11	-1	-1	1	10.34	13.76
12	2	0	0	15.58	17.41
13	0	0	0	28.66	29.99
14	0	0	0	29.5	29.99
15	0	0	0	30.98	29.99
16	-1	1	1	29.18	28.90
17	0	0	-2	22.29	24.52
18	0	0	0	30.34	29.99
19	1	-1	1	20.41	19.44
20	0	2	0	36.98	38.11

respective contour plots were obtained based on the effect of the levels of the two factors. From the 3D-plots, the simultaneous interaction of the two factors on the responses was studied. The optimum region was also identified based on the main parameters in the overlay plot.

2.4. Analytical methods

The samples were collected from the reactor using a 10-mL syringe. Ammonia and nitrate were analyzed in accordance with the standard method [20]. Nitrate was determined using an spectrophotometer at λ_{\max} 220 and 275 nm. The nitrite content was analyzed by

colorimetric method using sulfanilamide and naphthylethylenediamine di-hydro-chloride reagents at λ_{\max} 543 nm. The determination of ammonia was performed by phenate method (λ_{\max} 640 nm). The pH and ORP were determined by a portable pH and ORP tester (Euteck, Singapore).

3. Results and discussion

3.1. ANOVA

Twenty experimental conditions of the runs proposed by the CCD were conducted in the laboratory to determine the responses. Process performance was evaluated by analyzing the experimental results of all these responses [21]. The final second-order polynomial regression in terms of coded and actual factors was represented by the following equations.

Final equation in terms of coded factors:

$$\begin{aligned} \text{Ammonia stripping eff. (\%)} = & +29.99 + 2.89x_1 + 9.02 \\ & \times x_2 + 2.92x_3 + 1.76x_1x_2 \\ & + 1.71 \times x_1x_3 + 0.31x_2x_3 \\ & - 4.59x_1^2 - 2.48 \times x_2^2 \\ & + 0.092 \times x_3^2 \end{aligned} \quad (2)$$

Final equation in terms of actual factors:

$$\begin{aligned} \text{Ammonia stripping eff. (\%)} = & -150.15 - 0.01 \times \text{conc.} + 32.46 \times \text{pH} - 1.11 \\ & \times \text{Temp.} + 2.89\text{E} - 003 \times \text{conc.} \times \text{pH} + 8.78\text{E} - 004 \\ & \times \text{conc.} \times \text{Temp.} + 0.061 \times \text{pH} \times \text{Temp.} - 1.93\text{E} \\ & - 005 \times \text{conc.}^2 - 1.58 \times \text{pH}^2 + 5.76\text{E} - 003 \\ & \times \text{Temp.}^2 \end{aligned} \quad (3)$$

The optimal processing conditions from numerical optimization were obtained as 1,440 mg/L, 10.7, and 36°C for initial ammonia concentration, pH, and temperature, respectively.

The response surface models were validated statistically for adequacy by ANOVA. Table 3 shows the

Table 3
ANOVA for response surface quadratic model for ammonia stripping

Source	Sum of squares	df	Mean square	F-value	p-value
Model	2,258.8	9	250.98	51.16	<0.0001
x_1	133.29	1	133.29	27.17	0.0004
x_2	1,301.41	1	1,301.41	265.29	<0.0001
x_3	136.54	1	136.54	27.83	0.0004
x_1x_2	24.85	1	24.85	5.07	0.0481
x_1x_3	23.46	1	23.46	4.78	0.0536
x_2x_3	0.74	1	0.74	0.15	0.7051
x_1^2	530.66	1	530.66	108.18	<0.0001
x_2^2	154.68	1	154.68	31.53	0.0002
x_3^2	0.21	1	0.21	0.044	0.8389
Residual	49.06	10	4.91		
Lack of fit	45.89	5	9.18	14.51	0.0053
Pure error	3.16	5	0.63		
Cor total	2,307.85	19			
Std. dev.	2.21		R^2	0.9787	
Mean	24.4		Adj R^2	0.9596	
C.V. %	9.08		Pred R^2	0.8416	
Press	365.5		Adeq precision	25.274	

ANOVA of regression parameters of the predicted response surface quadratic model for ammonia removal efficiency. According to the results, the model F-value of 51.16 and low probability value (p -value < 0.0001) indicated that the model was significant for air stripping process. According to the obtained results, the optimal points of working conditions and predicated removal efficiencies of ammonia were established (Fig. 1). The experimental analysis value indicated a good agreement with the predicate values.

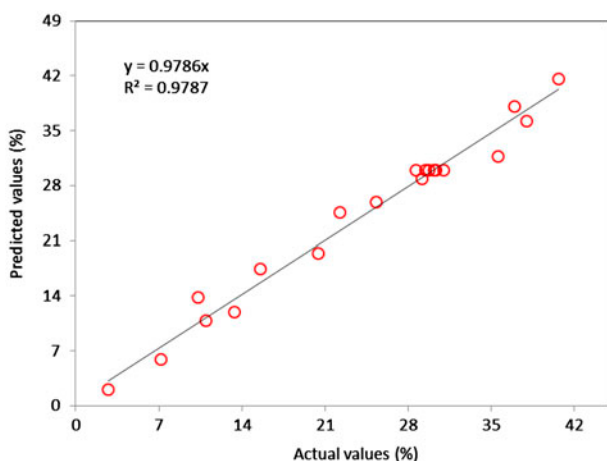


Fig. 1. Predicted vs. actual values plot for ammonia removal efficiency using air stripping process.

3.2. Effects of operational parameters on removal efficiency

The amount of ammonia removed from a liquid in a diffused aeration system depends on the concentration of ammonium nitrogen, pH, temperature, rate of air flow, volume of liquid, duration of desorption, and mass transfer coefficient [22]. The 3D surface responses of the quadratic model were achieved by Design-Expert software and used to evaluate the interactive relationships between independent variables and response. In each plot, one variable was kept constant, while the other two were varied within the experimental ranges. Fig. 2(a) and (b) shows the effect of pH, temperature, and initial ammonia concentration on ammonia removal efficiency. Ammonia removal increased with a rise in temperature and pH values. The maximum ammonia removal was at pH 10.7 and 36°C. However, the effect of pH was more significant than temperature for the volatilization of ammonia from the solution. The obtained results were compatible with those of other studies that have investigated the performance of ammonia removal by air stripping process [23]. Higher ammonia removal can be obtained by displacement ammonium/ammonia equilibrium at higher pH values. Ammonium ions in wastewater exist in equilibrium with gaseous ammonia (Eq. 4) [24]. Therefore, at higher pH, there is greater proportion of nitrogen in the form of ammonia gaseous and higher percent is stripped.



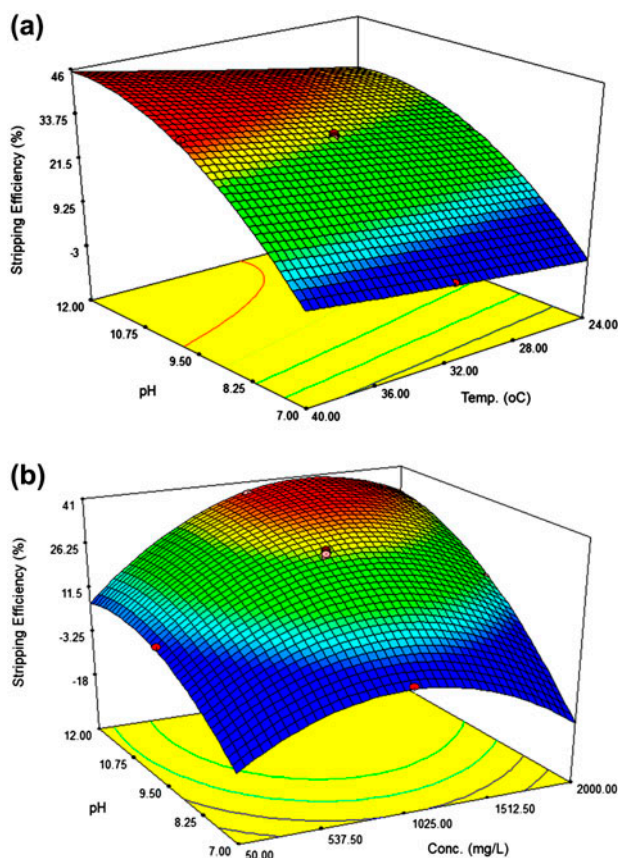


Fig. 2. Three-dimensional graphics of response surface for ammonia removal efficiency using air stripping process (experimental conditions: pH 7–12, temperature 24–40°C, and initial ammonia concentration 50–2,000 mg/L).

Ammonia removal efficiency increased with the aqueous phase temperature, as shown in Fig. 2(a). When the temperature increased over 30°C, the effect was more significant. The increase in temperature would promote the molecular diffusion of ammonia in a gas film [25]. In the case of ammonium bicarbonate, Campos et al. demonstrated that the ammonia removal efficiency increased significantly at 60°C over a constant time period (7 h) rather than lower temperatures (25 and 40°C) [26]. Their results revealed that the strong influence of temperature on the ammonia removal rate was between 40 and 60°C [26].

The effect of the initial ammonia concentration on the air stripping efficiency of ammonia is shown in Fig. 2(b). It exhibits the very high air stripping efficiency of ammonia in a wide range of ammonia concentration (500–2,000 mg/L). The highest stripping efficiency was obtained when the initial ammonia concentration was at the central point within the studied range. Under optimal conditions, effect of flow rates (1.8, 2, and 3 L/min) was evaluated on the air

stripping process (Fig. 3). The ammonia removal percentages were about 35.78, 38.9, and 51.64% for air flow rates (air-to-water ratio) of 1.8 (432 v/v), 2 (480 v/v), and 3 L/min (720 v/v), respectively. According to the obtained results, it can be concluded that the ammonia stripping efficiency was strongly influenced by the air flow rate or air-to-water ratio. Similar results were reported by Alam and Delwar Hossain [24]. Mass transfer of ammonia to the air depends on the difference in the concentration level of ammonia in the liquid phase and in the air phase [27]. Generally, a higher air to water ratio provides constant low concentration of ammonia in the air and promoting the transfer of ammonia into the air phase [10]. After 4 h of aeration, the rates of produced nitrate and nitrite were calculated. The increase in

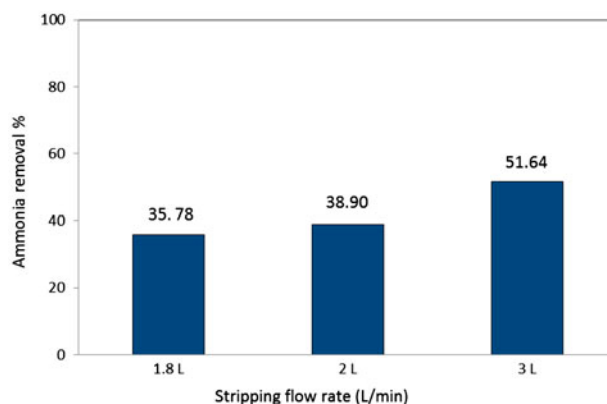


Fig. 3. Effect of air flow rate volume on ammonia air stripping (experimental conditions: pH 10.7, temperature 36°C, and initial ammonia concentration 1,440 mg/L).

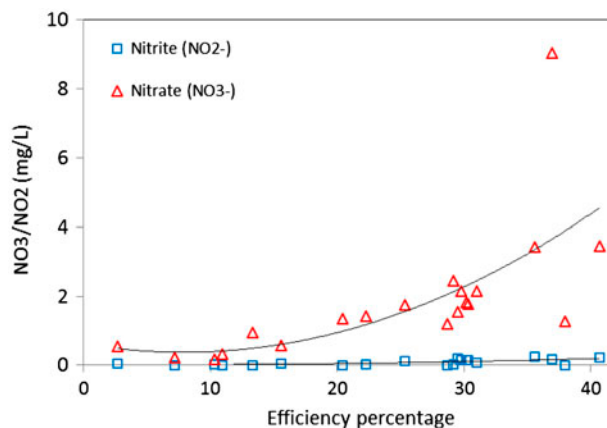


Fig. 4. Nitrate and nitrite production during air stripping (experimental conditions: pH 10.7, temperature 36°C, initial ammonia concentration 1,440 mg/L, and air flow rate 3 L/min).

ammonia removal efficiency caused the increased nitrate/nitrite production. The rate of nitrate and nitrite generation was determined less than 5 and 0.5 mg/L, respectively. Nitrite is an unstable form of nitrogen which can be converted into other forms of nitrogen compounds. Degasification of ammonia by air stripping occurs by the mass transfer of ammonia from liquid bulk (wastewater) to air. Therefore, the low concentrations of ammonia is converted. Therefore, low concentration of nitrite is observed during the air stripping process (Fig. 4). Valero and Mara reported that the negligible amount of nitrate and nitrite (undetectable) via ammonia volatilization in maturation ponds were provided [28].

Endpoint data of the ORP for ammonia volatilization are presented in Fig. 5. Generally, there is an inverse relationship between pH and ORP, regardless of the oxidant type or concentration [29]. When the initial pH was increased, the ORP was decreased. On the other hand, pH value can affect the reductive reaction of oxygen as demonstrated in the following equation:



So, the increase in hydroxyl content would slow down the reduction of oxygen, where ORP decreased with an increase in pH [30]. Moreover, higher ammonia removal percentage occurred at higher pH values. However, a decrease in ORP and increase in pH were observed with increasing the ammonia removal percentage.

3.3. Kinetic and half-life of ammonia volatilization

The obtained data were analyzed to investigate the kinetic of air stripping reaction. Under optimum

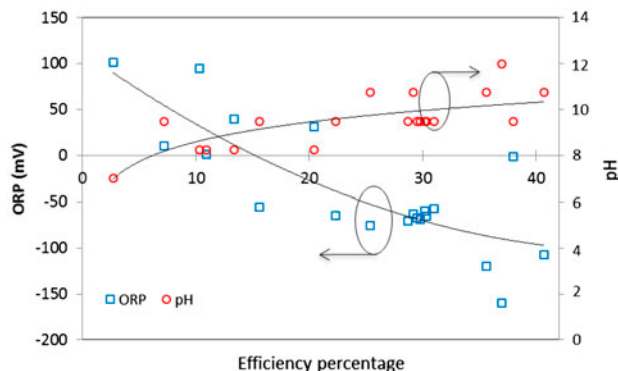


Fig. 5. Variations of pH and ORP during air stripping (experimental conditions: Temperature 36°C, air flow rate 3 L/min, and initial ammonia concentration 1,440 mg/L).

conditions, kinetic experiments were carried out by the air-to-water volume of 3 L/min at different contact times 0–12 h. In the present work, the air stripping of ammonia was evaluated by various common models.

The reaction rate equation of the first-order reaction kinetic can be expressed as follows (Eq. (6)):

$$\ln \frac{C}{C_0} = -kt \quad (6)$$

where C_0 and C are initial ammonia concentrations (mg/L) and ammonia at time t (h) and k is ammonia loss rate constant or ammonia stripping constant rate (1/h).

The first-order kinetic model is drawn using $\ln(C/C_0)$ vs. time (Fig. 6(a)). With regard to linear equation ($y = 0.1395x + c$) and its slope, the kinetic constant (k L/min) was calculated. Accordingly, constant value (k) of the kinetic was obtained as about 0.1395 per h. Experimental results showed that the first-order kinetic with $R^2 = 0.984$ was in good conformity with air stripping data.

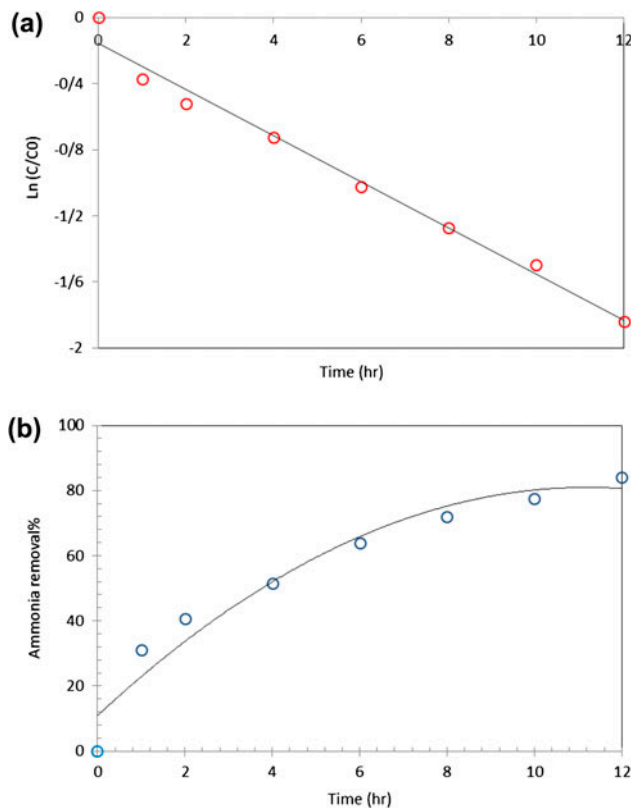


Fig. 6. First-order kinetic model fit for ammonia air stripping (a), ammonia removal efficiency plot (b) (experimental condition: Temperature 36°C, initial ammonia concentration 1,440 mg/L, pH 10.7, and flow rate 3 L/min).

The half-life equation for ammonia stripping can be obtained as follows (Eq. (7)):

$$t_{1/2} = \frac{\ln 2}{k} \approx \frac{0.693}{k} \quad (7)$$

The half-life constant ($t_{1/2}$) for ammonia volatilization was determined as ~4.97 h. Therefore, based on the half-life, to remove 50% of ammonia via air stripping, the time of about 5 h was needed. Finally, maximum removal percentage was acquired as equal to 84.11% at the end of 12 h (Fig. 6(b)). Similarly, Marttinen et al. [31] reported 89% ammonia removal at pH 11, 20°C, and 24 h. Also, Cheung and Silva reported the removal efficiency about 93 and >99.5% for 1,600 (24 h) and 800 mg/L (96 h), respectively [32,33].

4. Conclusion

In this work, ammonia volatilization using air stripping was investigated for modeling and kinetic aspects. Air stripping process optimization concentrated on the influence of operating variables, such as pH, temperature, initial concentration, and flow rate using RSM with CCD. Furthermore, interaction among all the components was evaluated employing RSM. The multiple correlation coefficient of determination R^2 was 0.978, showing that the actual data well fitted the predicted data. The optimum results obtained from the model indicated that 4 h of contact time was required to achieve ammonia removal when the temperature and pH were 36°C and 10.7, respectively. The kinetic followed the first-order. According to the obtained results, air stripping process can be used for the efficient removal of ammonia with high concentration.

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