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Optimization of Ammonia Removal from Landfill Leachate by Aeration Using Response Surface Methodology (RSM)

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Abstract

Landfill leachate has a high concentration of ammonia, making it a harmful pollutant for both surface and groundwater. One of the most favoured methods for removing ammonia from leachate is aeration, as it has been proven to remove a significant amount of ammonia in the most efficient and economical way. The effect of operational variables on ammonia removal efficiency by aeration was investigated in the current study by applying Response Surface Methodology (RSM) approach. Three operating parameters such as airflow rate, aeration time and lime dosage were investigated to achieve the optimization of ammonia removal. The optimal parameters for a favourable reaction of ammonia-nitrogen (NH3N) removal were found to be 6 L/min airflow, 90 minutes aeration time, and a lime dosage of 6 g/L. At these ideal conditions, Quadratic RSM predicted a maximum NH3N removal of 98.0%, which has been validated by the experiment and successfully removed 97.6%. The finding also showed that airflow rate and aeration time were more significant than lime dosage for NH3N removal. Due to increased contact time between air and liquid, regardless of the amount of lime used, increasing the aeration period ammonia removal efficiency. Considering the influential factors, determining the optimum condition for ammonia removal by aeration will explain the potential interferences that may inhibit the efficient recovery of NH3N. Hence, aeration is a promising approach for ammonia removal from landfill leachate.

1. Introduction

Landfill leachate is a by-product of sanitary or scheduled waste landfills produced when rainwater moves through waste in a landfill. It is primarily composed of a high level of organic matter, including dissolved organic matter, phosphate, NH3N, heavy metals, inorganic salts, solids, salinity, and other pollutants [3], [11]. Among all pollutants NH_3N is a particular concern due to its high concentration in leachate, which has been reported in many studies

[14]. The complexity of leachate characteristics makes it difficult to be treated. In fact, high concentrations of NH_3N in the leachate cause biological treatment to be impossible. This is because it inhibited not only nitrite-oxidizing bacteria (NOB) but also ammonia-oxidizing bacteria (AOB) [12]. Hence, before the downstream treatment process, a pre-treatment to remove the NH_3N is needed.

According to previous research, aeration is a viable method for removing NH_3N from landfill leachate [4], [7], [13]. The efficiency of aeration may be influenced by various factors, including the concentration of ammonia, temperature, pH levels, contact duration or stripping time, characteristics of volatile materials, airflow rate, types of packing materials used, and the ratio of surface area to volume. [5]. Among these factors, it is essential to determine the most economical and convenient operating factors to determine the efficiency of ammonia removal from landfill leachate. Considering the influential factors stated above, air flow rate, aeration time and lime dosage have been proven to be essential operating parameters. However, no study reported the effects of airflow rate, aeration time and lime dosage simultaneously on the removal efficiency of ammonia from landfill leachate.

Hence, this research studies the landfill leachate characteristics and determines the optimal condition for ammonia removal by aeration process under three operating parameters; airflow rate, aeration time and lime dosage. Determining the optimum condition for ammonia removal by aeration will explain the potential interferences that may inhibit the efficient removal of NH_3N.

This study uses the Design Expert Software Version 12.0 to apply the Central Composite Design (CCD) of response surface methodology (RSM) to optimize all operating parameters. Simultaneously, this study will contribute to environmental protection by attaining sustainable recovery of NH_3N from landfill leachate and producing valuable material, such as fertilizer. Furthermore, this can be considered a milestone for large-scale ammonia removal and recovery from landfill leachate.

2. Methods

2.1 Leachate Sampling and Characterization

Leachate samples were taken from the Jeram Sanitary Landfill in Kuala Selangor, Malaysia, located on an oil palm plantation. Grab sampling was done with a total of 20L of landfill leachate taken from the equalization pond. The samples were collected and preserved following the Standard Methods for the Examination of Water and Wastewater [2]. To av]oid biological and chemical interactions, all samples were promptly transferred to the laboratory and maintained in a refrigerator at 4°C before being used for experiments. The physicochemical characteristics of leachate samples were assessed based on the American Public Health Association [2]. The Biological Oxygen Demand (BOD), Chemical Demand (COD), NH_3N, Total Suspended Solids (TSS), Total Dissolved Solids (TDS), pH and heavy metals are among the parameters included, and to get an average value, all parameters were examined three times.

2.2 Analytical Methods

A portable photometer (Hanna Instruments, Model: 96733) was used to measure the concentration of NH_3N using the Nessler Method (Method: 8038). Eq. (1) was applied to calculate the efficiency of ammonia nitrogen removal:

$$\mathrm{NH_3N\ Removal\ (\%)} = \left(C_i - C_f\right)/C_i \times 100 \tag{1}$$

where C_i and C_f are the initial and final concentrations of NH_3N (mg/L), respectively.

Meanwhile, Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES Optima 4300DV) was used to analyse heavy metals, according to APHA Standard Methods 3120B. The elements that were determined in the leachate sample were Copper (Cu), Nickel (Ni), lead (Pb), Zinc (Zn) and Chromium (Cr). Before analysis, all leachate samples were digested in concentrated nitric acid (HNO_3), capped, and digested at 105°C for 20 minutes. The samples were filtered using Whatman 41 to form a final of 10 ml solution when cooled.

2.3 Experimental Set-up for Aeration

Fig. 1 depicts the experimental setup for the aeration process. After being characterized, 100 mL of leachate sample was aerated under different conditions. An air pump with an adjustable airflow rate is connected to a closed glass bottle, which supplies sufficient oxygen for ammonium oxidation. An airflow meter was deployed to control the air from the air pump. A few trials indicated that the DO concentration in the system increased to 10.57~16.51 mg/L when the airflow rate was 2- 6 L/min. The pH of the leachate sample was fixed at 11 as the ammonia removal efficiency is higher at pH 11 [13]; hence sodium hydroxide (NaOH) solution was used for pH adjustments before aeration.

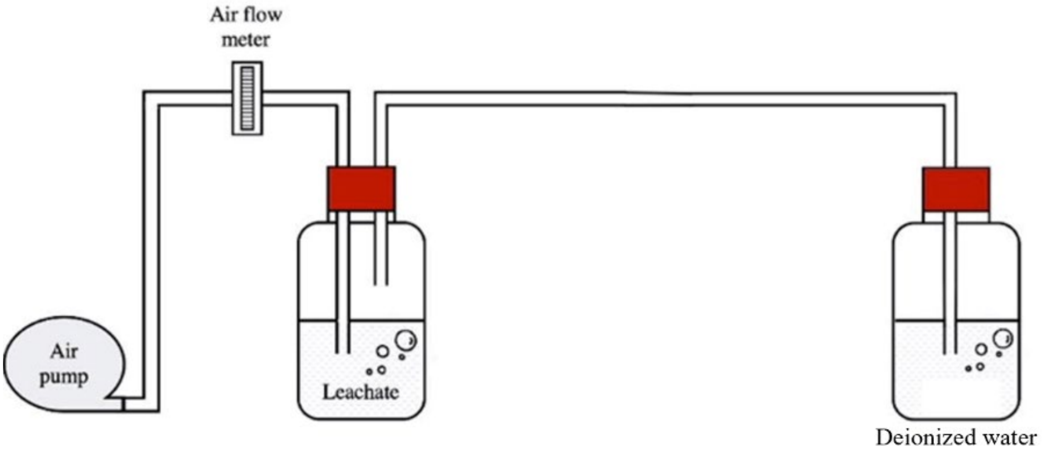

Fig. 1 *Experimental set up for aeration*

2.4 Experimental Design

Response Surface Methodology (RSM) was employed to construct an experimental procedure aimed at the removal of NH_3N from landfill environments. This methodology facilitated the assessment of the interplay between the response variable (ammonia nitrogen removal) and various independent factors. By optimizing the conditions of pertinent variables, the objective was to anticipate the optimal response outcome. In this context, the Central Composite Design (CCD), a widely utilized RSM technique, was implemented to ascertain the influence of operational factors on the efficiency of ammonia nitrogen removal. With the help of the Design-Expert software application, CCD and RSM were created. Airflow rate (A), aeration duration (B), and lime dosage (C) were the three significant independent variables evaluated in this investigation, as shown in Table 1.

Table 1 *Total of runs and respective values for three process parameters generated with Design Expert Version 12.0*

Run No.	Factor A Airflow rate (L/min)	Factor B Aeration time (minute)	Factor C Ca $(OH)_2$ dosage (g/L)
1	2	40	6
2	6	40	6
3	6	120	2
4	6	40	2
5	2	120	2
6	4	40	4
7	2	80	4
8	4	80	2
9	4	80	6
10	6	120	6
11	4	120	4
12	2	40	2
13	2	120	6
14	4	80	4
15	4	80	4
16	6	80	4

3. Results and Discussion

3.1 Leachate Characterization

There are 11 parameters involved for leachate characterization. The value for the parameters of the leachate sample is shown in Table 2.

TDS levels in leachate samples taken from a planned waste disposal were higher in 2012 (5306 mg/L). The composition of the waste and the amount of water in the total waste may determine the physio-chemical parameters of the landfill leachate. Table 2 shows the characteristics of the leachate samples obtained from the Jeram landfill site as an average of all samples. The leachate studies reveal that the COD concentration was 1536 mg/L; the BOD was 182; TSS was 691 mg/L, and TDS was 2330 mg/L. The comparatively high TDS (2330 mg/L) values in the samples indicate the presence of substantial inorganic material. Leachate samples from a scheduled waste landfill were collected in 2012, and a higher level of TDS (5306 mg/L) was found [6]. In landfill leachate, a more significant proportion of TDS comprises inorganic salts and dissolved organics [15].

Table 2 *Comparison of leachate samples collected from various landfills*

Parameter	Units	This study (Municipal Waste)	Sani et al. [12] (Scheduled Waste)	Halim et al. [6] (Scheduled Waste)
pH	-	8.28	9.92	8.30
BOD	mg/L	182	196	358
COD	mg/L	1536	4,852	1788
NH_3N	mg/L	3467	2403	1380
TSS	mg/L	691	NA	NA
TDS	mg/L	2330	NA	5306
Pb	µg/L	17	0	3
Cu	µg/L	53	30	10
Zn	µg/L	493	0	300
Cr	µg/L	223	65	200
Ni	µg/L	183	500	100

*NA-not available

Subsequently, it is notable that the leachate sample exhibited a notably high NH3N concentration, measuring at 3467 mg/L. This elevated concentration could likely be attributed to the processes of hydrolysis and fermentation involving the nitrogen-rich components of biodegradable substrates. Additionally, the release of soluble nitrogen from municipal solid waste could contribute to this heightened level of concentration. According to [6], [12], they also found a high number of NH_3N, 2403 mg/L and 1380 mg/L, respectively [6], [12].

Meanwhile, for heavy metals, the result shows that the amount of Pb, Cu, Zn, Cr and Ni was 17 µg/L, 53 µg/L, 493 µg/L, 223 µg/L, and 184 µg/L, respectively. Zn and Cr are the most abundant heavy metals recorded in this study. These results are almost identical to the values of Zn and Cr recorded by [12], which are 300 µg/L and 200 µg/L, respectively. Fluorescent bulbs, batteries, and various food wastes are all sources of Zn, whereas discarded food is the primary source of Cu, Pb, Cr, and Cr, which are harmful heavy metals found in the samples [8]. Not only are these common metals among the most poisonous and carcinogenic pollutants, but they also do not decompose, posing a significant hazard to the environment and human health [1].

In addition, leachate characteristics can be influenced by the quality and quantity of wastewater from different areas, which has been shown to affect leachate quality considerably. For instance, the amount of ammonia nitrogen in leachate from European and American countries, for example, is often less than 1000 mg/L, whereas it is typically more than 1000 mg/L in Asian countries. These discrepancies could be due to regional cultural and behavioral variances and the age of the studied leachate landfills.

3.2 Analysis of Variance (ANOVA)

The performance of the aeration process was assessed by examining the experimental results. Table 3 shows the removal efficiency of 16 runs. From the table, the ammonia removal efficiency was recorded at 97.2%, where the range of airflow rate, aeration time and lime dosage were set on 6 L/min, 120 mins and 6g/L, respectively. The point of lowest removal efficiency, registering at 40.2%, was observed under specific conditions where the airflow rate, aeration time, and lime dosage were set at 2 L/min, 40 minutes, and 2 g/L, respectively. From this data, it becomes evident that heightened removal efficiency is achieved when all three operational parameters are set to higher values. Meanwhile, for the analysis of variance (ANOVA), the following Eq. (2) represented all terms of coded factors whereby Y is the Ammonia Removal Efficiency:

$$\mathrm{Y_{NH3N}} = 83.06 + 10.61A + 14.56\,B + 3.67\,C - 1.86\,AB - 0.2625\,\mathrm{AC} - 0.8875\,BC - 0.7879\,A^2 - 10.04\,\mathrm{B}^2 - 0.2879\,\mathrm{C}^2 \tag{2}$$

The best parameters for NH_3N removal efficiency were chosen based on the responses. ANOVA was used to test the appropriateness of the response surface models statistically. The ANOVA of regression parameters of the anticipated response surface quadratic model for NH_3N removal efficiency is shown in Table 3, indicating that the model was significant ($p<0.0001$) for the aeration process, whereby model terms with P-values less than 0.0500 are substantial. A, B, C, and B^2 are essential to model terms in this case. The model terms are not significant if the value is more than 0.1000. The F-value of 8.99 for the Lack of Fit indicates that the Lack of Fit is insignificant compared to the pure error. Due to noise, a significant Lack of Fit F-value has a 24.78% chance of happening.

Table 3 *Removal efficiency of 16 runs*

Run No.	Factor A	Factor B	Factor C	Response
	Airflow rate (L/min)	Aeration time (minute)	$Ca(OH)_2$ dosage (g/L)	Removal efficiency of NH_3N (%)
1	2	40	6	49.5
2	6	40	6	73.0
3	6	120	2	92.5
4	6	40	2	64.3
5	2	120	2	75.4
6	4	40	4	61.0
7	2	80	4	70.3
8	4	80	2	79.8
9	4	80	6	87.6
10	6	120	6	97.2
11	4	120	4	86.9
12	2	40	2	40.2
13	2	120	6	81.6
14	4	80	4	80.6
15	4	80	4	81.8
16	6	80	4	96.1

Table 4 *ANOVA for response surface quadratic model for ammonia removal*

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3834.03	9	426.00	77.30	< 0.0001	Significant
A-Airflow rate	1125.72	1	1125.72	204.27	< 0.0001	
B-Aeration time	2119.94	1	2119.94	384.67	< 0.0001	
C-Lime dosage	134.69	1	134.69	24.44	0.0026	
AB	27.75	1	27.75	5.04	0.0660	
AC	0.5512	1	0.5512	0.1000	0.7625	
BC	6.30	1	6.30	1.14	0.3261	
A^2	1.64	1	1.64	0.2970	0.6054	
B^2	265.64	1	265.64	48.20	0.0004	
C^2	0.2186	1	0.2186	0.0397	0.8487	
Residual	33.07	6	5.51			
Lack of Fit	32.35	5	6.47	8.99	0.2478	
Pure Error	0.72	1	0.72			
Cor Total	3867.10	15				
Std. Dev	2.35			R^2	0.9914	
Mean	76.11			Adj R^2	0.9786	
C.V%	3.08%			Pred R^2	0.9443	
Press				Adequate precision	31.0790	

Furthermore, as depicted in Table 4, a noteworthy observation is that the coefficient of determination (R^2) stands at 0.9443. This signifies a substantial concurrence between the anticipated NH_3N removal efficiencies and the optimal operational conditions, aligning the experimental findings closely with the predicted values. With a difference of less than 0.2, the anticipated R^2 of 0.9443 is in reasonable agreement with the corrected R^2 of 0.9786. The correlation coefficient should be at least 0.80, according to [9], for a decent fit of a model. The response surface model developed in this work for predicting NH_3N removal efficiency was found to be reasonable based on the results. Simultaneously, the signal-to-noise ratio is determined with a commendable degree of accuracy, with a preference for ratios exceeding four. In this context, the model serves as a valuable tool for traversing the design space, owing to the substantial ratio of 31.079 that delivers a robust signal strength.

3.3 The Efficiency of Ammonia Removal

Leveraging the Design-Expert software, a comprehensive 3D surface response of the quadratic model was generated. This visualization facilitated the examination of interaction effects between the independent factors and the corresponding response variable. The influence of airflow rate, aeration period, and lime dose on NH_3N removal efficiency for raw leachate is shown in Fig. 2 to Fig. 4. The removal of NH_3N increased as the airflow rate and aeration time increased. The NH_3N removal response surface showed that the higher the airflow rate, the greater the NH_3N removal efficiency (Fig. 2). At a flow rate of 6 L/min and aeration time of 120 minutes, the most effective NH_3N removal was observed. The higher airflow rate of the aeration process resulted in higher ammonia removal efficiency [10]. An increased airflow rate enhances ammonia transfer into the air phase [7].

However, this study found that airflow rate and aeration time were more significant than lime dosage for NH_3N removal. The enhancement in ammonia removal efficiency is evident as aeration time is prolonged, irrespective of the lime dosage. This improvement can be attributed to the extended interaction period between the air and the liquid, resulting in increased contact time [7]. The maximum time (120 min) showed higher ammonia removal efficiency (Fig. 2 and Fig. 3).

Furthermore, a notable observation emerged when the lime ($Ca(OH)_2$) dosage was configured at 6 g/L, resulting in elevated NH3N removal. Notably, these outcomes align with earlier research findings [7]. To achieve substantial ammonia removal through the displacement of ammonium equilibrium, it becomes imperative to employ alkaline agents like lime for pH elevation Ammonium ions (NH_4^+) are in equilibrium with gaseous ammonia (NH_3) in wastewater. When the hydrogen ion (OH^-) from Ca $(OH)_2$ reacts with the ammonium ion (NH_4^+) in the leachate, NH_3 gas is produced. Most ammonium ions (NH_4^+) in the pH range of 10.5 to 12 are in the form of NH_3. As a result, greater pH causes more nitrogen in the form of gaseous ammonia, which is then eliminated from the liquid [7].

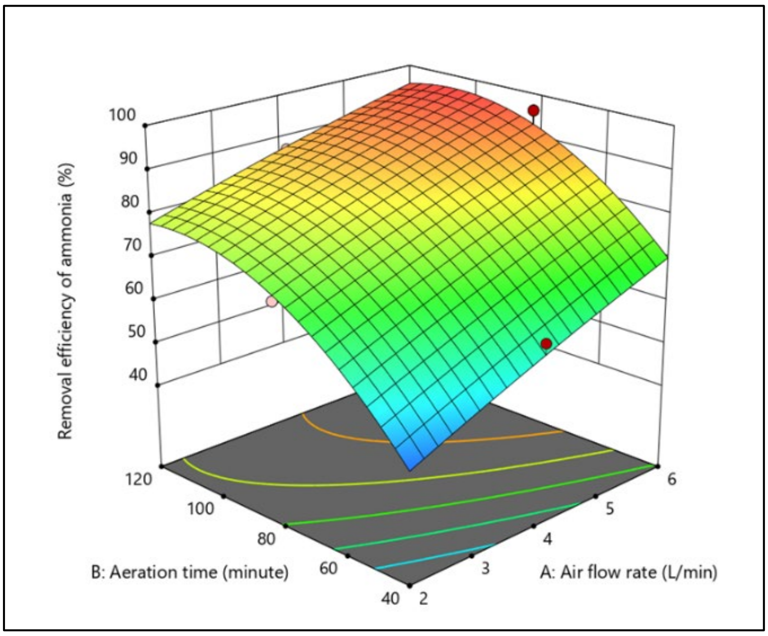

Fig. 2 *Response surface plot for NH_3N removal at different airflow rates and aeration times*

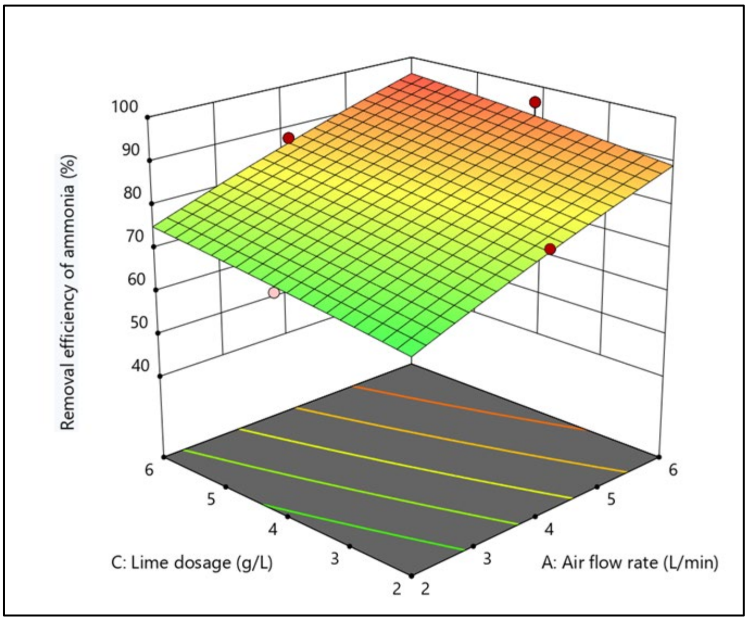

Fig. 3 *Response surface plot for NH_3N removal at different aeration times and lime dosage*

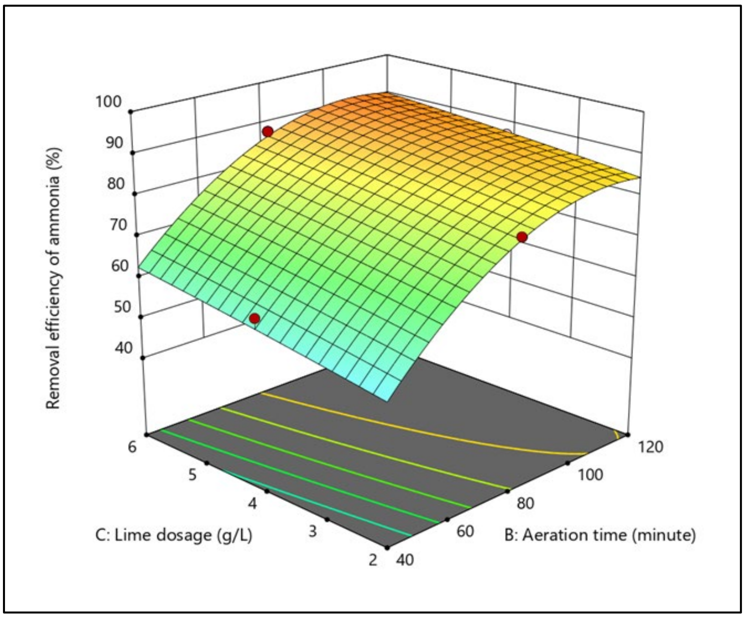

Fig. 4 *Response surface plot for NH3N removal at different aeration*

3.4 Optimization of Operating Parameters

An optimization procedure was carried out using the Design-Expert software to establish the best value of NH_3N removal efficiency. The experimental statistical procedures were validated using the second-order polynomial model obtained from RSM at the optimum airflow rate, aeration time, and lime dosage. The criteria for optimization can be chosen to identify the most optimum operating conditions after the model has been constructed from experimental findings and validated for adequacy. In order to achieve the best results, the target aim for each operating condition (airflow rate, aeration time, and lime dosage) was chosen "within the range" in the software optimization step, whereas the response (NH_3N removal efficiency) was set as "maximum". Simultaneously, the range also integrates the various desires into a single number, attempting to maximise the response. As a result, the best operating circumstances and per cent removal efficiencies were determined, and the findings are shown in Table 5.

Table 5 *Optimum working conditions and respective per cent removal efficiency*

Solution No.	Airflow Rate (L/min)	Aeration Time (min)	Lime Dosage (g/L)	Removal Efficiency of Ammonia (%)	Desirability
1	6	90	6	98.1	1
2	6	96	5	98.1	1
3	6	98	5	97.4	1
4	6	111	5	97.2	1
5	6	118	6	97.6	1

As indicated in Table 5, the model anticipated that under optimal operational conditions, 98.1% of NH_3N would be removed (airflow rate of 6; aeration time of 90 min; and lime dosage of 6). For these ideal conditions, the desirability function value was found to be 1. After that, a second experiment was conducted to validate the best outcomes. Verification experiments based on the projected RSM parameters were carried out three times to validate this. The experimental validation as shown in Table 6 run yielded an average of 97.6% NH_3N removal, which is consistent with the RSM's predicted response value.

Table 6 *Validation of the NH_3N removal efficiency*

Solution No.	Airflow Rate (L/min)	Aeration Time (min)	Lime Dosage (g/L)	Removal Efficiency of NH_3N (%) from RSM	Removal Efficiency of NH_3N (%) from Experiment
1	6	90	6	98.1	97.6

4. Conclusion

The efficacy of aeration in removing ammonia nitrogen from landfill leachate was investigated in this study. The results prove that air flow rate, aeration time and lime dosage are crucial factors in determining the efficiency of ammonia removal from leachate landfills. The optimization of the ammonia removal that has been done by using RSM with CCD focused on these three influencing factors (airflow rate, aeration time, and lime dosage). In addition, RSM was used to analyze the relationship between all of the components. The R^2 multiple correlation coefficient of determination recorded was 0.9914, indicating that the actual and predicted data were closely aligned. The optimum results from the model suggest that 90 min of contact time was needed to achieve 98.1% of NH_3N removal when the airflow rate and lime dosages were 6 L/min and 6g/L, respectively. According to this study, aeration can be used to remove ammonia nitrogen from landfill leachate efficiently.
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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Author H. Haslina, I. Norfatiha;* ***data collection:*** *Author Y;* ***analysis and interpretation of results:*** *J. NorRuwaida, M. Dewika, M.R. Ammar, Y.Y. Sara and N. Abdul Manaf;* ***draft manuscript preparation*** *H. Haslina, I. Norfatiha, J. NorRuwaida and Y.Y. Sara. All authors reviewed the results and approved the final version of the manuscript.*

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