

Application of response surface methodology (RSM) for optimization of semi-aerobic landfill leachate treatment using ozone

Salem S. Abu Amr · Hamidi Abdul Aziz ·
Mohammed J. K. Bashir

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Abstract The removal efficiencies for chemical oxygen demand (COD), ammoniacal nitrogen ($\text{NH}_3\text{-N}$), and color, as well as ozone consumption (OC) from the Malaysian semi-aerobic landfill stabilized leachate using ozone reactor, were investigated. Central composite design with response surface methodology was applied to evaluate the interaction and relationship between operating variables (i.e., ozone dosage, COD concentration, and reaction time) and to develop the optimum operating condition. Based on statistical analysis, Quadratic models for the four responses (COD, $\text{NH}_3\text{-N}$, color, and OC) proved to be significant with very low probability values (<0.0001). The obtained optimum conditions were 70 g/m^3 ozone, 250 mg/l COD, and 60 min reaction time. The results obtained by the predicted model were 26.7, 7.1, and 92 % removal for COD, $\text{NH}_3\text{-N}$, and color, respectively, with 9.42 ($\text{kgO}_3/\text{kg COD}$) OC. The predicted results fitted well with the results of the laboratory experiment.

Keywords Stabilized leachate · Ozonation · Optimization process · Response surface methodology (RSM)

Introduction

Sanitary landfill is recognized as the most common and desirable method for eliminating urban solid waste. It is also considered as the most economical and environmentally acceptable method for eliminating and disposing of municipal and industrial solid wastes (Tengrui et al. 2007). However, sanitary landfill generates a large amount of heavily polluted leachate (Zazouil and Yousefi 2008). The generation of leachate is mainly caused by a release from waste due to successive biological, chemical, and physical processes of waste deposited in a landfill. The quality and quantity of the water formed at landfills depend on several factors, including seasonal weather variations, land filling technique, phase sequencing, piling, and compaction method (Amonkrane et al. 1997; Trebouet et al. 2001).

Landfill leachate is a high-strength wastewater that is very difficult to deal with. Leachate generated from mature landfills (age >10 years) is typically characterized by large amounts of organic contaminants measured as chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), ammonia, halogenated hydrocarbons suspended solid, significant concentration of heavy metals, and many other hazardous chemicals identified as potential sources of ground and surface water contamination (Schrab et al. 1993; Christensen et al. 2001; Renou et al. 2008; Aziz et al. 2009; Foul et al. 2009). Moreover, the sequent migration of leachate away from landfill and its release into the environment are serious environmental pollution concerns, threatening public health and safety (Read et al. 2001). Accordingly, many environmental specialists are determined to find efficient treatments for large quantities of polluted leachate.

A number of leachate treatment techniques have been applied, which include biological, physical, and chemical processes (Baig and Liechti 2001; Goi et al. 2009). Given the

S. S. Abu Amr · H. A. Aziz (✉)
School of Civil Engineering, Engineering Campus, Universiti
Sains Malaysia, 14300, Nibong Tebal, Penang, Malaysia
e-mail: cehamidi@usm.my; cehamidi@yahoo.com

M. J. K. Bashir
Department of Environmental Engineering, Faculty
of Engineering and Green Technology, UniversitiTunku
Abdul Rahman, 31900 Kampar, Perak, Malaysia

oxidation efficiency, ozone has been suggested as one of the chemical processes used for the treatment of stabilized landfill leachate to reduce the risk of strength and un-biodegradable organics (Beaman et al. 1998). Ozonation processes are effective means for the treatment of landfill leachates due to the high oxidative power of ozone (Huang et al. 1993; Rice 1997; Haapea et al. 2002; Wu et al. 2004). During ozonation, the biodegradability of leachate will be enhanced due to the fragmentation of organic compounds with long chains to lower chains degraded to carbon dioxide (Geenens et al. 2001). The performance of ozone for removing COD and color from mature landfill leachate has been demonstrated in the literature (Rivas et al. 2003; Chaturapruek et al. 2005; Hagman et al. 2008; Goi et al. 2009; Cortez et al. 2011). However, none of these reports have evaluated the effects of different O_3 dosages for different concentrations of leachate during different reaction times.

In the present study, the statistical relationships among three independent factors (ozone dosage, COD concentration, and reaction time) for the treatment of semi-aerobic stabilized leachate were assessed through RSM. The RSM is a mathematical and statistical technique that is useful for the optimization of chemical reactions and industrial

processes and is commonly used for experimental designs. The main objectives of the present study include the following:

1. To investigate the efficiency of ozone for treating semi-aerobic stabilized leachate with different concentration levels.
2. To build up the equations of COD, ammoniacal nitrogen, and color removal efficiency from stabilized leachate and ozone consumption with respect to operational conditions [i.e., ozone dosage, reaction time, and COD concentration using RSM and central composite design (CCD)].
3. To determine the optimum operational condition of the studied application.

Materials and methods

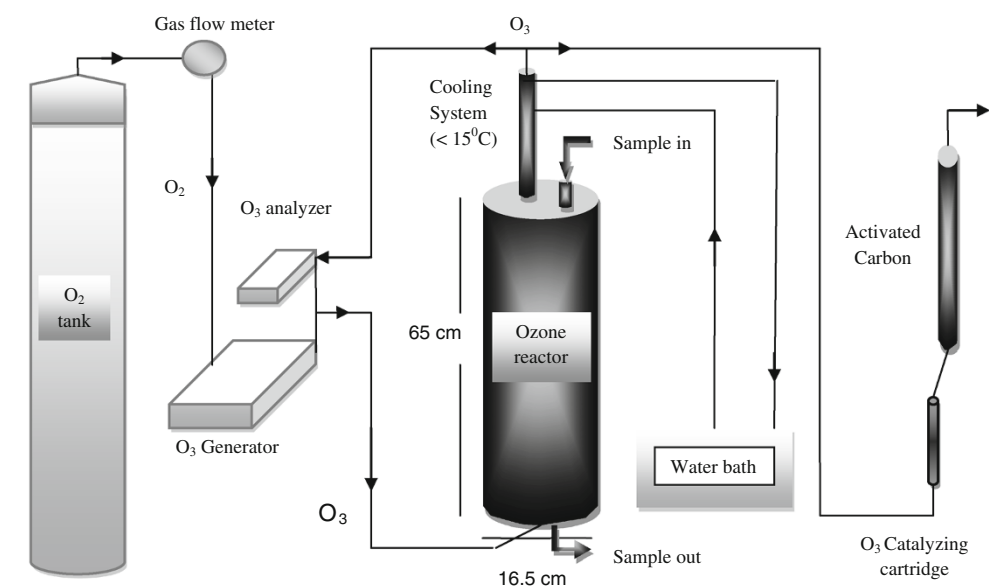
Leachate sampling and characteristics

The leachate samples used in the current study were collected from the aeration pond of a semi-aerobic stabilized leachate of the Pulau Burung landfill site (PBLs) in Nibong Tebal, Penang, Malaysia. The total landfill site area is 62.4 ha; however, only 33 ha are currently utilized to receive about 2,200 tons of solid waste daily (Bashir et al. 2011). This landfill produces a dark-colored liquid with pH level of more than 7.0, and is classified as stabilized leachate with high concentration of COD, NH_3-N , and low BOD/COD ratio (Aziz et al. 2007). All samples were collected manually in 20 l plastic containers, and then transferred, characterized, and refrigerated immediately in accordance with the Standard Methods for the Examination

Table 1 Characteristics of semi aerobic landfill leachate from PBLs

Parameters	Value
COD (mg/l)	2,000
NH_3-N (mg/l)	960
Color (PT Co.)	3,670
pH	8.5
Suspended solids (mg/l)	197
Conductivity ($\mu S/cm$)	16,650

Fig. 1 Schematic diagram of ozone equipment



of Water and Wastewater (APHA 2005). Table 1 shows some characteristics of the leachate sample.

Experimental procedures

All experiments were carried out in a 2-L volume of sample using an ozone reactor with a height of 65 cm and an inner diameter of 16.5 cm and supported by a cross-column ozone chamber for enhancing ozone gas diffusion (Fig. 1). Ozone was produced by a BMT 803 generator (BMT Messtechnik, Germany) fed with pure dry oxygen with recommended Gas flow rate of 100–1,000 ml/min under 1 bar pressure. Gas ozone concentration (in g/m³ NTP) was measured by an ultraviolet gas ozone analyzer (BMT 964). The water bath and cooling system supported the ozone reactor to keep the internal reaction temperature at <15 °C. The process variables include ozone dosage,

reaction time, and varied COD concentrations of leachate. Concentrations of COD, color, and ammonia were tested before and after each ozonation process, and the removal efficiency was then conducted. All tests were conducted according to the standard methods for the examination of water and wastewater (American Public Health Association (APHA) 2005). Ozone consumption (OC) in removing a certain amount of COD during ozonation under experimental conditions is given in the following Eq. (1):

$$OC = \frac{Q_G}{V} \times \frac{\int_0^t \left(1 - \frac{C_{AG}}{C_{AG0}}\right) dt}{(COD_0 - COD)}, \quad (1)$$

where Q_G is the gas flow rate (ml/min); V is the sample volume (ml); C_{AG} is the off-gas ozone concentration (g/m³); C_{AG0} is the input ozone concentration (g/m³); t is the time (min); and COD_0 and COD correspond to the initial and final COD (mg/l).

Experimental design and analysis

The Design Expert Software (version 6.0.7) was used for the statistical design of experiments and data analysis. In the present study, the CCD and response surface methodology (RSM) were applied to optimize and assess the relationship among three significant independent variables: (1) ozone

Table 2 Independent variables of the CCD design

Level of value	Ozone dosage (g/m ³)	COD concentration (mg/l)	Reaction time (min)
−1	30	250	10
0	55	1,125	35
+1	80	2,000	60

Table 3 Response values for different experimental conditions

Run no.	Factor A Ozone dosage (g/m ³)	Factor B COD concentration (mg/l)	Factor C Reaction time (min)	Response 1 COD removal (%)	Response 2 NH ₃ -N removal (%)	Response 3 Color removal (%)	Response 4 OC (kgO ₃ /kg COD)
1	80	250	60	27.2	8.5	90	19.40
2	55	1,125	35	18.8	1.1	31.8	3.62
3	30	250	10	16	0.0	25	3.44
4	55	2,000	35	21	0.0	24	1.80
5	80	2,000	10	10	0.0	18.5	2.04
6	55	250	35	24	6.5	72	7.72
7	55	1,125	35	17.5	1.2	32.5	3.41
8	80	2,000	60	15	0.0	27.3	6.96
9	55	1,125	35	18	1.1	33	3.33
10	55	1,125	35	18.5	1.2	32	3.41
11	30	2,000	10	4	0.0	11	1.80
12	55	1,125	35	17	0.9	31	3.70
13	30	2,000	60	11	0.0	23	2.09
14	55	1,125	10	15.5	0.0	16	1.60
15	30	1,125	35	12.5	1.0	38	5.15
16	80	250	10	15	4.7	45	9.47
17	55	1,125	35	17.5	1.2	33.6	4.50
18	30	250	60	20.8	2.0	88	4.72
19	55	1,125	60	22	1.4	58	3.18
20	80	1,125	35	19	2.2	31	6.09

dosage, (2) reaction time, and (3) COD concentrations in leachate as presented in Table 2. COD, color, and $\text{NH}_3\text{-N}$ removal were considered as the dependent factors (response). Performance of the process was evaluated by analyzing the COD, color, and $\text{NH}_3\text{-N}$ removal efficiencies. Each independent variable was varied over three levels between -1 and $+1$ at the determined ranges based on a set of preliminary experiments. The total number of experiments obtained for the three factors was $20 (=2^k + 2k + 6)$, where k is the number of factors ($k = 3$). Fourteen experiments were enhanced with 6 replications to assess the pure error. Considering that there are only three levels for each factor, the appropriate model is the quadratic model Eq. (2).

$$Y = \beta_0 + \sum_{j=1}^k \beta_j X_j + \sum_{j=1}^k \beta_{jj} X_j^2 + \sum_i \sum_{<j=2}^k \beta_{ij} X_i X_j + e_i \quad (2)$$

where Y is the response; X_i and X_j are the variables; β is the regression coefficient; k is the number of factors studied and optimized in the experiment; and e is the random error.

Analysis of variance (ANOVA) was used for graphical analyses of the data to obtain the interaction between the process variables and the responses. The quality of the fit polynomial model was expressed by the value of correlation coefficient (R^2), and its statistical significance was checked by the F test in the same program. Model terms

Table 4 ANOVA for analysis of variance and adequacy of the quadratic model for COD, $\text{NH}_3\text{-N}$, and Color removal and OC

	Source	Sum of squares	Degree of freedom	Mean square	F value	Prob > F
COD	Model	427.61	4	106.90	20.73	<0.0001
	A	47.96	1	47.96	9.30	0.0081
	B	176.40	1	176.40	34.21	<0.0001
	C	126.03	1	126.03	24.44	0.0002
	A^2	77.22	1	77.22	14.97	0.0015
	Residual	77.35	15	5.16		
	Lack of fit	75.05	10	7.50	16.26	0.0033
	Pure error	2.31	5	0.46		
	Model	95.79	7	13.68	28.82	<0.0001
	A	15.38	1	15.38	32.38	0.0001
$\text{NH}_3\text{-N}$	B	47.09	1	47.09	99.17	<0.0001
	C	5.18	1	5.18	10.92	0.0063
	B^2	8.26	1	8.26	17.39	0.0013
	C^2	2.85	1	2.85	6.00	0.0306
	AB	15.68	1	15.68	33.02	<0.0001
	BC	4.20	1	4.20	8.86	0.0116
	Residual	5.70	12	0.47		
	Lack of fit	5.63	7	0.80	58.85	0.0002
	Pure error	0.068	5	0.014		
	Model	95.79	7	13.68	28.82	<0.0001
Color	Model	8,919.57	4	2,229.89	77.04	<0.0001
	B	4,674.24	1	4,674.24	161.48	<0.0001
	C	2,917.26	1	2,917.26	100.78	<0.0001
	B^2	377.58	1	377.58	13.04	0.0026
	BC	950.48	1	950.48	32.84	<0.0001
	Residual	434.20	15	28.95		
	Lack of fit	429.95	10	42.99	50.60	0.0002
	Pure error	4.25	5	0.85		
	Model	161.97	2	80.99	9.54	<0.0017
	A	71.61	1	71.61	8.44	0.0099
OC	B	90.36	1	90.36	10.65	0.0046
	Residual	144.27	17	8.49		
	Lack of fit	143.33	12	11.94	63.35	<0.0001
	Pure error	0.94	5	0.19		

COD: SD = 2.27,
PRESS = 148.07, $R^2 = 0.8468$,
Adj $R^2 = 0.8060$, Adeq
precision = 17.508

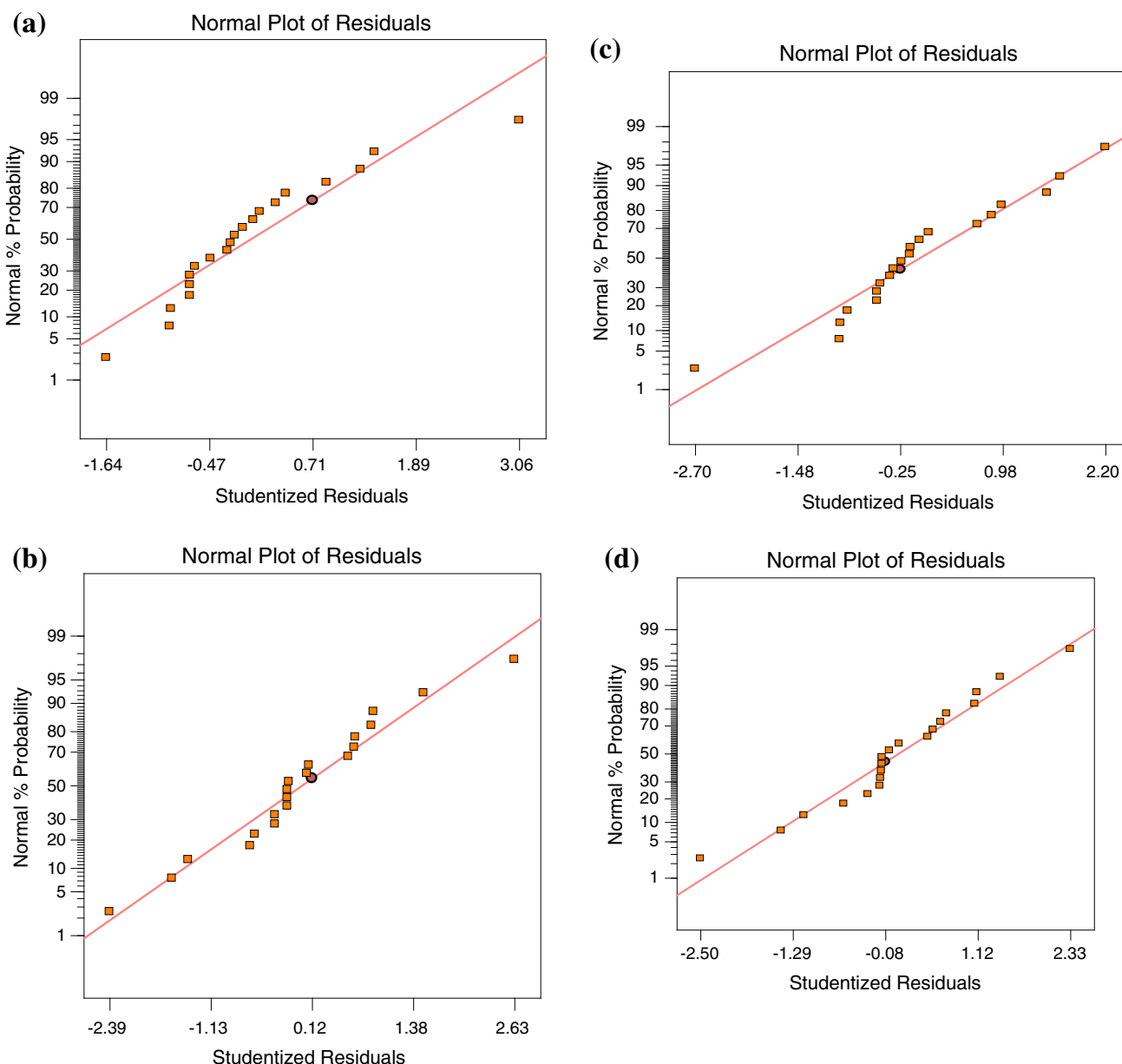
$\text{NH}_3\text{-N}$: SD = 0.69,
PRESS = 24.12, $R^2 = 0.9439$,
Adj $R^2 = 0.9111$, Adeq
precision = 19.710

Color: SD = 5.38,
PRESS = 1,053.30,
 $R^2 = 0.9536$, Adj $R^2 = 0.9412$,
Adeq Precision = 28.772

OC: SD = 2.91,
PRESS = 236.61, $R^2 = 0.5289$,
Adj $R^2 = 0.4735$, Adeq
Precision = 10.072

Table 5 Final equations in terms of coded and actual factors for parameters

	Final equation in terms of coded factors	Final equation in terms of actual factors
COD removal (%)	$+18.98 + 2.19 A - 4.20 B + 3.55 C - 3.93 A^2$	$-4.42920 + 0.77928 A - 4.80000E-003 B + 0.14200 C + 6.28800E-003 A^2$
NH ₃ -N removal (%)	$+1.32 + 1.24 A - 2.17 B + 0.72 C + 1.61 B^2 - 0.94 C^2 - 1.40 A B - 0.72 BC$	$-4.08677 + 0.12160 A - 2.52041E-003 B + 0.17179 C - 2.09796E-006 B^2 - 1.51000E-003 C^2 - 6.40000E-005 A B - 3.31429E-005 BC$
Color removal (%)	$+33.69 - 21.62 B + 17.08 C + 8.69 B^2 - 10.90 BC$	$+32.32024 - 0.032807 B + 1.24377 C + 1.13502E-005 B^2 - 4.98286E-004 BC$
OC (kgO ₃ /kg COD)	$+4.87 + 2.68 A + 3.01 B$	$+2.84916 + 0.10704 A + 3.43543E-003 C$

**Fig. 2** Design expert plot; normal probability plot of the standardized residual for **a** COD, **b** NH₃-N, **c** color removal and **d** OC

were evaluated by the P value (probability) with 95 % confidence level.

Results and discussion

There were a total of 20 runs of the CCD experimental design, and the results are shown in Table 3. The observed percent removal efficiencies varied between 4 and 27.2 % for COD, 0–8.5 % for $\text{NH}_3\text{-N}$, and 11–90 % for color. Several researchers have conducted studies on the treatment of mature landfill leachate using ozone. Tizaoui et al. (2007) obtained 27 and 87 % removal for COD and color, respectively, after 60 min Ozonation of raw leachate. In the same way, Hagman et al. (2008) obtained 22 % COD reduction. Rivas et al. (2003) obtained a 30 % depletion of COD. Accordingly, the efficiency of ozone technique for solely removing organics and ammonia from leachate is

relatively weak; the technique is more efficient for color removal, which may be attributed to the strength of organic components in leachate, improving the removal efficiency in lower initial COD concentration as shown in Table 3. Thus, many researchers have employed several advanced oxidation agents and techniques to improve the efficiency of ozone for leachate treatment, such as hydrogen peroxide (H_2O_2) and UV (Wu et al. 2004; Tizaoui et al. 2007). Other experiments have used lower pH and adsorbent materials, such as activated carbon, to enhance the removal of ammonia from leachate during ozonation (Park and Jin 2005).

Ozone consumption was also calculated under conditions of each run by following Eq. (1), and ranged from 1.6 to 19.40 ($\text{kgO}_3/\text{kg COD}$). OC is defined as the amount of ozone gas consumed for removing a certain amount of COD during ozonation under experimental conditions. OC value increased at minimum reaction time (10 min) and

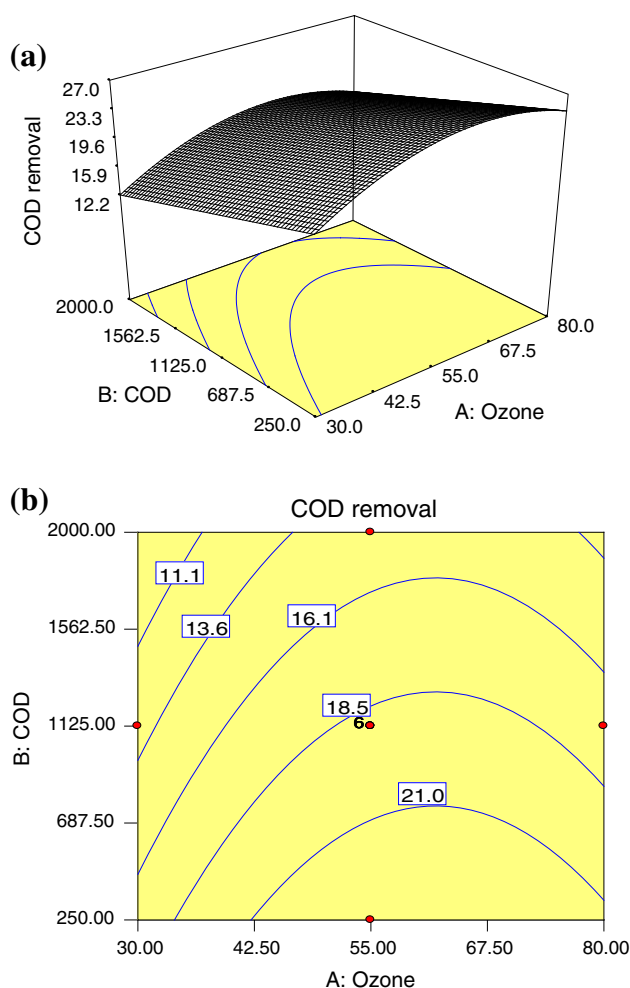


Fig. 3 Response surface (a) and contour plots (b) for COD removal efficiency as a function of ozone dosage, (30 g/m^3), COD concentration, (250 mg/l) and reaction time, (60) min

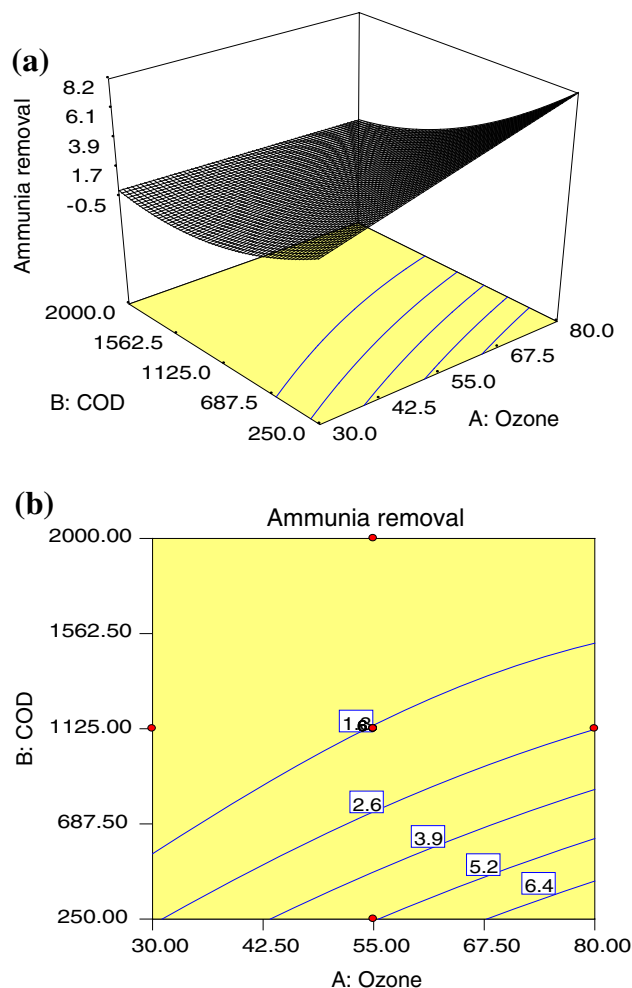


Fig. 4 Response surface (a) and contour plots (b) for $\text{NH}_3\text{-N}$ removal efficiency as a function of ozone dosage, (30 g/m^3), COD concentration, (250 mg/l) and reaction time, (60) min

maximum initial COD concentration (2,000 mg/l). This result suggests that ozone running at maximum time (60 min) will reduce the amount of OC compared with an improved COD removal efficiency. Several experiments with ozone consumption values have been conducted from less than 1 kgO₃/kg COD (Ho et al. 1974), 0.63 kgO₃/kg COD (Abu Amr and Aziz 2012) 3.5 kgO₃/kg COD (Tizaoui et al. 2007), up to 16 kgO₃/kg COD (Wang et al. 2003), and between 2 and 3 for ozone alone systems (Geissen 2005).

Analysis of variance

Table 4 present the ANOVA of regression parameters of the predicted response surface quadratic models and other statistical parameters for COD, NH₃-N, color removal, and OC. Data given in these tables demonstrate that all the models were significant at the 5 % confidence level, given that *P* values were less than 0.05. The values of correlation

coefficient ($R^2 = 0.8468, 0.9439, 0.9536$, and 0.8949) obtained in the present study for COD, NH₃-N, color removal, and OC were higher than 0.80. For a good fit of model, the correlation coefficient should be at a minimum of 0.80. A high R^2 value close to 1 illustrates good agreement between the calculated and observed results within the range of experiment and shows that a desirable and reasonable agreement with adjusted R^2 is necessary (Joglekar and May 1987; Nordin et al. 2004). The “Adequate Precision” ratio of the models varies between 16.214 and 28.772, which is an adequate signal for the model. AP values higher than 4 are desirable and confirm that the predicted models can be used to navigate the space defined by the CCD.

In the current study, four quadratic models are significant model terms (Table 4). Insignificant model terms, which have limited influence, were excluded from the study to improve the models. Based on the results, the response surface models constructed for predicting COD, NH₃-N, color removal efficiency, and OC were considered reasonable.

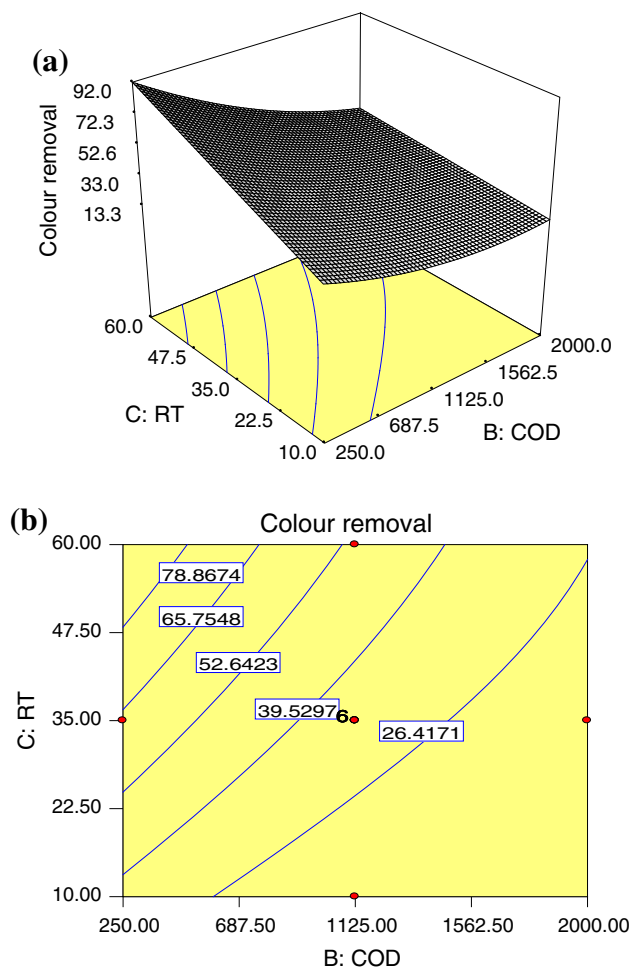


Fig. 5 Response surface (a) and contour plots (b) for color removal efficiency as a function of ozone dosage, (30 g/m³), COD concentration, (250 mg/l) and reaction time, (60) min

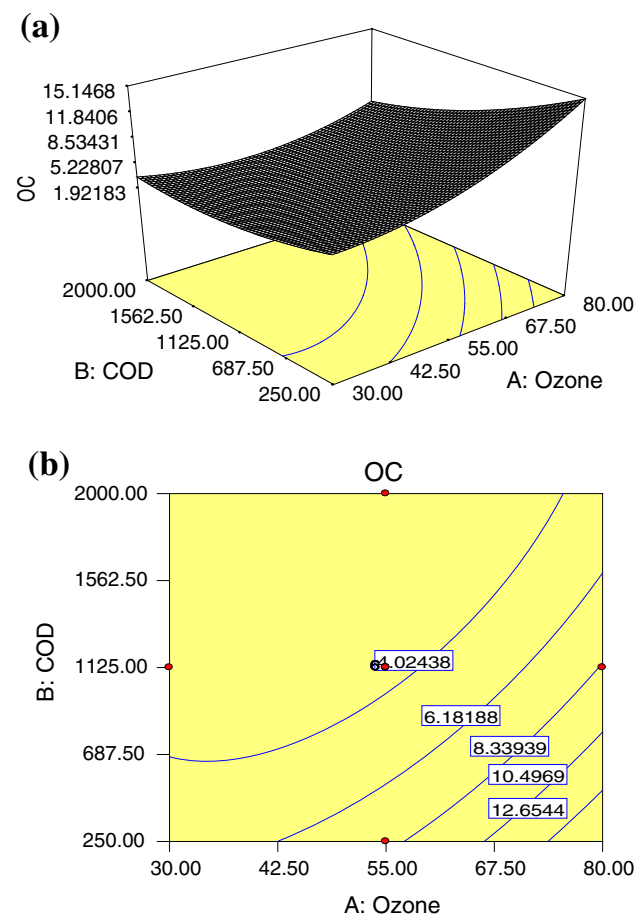


Fig. 6 Response surface (a) and contour plots (b) for OC as a function of ozone dosage, (30 g/m³), COD concentration, (250 mg/l) and reaction time, (60) min

Table 6 Optimization results for COD, NH₃-N Color maximum removal efficiency and minimum amount of OC

NO	Ozone g/m ³	COD mg/l	RT min	COD removal (%)	Ammonia removal (%)	Colour removal (%)	OC (kgO ₃ /kg COD)	Desirability
1	70	250	60	26.7	7.1	92	9.42	0.823
Lab. experiment				24.7	6.4	90.8	9.5	

RT reaction time

The final regression models, in terms of their coded and actual factors, are presented in Table 5. To confirm if the selected model provides an adequate approximation of the real system, the normal probability plots of the studentized residuals and diagnostics are provided by the Design Expert 6.0.7 software. The normal probability plots that helped us judge the models (Fig. 2a–c) demonstrate the normal probability plots of the standardized residuals for COD, NH₃-N, color removal, and OC. A normal probability plot indicates that if the residuals follow a normal distribution, as shown in Fig. 1, the points will follow a straight line for each case. However, some scattering is expected even with the normal data. Accordingly, the data can be possibly considered as normally distributed in the responses of certain models.

Treatment efficiency

To assess the interactive relationships between independent variables and the responses of certain models, the 3D surface response and contour plots utilized the Design Expert 6.0.7 software (Figs. 3, 4, 5, 6). As shown in Figs. 3a and 4a, the maximum observed removal of COD and NH₃-N were 27 and 8.2 %, respectively, at ozone dosage 80 g/m³ and COD concentration 2,000 mg/l. The contour plots demonstrate that the improvement of removal efficiencies for COD and NH₃-N is attributed to the decrease in COD concentration and increase in ozone dosage (Figs. 3b, 4b). The maximum removal of color was 92 % at ozone dosage 80 g/m³ and 60 min reaction time (Fig. 5a). The increase in reaction time and decrease in COD are the two main factors for improving color removal (Fig. 5b). Based on the target of OC as a minimum value, Fig. 6 shows the response and contour plot for the amount of ozone gas consumption for COD reduction-based cretin-independent variables; a minimum value of OC will follow the increase in reaction time and ozone dosage.

Optimization process

The optimization process was carried out to determine the optimum value of COD, NH₃-N, and color removal efficiency, in addition to OC for COD removal using the

Design Expert 6.0.7 software. According to the software optimization step, the desired goal for each operational condition (ozone dosage, COD concentration, reaction time) was chosen “within” the range. The responses (COD, NH₃-N, and color) were defined as maximum to achieve the highest performance, whereas the OC response was defined as the minimum to achieve the lowest value of ozone Gas consumed for removing the highest amount of COD. The program combines the individual desirabilities into a single number and then searches to optimize this function based on the response goal. Accordingly, the optimum working conditions and respective percent removal efficiencies were established, and the results are presented in Table 6. As shown in Table 6, 26.7, 7.1, and 92 % removal of COD, NH₃-N, and color are predicted, respectively, whereas OC is presented as 9.40 (kgO₃/kg COD) based on the model under optimized operational conditions (ozone dosage 70 g/m³; COD concentration 250 mg/l; and reaction time 60 min). The desirability function value was found to be 0.823 for these optimum conditions. An additional experiment was then performed to confirm the optimum results. The laboratory experiment agrees well with the predicted response value.

Conclusion

Using ozone, the optimization of semi-aerobic stabilized landfill leachate treatment was investigated. The interaction between operational variables for the treatment optimization process, such as ozone dosage, COD concentration, and reaction time, was applied using RSM with CCD. Statistical analysis for the interaction of models' responses (COD, NH₃-N, color removal, and OC) was significant at *P* value less than 0.05. The optimum operational condition obtained 80 g/m³ of ozone gas applied on leachate with 250 mg/l COD concentration during 60 min reaction time was required to achieve 26.7, 7.1, and 92 % removal for COD, NH₃-N, and color, respectively. OC value (9.40 kgO₃/kg COD) was also obtained.

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