Acta Chemica Malaysia (ACMY)

DOI : http://doi.org/10.26480/acmy.02.2017.11.17

ISSN: 2576-6732 (Print)
ISSN: 2576-6724(Online)
CODEN: ACMCCG

RESEARCH ARTICLE

THE EFFECT OF COMBINED AL_2SO_4 AND PERSULFATE ON COD, COLOR AND NH_3–H REMOVAL FROM LEACHATE

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ARTICLE DETAILS

Article History:

Received 28 November 2017
Accepted 29 December 2017
Available online 1 January 2018

ABSTRACT

In this paper, Al_2SO_4 was added to persulfate as an activator to increase reaction and to help insure oxidation during the leachate oxidation process. The effect of Al_2SO_4, persulfate dosage, pH and reaction time on COD, color and ammonia removal was investigated during landfill leachate treatment. A face centered composite design (FCCD) was employed to model and optimize the process. The results of the analysis of variance (ANOVA) showed that Al_2SO_4 has a significant effect on the selected responses. Furthermore, a second order model was selected to describe the relationship between input factors and the three responses. The maximum removal for COD, color and NH_3-N was reported as 67%, 81%, and 48%, respectively, by using 5.5 ml from each persulfate and Al_2SO_4 dosage (0.2 M), 6 pH, and 105 (min) reaction time. The performance of combined $S_2O_8^{2-}$/Al_2SO_4 process compared with $S_2O_8^{2-}$ alone and Al_2SO_4 alone, and the results showed that the combined method achieved a higher removal efficiency for COD, color, and NH_3–N.

KEYWORDS

Al_2SO_4, persulfate, oxidation, removal, face centered composite design (FCCD), optimization.

1.INTRODUCTION

Increesing of population growth and industries led to increase solid waste generation from residential areas. Alghtough landfilling is still considerrign the most common and suitable method for municipal solid waste (MSW) desposal [1]. Large quantity of leachate generated from landfilling may cause of significant environmental concern [2]. Leachate contains high level organic and inorganic pollutant, such as ammonia and heavy metals which is directly affect to the surface and groundwater and may affect human health and aquatic environment. Leachate pollution in Malaysia is very serious, and generation of large quantity of this type of leachate in tropical areas such as Malaysia is mainly attributed to the high amount of rainfall [3]. Suitable and effective treatment for leachate is requires before final discharge [4]. Recently, the number of leachate treatment applications have been applied [5-10] .

Advanced oxidation processes (AOPs) recently received attraction as an efficient methods for reducing the high organic level in leachate. persulfate reagents recently received attraction in removing organics from wastewater and landfill leachate. Although persulfate can act as a direct oxidant, the performance can be improved when applying different activation processes on persulfate reagent to initiate sulfate radicals. Persulfate oxidation works by releasing sulfate radicals that have powerful effects on the oxidation of organics [11, 12]. Generation of sulfate radicals during persulfate oxidation can be significantly enhanced by catalysts, namely, heat, UV radiation, ozone, H_2O_2, and high pH. [13–16]. Rostagy employed ferrous peroxymonosulfate oxidative to generate sulfite radicals from persulfate for PCBs degradation in aqueous solution [17].

Al_2SO_4 reagent is widely used for wastewater treatment, however, the performance utilize Al_2SO_4 in activating persulfate for leachate treatment has not been investigated. The relationships between various parameters (interactions) for each advanced oxidation process and optimization have not been well studied. Thus, response surface methodology (RSM) was used to evaluate the statistical relationship between experimental variables and response (COD, color and NH_3-H removal). In this work, experimental operational conditions are optimized, and quadratic model equations for COD, color and NH_3-H removal are provided. Moreover, the performance of simultaneous persulfate/ Al_2SO_4 was compared with persulfate alone and Al_2SO_4 alone.

2.EXPERIMENTAL DESIGN, MATERIALS AND METHODS

2.1.Leachate Sampling and Characteristics

Leachate samples were collected from the detention pond at Sungai Udang Landfill Site (SULS), Melaka, Malaysia. SULS has an area of 7 ha, receiving approximately 1200 tons of municipal solid waste daily and start receiving waste at 1st of April 2015. In this study, the leachate samples were collected 6 times manually from February 2017 to Jun 2017 using 2 L plastic containers. The collected samples were immediately transported to the laboratory, characterized, and stored in cool room to 4 °C. The general characteristics of the leachate used in the study are presented in Table 1. All samples were collected, preserved and analysed by following Standard Methods for the Examination of Water and Wastewater [18].

Table 1: Characteristics of Sungai Udang landfill leachate

Parameters	Value*
COD (mg/L)	2300
BOD (mg/L)	110
NH_3-N (mg/L)	870
Color (PT Co.)	4800
pH	8.6
Suspended solids (mg/L)	88
Conductivity, (µS/cm)	18,940

*Average of two samples taken from March and June 2017.

2.2. Experimental Procedures

In the current study, Sodium persulfate ($Na_2S_2O_8$ M = 238 g/mol) and Aluminum sulfate (Al_2SO_4 342.15 g/mol) were used for advanced oxidation during the oxidation of leachate samples. Several dosages of S_2O_8 and Al_2SO_4 were gradually mixed with 100 mL of leachate samples to determine the optimum $S_2O_8^{2-}$ and Al_2SO_4 dosage according to the efficiencies of COD, Color and NH_3–N removal. Orbital Shaker (Luckham R100/TW Rotatable Shaker 340 mm X 245 mm) with at 200 rpm was used for samples shaking [10]. All experiments were performed at room temperature (28 0C) using 100 mL leachate samples in conical flasks with a 250 mL capacity. pH of the samples was controlled by using 3 M sulphuric acid solution and 3 M sodium hydroxide solution [9]. All experiments were performed at laboratory of Malaysian Institute of chemical & Bioengineering Technology, University of Kuala Lumpur, Melaka, Malaysia.

2.3. Experimental design

The effect of four factors, namely persulfate dosage ($X_{1)}$, Al_2SO_4 dosage (X_2), pH (X_3) and reaction time (X_4) on three responses COD (Y_1), color (Y_2) and ammonia (Y_3) removal efficiencies from leachate was studied. The relationship between the factors and the three responses was modelled and optimized by using face centred composite design (FCCD). FCCD is one of the frequently used design in response surface methodology (RSM) to model and optimize the relationship between the input factors and the output responses. The levels of selected factors were chosen based on literature and preliminary experiments, the actual and coded levels are given in Table 2.

Table 2: Independent variables (factors) and corresponding levels used for optimization

Variables	Symbol	Range and levels		
		Low level (-1)	Center (0)	High level +1
Persulfate dosage	X_1	1 ml	5.5 ml	10 ml
$Al_2(SO_4)3$ dosage	X_2	1 ml	5.5 ml	10 ml
pH	X_3	3	6	9
Reaction time	X_4	30	105	180

The relationship between the selected factors (X_1, X_2, X_3, X_4)and each of the responses ($Y_1, Y_2,\ Y_3$).is usually described in response surface methodology (RSM) by a second-order polynomial as given in Eq. (1).

$$Y = \beta_0 + \sum_{i=1}^{4}\beta_i X_i + \sum_i^4 \beta_{ii} X_i^2 + \sum\sum_{i<j}\beta_{ij}X_{ij} \quad (1)$$

where Y represents the dependent variable, β_0, β_i and $\beta_{ii}$are linear coefficient, quadratic coefficient and interaction coefficients respectively, need to be estimated, and X_i represents the independent variables.

Thirty runs are required for FCCD to cover all possible combination of X_1, X_2, X_3, and X_4 distributed as follows: sixteen runs for the factorial design, eight runs are for axial (star) points and six runs at the center of the design [19,20]. To avoid or minimize the effect of unexpected variability in the responses, thee experiments were run in random order. The data for the thirty-run of FCCD with the coded and actual levels of the four factors are given in Table 3.

2.4. Analytical Methods

COD, color and NH_3–N, were immediately tested before and after each experiment. Leachate sample was shacked well analyzed. NH_3–N concentration was measured by the Phenol Method No. (4500) using a UV-VIS spectrophotometer at 640 nm with a light path of 1 cm or greater. pH was measured using a portable digital pH/Mv meter. COD concentration was determined by the open reflux method No. (5220). The test values are presented as the average of the three measurements, and the difference between the measurements of each value was less than 3%. The removal efficiencies of COD and NH3–N were obtained using the following equation (2):

$$\text{Removal (\%)} = [(Ci - Cf) / Ci] \times 100 \quad (2)$$

where Ci and Cf refer to the initial and final COD, color and NH_3–N concentrations respectively.

3. RESULTS AND DISCUSSION

Four independent variables (factors), namely; persulfate dosage (X_1), Al_2SO_4 (X_2), pH (X_3), and reaction time (X_4) were identified to be influential in removing chemical Oxygen COD, color and NH_3-N from landfill leachate. Thirty-runs were performed using the FCCD; the interactions between the four experimental variables were considered in each run to evaluate the validity of leachate treatment using simultaneous $S_2O_8^{2-}$ and Al_2SO_4 oxidation process. Al_2SO_4 reagent was used to activate $S_2O_8^{2-}$ during the oxidation of the leachate. As shown in Table 3, the removal efficiencies ranged between 38% to 68% for COD, 75% to 81% for color and 18% to 47% for ammonia.

Activation of persufate under the effect of pH was discussed by Furman [21]. Sulfate radical can be initiated at high pH (Eq. 6), as shown in table 3, the maximum removal for COD was achieved at shorter reaction time (30 min) under low pH value (3) [22]. Deng and Ezyske and Mohajeri obtained higher removal of COD from landfill leachate at lower pH value, but the higher removal for ammonia can be obtained by persulfate oxidation at high pH [23,24].

$$S_2O_8^{2-} + OH^{\cdot} \rightarrow HSO_4^{-} + SO_4^{-} + 1/2\ O_2 \quad (3)$$

The results of thirty-run obtained from face-centered composite design (FCCD) were analyzed using analysis of variance (ANOVA) to investigate the relationship between the four independent variables (factors) and the three responses (COD removal, Color removal, and NH_3-N) and presented in Table 4. The effect of persulfate dosage (X_1) was significant (p<0.0001). Persulfate and pH showed a significant quadratic effect on COD removal (p< 0.0047 and 0.0001 respectively). Two-factor interaction between X_1 and X_2, X_2 and X_3, X_2 and X_4 was significant as presented in Table 4. Significant interaction was exhibited between persulfate and pH, and between Al_2SO_4 and pH while other two-factor interactions were insignificant (Table 4).

Table 3: The results of FFCD including coded and actual variable with the results of three responses (Color, COD, NH3 removals)

Coded variable				Actual variable				Responses		
Al2SO4	Persulfate	pH	RT	AlSO4	Persulfate	pH	RT	Color removal	COD removal	NH3 removal
-1	0	0	0	1	5.5	6	105	69	67.3	47.8
0	0	0	0	5.5	5.5	6	105	69.53	51.4	35.6
-1	1	-1	-1	1	10	3	30	70.6	47.9	34.33
-1	-1	1	-1	1	1	9	30	64.24	54.5	31.2
0	-1	0	0	5.5	1	6	105	56.8	39.78	31.07

0	0	0	-1	5.5	5.5	6	30	72.24	55.6	26.78
1	1	-1	-1	10	10	3	30	77.53	61.6	44.33
1	-1	-1	-1	10	1	3	30	63.31	50.9	42.22
0	0	0	1	5.5	5.5	6	180	77.87	48.8	25.4
1	0	0	0	10	5.5	6	105	73.67	67	38.56
-1	-1	-1	1	1	1	3	180	71	50.7	28.67
1	-1	-1	1	10	1	3	180	69.97	38	19.13
-1	1	1	1	1	10	9	180	75.54	47.5	19.33
-1	1	-1	1	1	10	3	180	74.67	38.5	21.2
1	-1	1	1	10	1	9	180	68.45	31.7	19.73
0	1	0	0	5.5	10	6	105	67.34	47.89	26.78
1	1	-1	1	10	10	3	180	81.66	40.92	20.53
1	1	1	1	10	10	9	180	70.27	42.2	18.76
-1	1	1	-1	1	10	9	30	67.02	39.7	19.67
0	0	0	0	5.5	5.5	6	105	69.53	56.76	35.57
0	0	1	0	5.5	5.5	9	105	75.89	33.67	32.47
0	0	-1	0	5.5	5.5	3	105	80.22	46.8	32.33
1	1	1	-1	10	10	9	30	68.45	42.2	25.53
0	0	0	0	5.5	5.5	6	105	69.23	55.37	35.56
-1	-1	-1	-1	1	1	3	30	57.83	47.6	25.87
1	-1	1	-1	10	1	9	30	68.98	40.4	22.33
0	0	0	0	5.5	5.5	6	105	69.53	57.32	35.55
0	0	0	0	5.5	5.5	6	105	74.98	56.8	35.45
-1	-1	1	1	1	1	9	180	79.78	60.2	28.93
0	0	0	0	5.5	5.5	6	105	72.62	51.6	29.23

Table 4: The results of ANOVA for color, COD and NH3–N removals

Color removal %	(R-square = 0.904)				
Source	Sum of Squares	DF	Mean Square	F Value	P-value F
Model	896.92	14	64.07	10.09	< 0.0001
X_1	8.83	1	8.83	1.39	0.2565
X_2	154.41	1	154.41	24.32	0.0002
X_3	3.71	1	3.71	0.58	0.4565
X_4	193.45	1	193.45	30.47	< 0.0001
X_1^2	0.58	1	0.58	0.091	0.7665
X_2^2	245.70	1	245.70	38.71	< 0.0001
X_3^2	101.10	1	101.10	15.93	0.0012
X_4^2	27.31	1	27.31	4.30	0.0557
X_1X_2	9.33	1	9.33	1.47	0.2441
X_1X_3	51.84	1	51.84	8.17	0.0120
X_1X_4	53.36	1	53.36	8.41	0.0110
X_2X_3	113.00	1	113.00	17.80	0.0007
X_2X_4	16.61	1	16.61	2.62	0.1266
X_3X_4	0.45	1	0.45	0.071	0.7939
Residual	95.22	15	6.35		
Lack of Fit	67.20	10	6.72	1.20	0.4458
Pure Error	28.02	5	5.60		
Total	992.14	29			

COD removal %	(R-square = 0.901)				
Source	Sum of Squares	DF	Mean Square	F Value	P-value
Model	2206.30	14	157.59	9.82	< 0.0001
X_1	84.41	1	84.41	5.26	0.0367
X_2	1.60	1	1.60	0.100	0.7564
X_3	52.87	1	52.87	3.29	0.0896
X_4	97.44	1	97.44	6.07	0.0263
X_1^2	527.70	1	527.70	32.88	< 0.0001
X_2^2	211.90	1	211.90	13.20	0.0025
X_3^2	414.18	1	414.18	25.80	0.0001
X_4^2	1.19	1	1.19	0.074	0.7889
X_1X_2	266.67	1	266.67	16.61	0.0010
X_1X_3	169.78	1	169.78	10.58	0.0054
X_1X_4	153.02	1	153.02	9.53	0.0075
X_2X_3	17.89	1	17.89	1.11	0.3078

Source	Sum of Squares	DF	Mean Square	F Value	P-value
X_2X_4	5.62	1	5.62	0.35	0.5630
X_3X_4	124.77	1	124.77	7.77	0.0138
Residual	240.77	15	16.05		
Lack of Fit	204.48	10	20.45	2.82	0.1322
Pure Error	36.28	5	7.26		
Total	2447.07	29			

NH3-N removal % (R-square = 0.86)

Source	Sum of Squares	DF	Mean Square	F Value	P-value
Model	1557.91	14	111.28	6.70	0.0004
X_1	1.92	1	1.92	0.12	0.7385
X_2	19.41	1	19.41	1.17	0.2967
X_3	142.58	1	142.58	8.59	0.0103
X_4	276.75	1	276.75	16.67	0.0010
X_1^2	186.08	1	186.08	11.21	0.0044
X_2^2	86.57	1	86.57	5.21	0.0374
X_3^2	13.77	1	13.77	0.83	0.3769
X_4^2	192.30	1	192.30	11.58	0.0039
X_1X_2	41.86	1	41.86	2.52	0.1332
X_1X_3	52.27	1	52.27	3.15	0.0963
X_1X_4	117.29	1	117.29	7.06	0.0179
X_2X_3	34.22	1	34.22	2.06	0.1716
X_2X_4	22.28	1	22.28	1.34	0.2648
X_3X_4	127.92	1	127.92	7.70	0.0141
Residual	249.07	15	16.60		
Lack of Fit	215.81	10	21.58	3.24	0.1030
Pure Error	33.26	5	6.65		
Total	1806.98	29			

The normal probability plots for COD, color and NH_3–N removal is demonstrated in Figure 1. General most of the points following the straight line for COD color and ammonia. A significant interaction between persulfate and Al_2SO_4 is presented in Fig. 2, showing the effect of the combination of the two reagents on color removal which is higher than the effect on COD removal.

The mathematical equation model was established to understand the behavior of relationship between the independent variables (factors) and responses to investigate the ability of optimizing the process. A second-order polynomial model was found as the best model that describe the relationship between the independent variables and the responses.

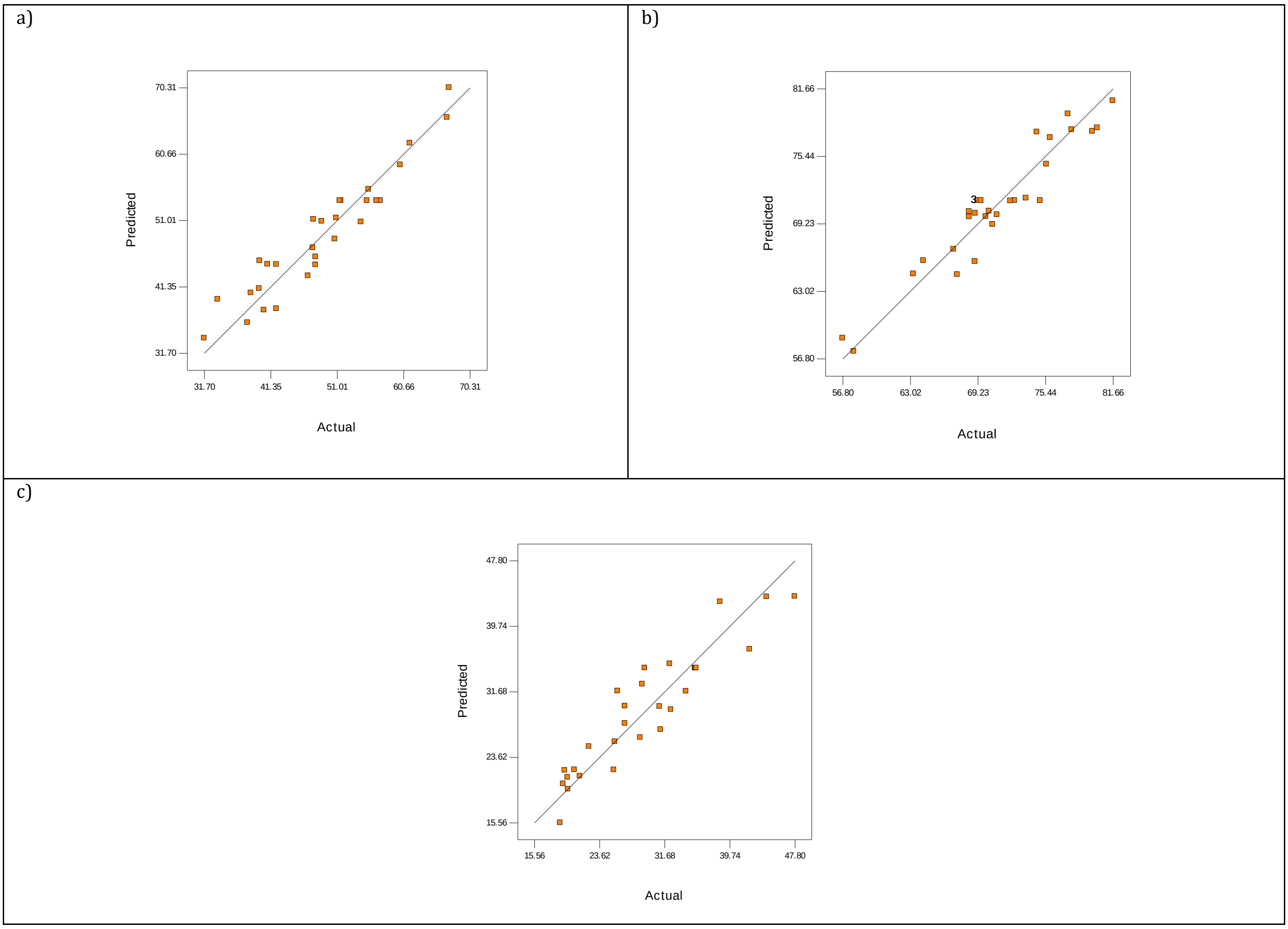

Figure 1: Design Expert plot; Predicted and actual standardized residual for (A) COD, (B) color (C) NH_3–N, removal

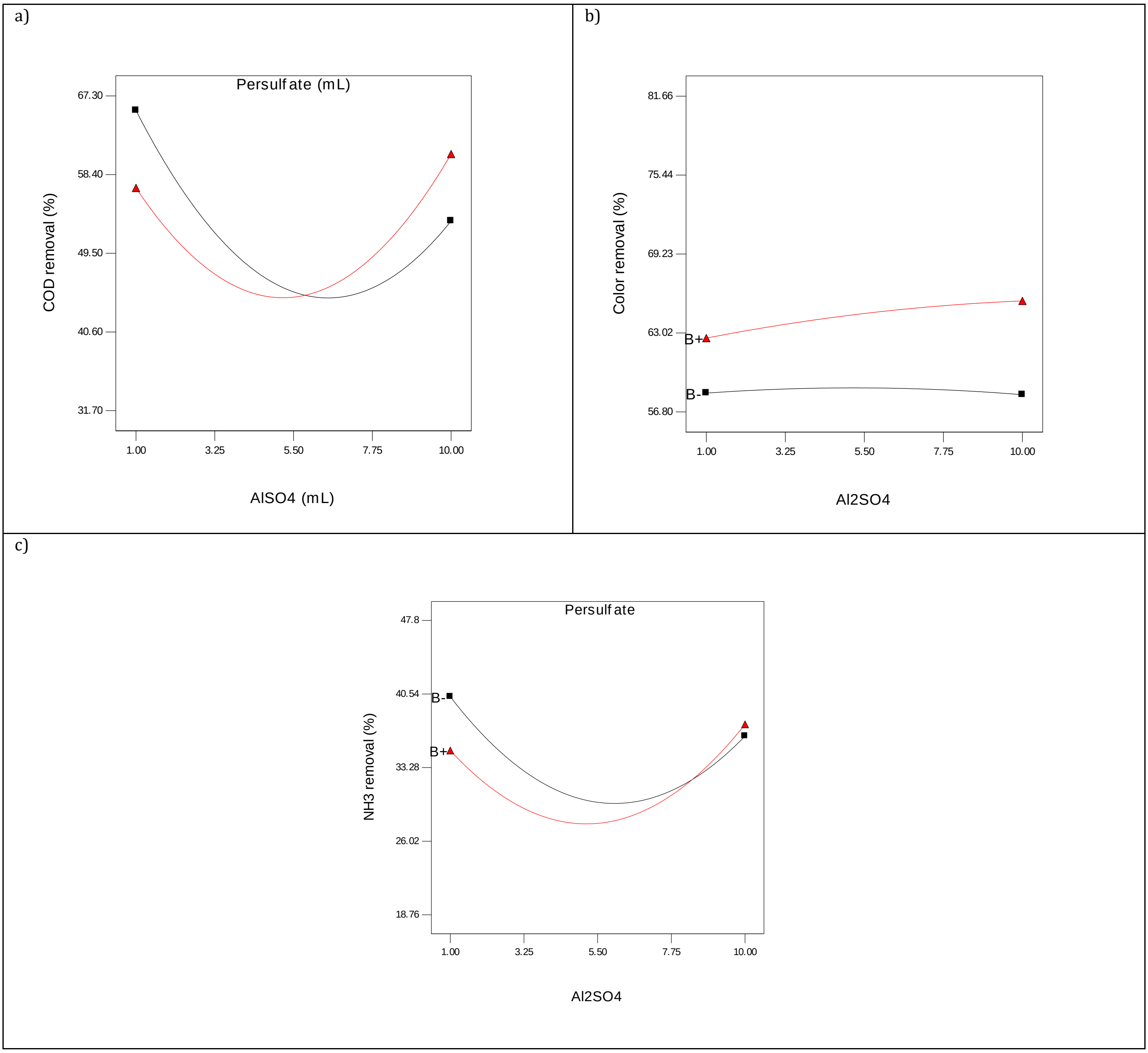

Figure 2: Two-factor interaction plot showing the behavior of Al_2SO4 and persulfate (■ = 1, ▲= 10 mL) on (A) COD color (B) and (C) NH_3-N removal at 6 pH and 105 min reaction time.

The second-order polynomial model for COD, color, and NH_3-N removals are given in Eqs. 4- 6, respectively.

$$COD = 53.88 - 2.17X_1 - 0.30X_2 - 1.71X_3 - 2.33X_4 + 14.27X_1^2 - 9.04X_2^2 - 12.64X_3^2 - 0.68X_4^2 + 4.08X_1X_2 - 3.26X_1X_3 - 3.09X_1X_4 - 1.06X_2X_3 - 0.59X_2X_4 + 2.79X_3X_4 \quad (4)$$

$$Color = 71.37 + 0.69X_1 + 2.93X_2 - 0.45X_3 + 3.28X_4 - 0.40X_1^2 - 9.77X_2^2 + 6.22X_3^2 + 3.22X_4^2 + 0.76X_1X_2 - 1.80X_1X_3 - 1.83X_1X_4 - 2.66X_2X_3 - 1.02X_2X_4 - 0.17X_3X_4 \quad (5)$$

$$NH3-N = 34.60 - 0.33X_1 - 1.04X_2 - 2.81X_3 - 3.92X_4 + 8.47X_1^2 - 5.78X_2 - 2.31X_3^2 - 8.62X_4^2 + 1.62X_1X_2 - 1.81X_1X_3 - 2.71X_1X_4 - 1.46X_2X_3 - 1.18X_2X_4 + 2.83X_3X_4 \quad (6)$$

where Y_1 , Y_2, and Y_3 represent the COD rem oval, Color removal and ammonia (NH_3), respectively.

The second-order polynomial models explained most of the variation in the COD, color, and NH_3-N data since the coefficient of determination (R^2) for the model is high and close to 1. The values of R^2 for COD, color, and NH_3-N models are 0.97, 0.98, and 0.94 respectively which shows that most of the variation is explained and a very small amount is unexplained by a model. The effect of each independent variable can be assessed by the coefficient associate with the variable, positive coefficient tells us the ability of the variable to increase the response with high stetting while negative coefficient indicates the ability to decrease the value of the response with the high setting.

The behavior of the independent variables and the selected responses can be represented in three-dimensional response surface plot as given in Figure 4 for COD, color, and NH_3-N removals, showing the behavior of pH and persulfate (a) and Al_2SO_4 (b). For COD, color, and NH_3-N removals revealed a clear picture for maximum removal for all selected responses. Furthermore, maximum percentage of removal for COD, color, and NH_3-N was observed within the selected intervals of the independent variables (factors).

The models presented in Figure 3 were used simultaneously to optimize the process and provide the suitable setting for the persulfate dosage, Al_2SO_4, pH, and reaction time to produce maximum removals for COD, color, and NH_3-N. The optimum solution (maximum removals) for advanced oxidation process was found at persulfate 10 ml, Al_2SO_4 5.5 ml, pH 5, and RT 105 min with removals of 68%, 82% and 48% for COD, color, and NH_3-N, respectively.

To evaluate the effectiveness of simultaneous $S_2O_8^{2-}$ and Al_2SO_4oxidation, leachate sample was treated with $S_2O_8^{2-}$ only and $Al2_2SO_4$ only (Figure 4). The efficiency of persulfate only was found to be limited for COD, color and ammonia removal (35%, 42% and 18%, respectively). Although $Al2_2SO_4$

achieved higher removal for COD and color (42% and 53%, respectively), the effectiveness of simultaneous $S_2O_8^{2-}$/ Al_2SO_4 oxidation was higher than other applications. (67%, 82% and 47% removal for COD, color and ammonia, respectively).

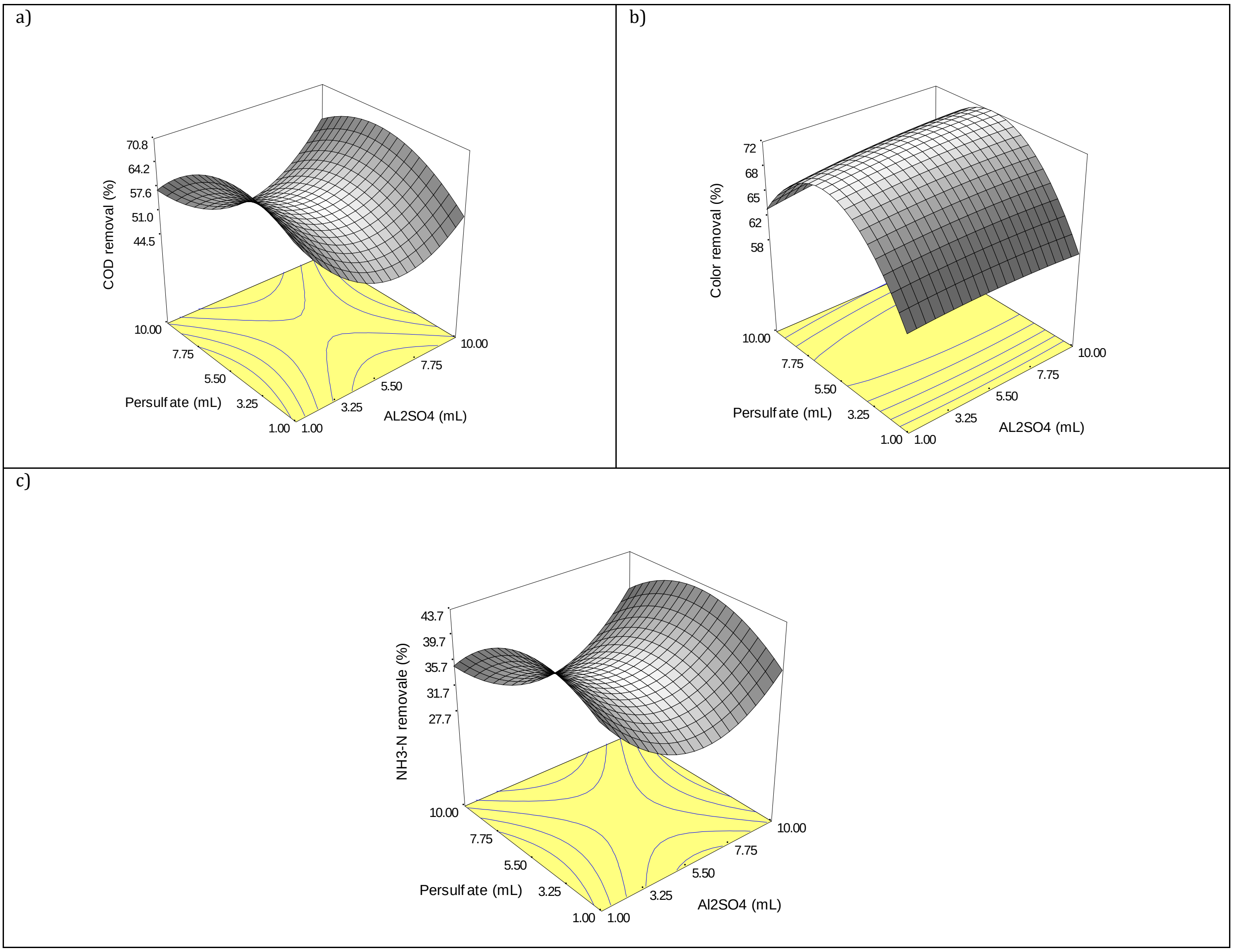

Figure 3: Three-dimensional response surface showing the effect of persulfate/Al_2SO_4 on (A) COD (B) color (C) and NH_3-N removal at 5.5 ml of persulfate and 105 min reaction time.

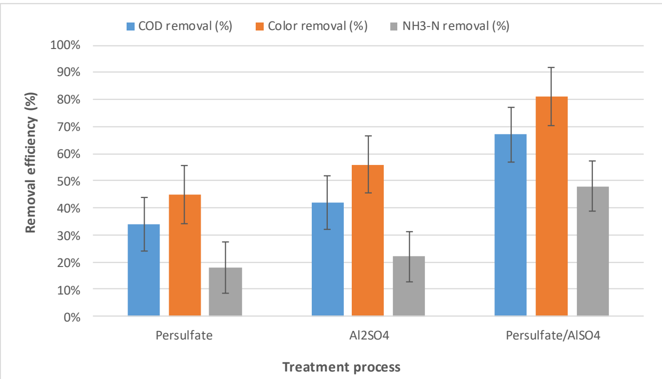

Figure 4: Comparison the performance of persulfate, Al_2SO_4 and combined persulfate/Al_2SO_4 for COD, color and NH_3-N removal.

4.CONCLUSION

In the current study, the performance of employing Al_2SO_4 to activate persulfate during oxidation of leachate was performed and evaluated. The optimum experimental conditions for the treatment was conducted with respect to operational conditions, namely, persulfate and Al_2SO_4 concentration, pH variation, and oxidation time. The performance of combined $S_2O_8^{2-}$ / Al_2SO_4 oxidation process is more efficient than the $S_2O_8^{2-}$ alone and Al_2SO_4 alone for leachate treatment. Accordingly, the combined $S_2O_8^{2-}$ / Al_2SO_4 treatment process improved the oxidation potential of organics and ammonia in landfill leachate.

ACKNOWLEDGMENT

This study was made possible through the support of the Malaysian Institute of chemical & Bioengineering Technology, Universiti Kuala Lumpur, (UniKL, MICET).

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