RESEARCH ARTICLE

THE EFFECT OF COMBINED Al$_2$SO$_4$ AND PERSULFATE ON COD, COLOR AND NH$_3$–H REMOVAL FROM LEACHATE

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ARTICLE DETAILS

ABSTRACT

In this paper, Al$_2$SO$_4$ was added to persulfate as an activator to increase reaction and to help increase oxidation during the leachate oxidation process. The effect of Al$_2$SO$_4$, 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$_2$SO$_4$ 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$_2$SO$_4$ dosage (0.2 M), 6 pH, and 105 (min) reaction time. The performance of combined S$_2$O$_8^2$/Al$_2$SO$_4$ process compared with S$_2$O$_8^2$ alone and Al$_2$SO$_4$ alone, and the results showed that the combined method achieved a higher removal efficiency for COD, color, and NH$_3$–N.

KEYWORDS

Al$_2$SO$_4$, persulfate, oxidation, removal, face centered composite design (FCCD), optimization.

1. INTRODUCTION

Increasing population growth and industries led to increase solid waste generation from residential areas. Although landfilling is still considered the most common and suitable method for municipal solid waste (MSW) disposal [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 application has 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$_2$O$_2$, and high pH [13-16]. Rostaghy employed ferrous persulfomonsulphate oxidant to generate sulfate radicals from persulfate for PCBs degradation in aqueous solution [17].

Al$_2$SO$_4$ reagent is widely used for wastewater treatment, however, the performance utilize Al$_2$SO$_4$ in activating persulfate for leachate treatment has not been investigated. The relationships between various parameters (intetactions) 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$_3$–N removal are provided. Moreover, the performance of simultaneous persulfate/ Al$_2$SO$_4$ was compared with persulfate alone and Al$_2$SO$_4$ 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].

| 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, ($\mu$S/cm) | 18,940 |

*Averaged of two samples taken from March and June 2017.
2.2. Experimental Procedures

In the current study, Sodium persulfate (Na2S2O8: M = 238 g/mol) and Aluminum sulfate (Al2(SO4)3: 342.15 g/mol) were used for advanced oxidation during the oxidation of leachate samples. Several dosages of S2O82− and Al2(SO4)3 were gradually mixed with 100 ml of leachate samples to determine the optimum S2O82− and Al2(SO4)3 dosage according to the efficiencies of COD, Color and NH3–N removal. Orbital Shaker (Lachham 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 °C) 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, Malaka, Malaysia.

2.3. Experimental design

The effect of four factors, namely persulfate dosage (X1), Al2(SO4)3 dosage (X2), pH (X3) and reaction time (X4) on three responses COD (Y1), color (Y2) and ammonia (Y3) removal efficiencies from leachate was studied. The relationship between the factors and the three responses was modelled and optimized by using face centered composite design (FFCD). FFCD 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.

| Variables                  | Symbol | Range and levels |
|----------------------------|--------|------------------|
| Persulfate dosage          | X1     | Low level (-1)   |
|                            |        | Center (0)       |
|                            |        | High level (+1)  |
| Al2(SO4)3 dosage           | X2     | 1 ml             |
|                            |        | 5.5 ml           |
|                            |        | 10 ml            |
| pH                        | X3     | 3                |
|                            |        | 6                |
|                            |        | 9                |
| Reaction time              | X4     | 30               |
|                            |        | 105              |
|                            |        | 180              |

The relationship between the selected factors (X1, X2, X3, X4) and each of the responses (Y1, Y2, Y3) is usually described in response surface methodology (RSM) by a second-order polynomial as given in Eq. (1).

\[ Y = \beta_0 + \sum_{i=1}^{4} \beta_i X_i + \sum_{i=1}^{4} \sum_{j=1}^{4} \beta_{ij} X_i X_j + \sum_{i=1}^{4} \sum_{k=1}^{4} \beta_{ik} X_i^2 + \sum_{i=1}^{4} \sum_{k=1}^{4} \beta_{ik} X_i X_k \]  

(1)

where Y represents the dependent variable, \( \beta_0, \beta_i, \) and \( \beta_{ij} \) are linear coefficient, quadratic coefficient and interaction coefficients respectively, need to be estimated, and \( X_i \) represents the independent variables.

Thirty runs were required for FFCD to cover all possible combination of \( X_1, X_2, X_3, X_4 \), and 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, these experiments were run in random order. The data for the thirty-run of FFCD with the coded and actual levels of the four factors are given in Table 3.

2.4. Analytical Methods

COD, color and NH3–N, were immediately tested before and after each experiment. Leachate sample was shocked well analyzed. NH3–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):

\[ \text{Removal}(\%) = \left( \frac{C_{i} - C_{f}}{C_{i}} \right) \times 100 \]  

(2)

where Ci and Cf refer to the initial and final COD, color and NH3–N concentrations respectively.

3. RESULTS AND DISCUSSION

Four independent factors (variables), namely: persulfate dosage (X1), Al2(SO4)3 (X2), pH (X3), and reaction time (X4) were identified to be influential in removing chemical Oxygen COD, color and NH3–N from landfill leachate. Thirty-runs were performed using the FFCD; the interactions between the four experimental variables were considered in each run to evaluate the validity of leachate treatment using simultaneous S2O82− and Al2(SO4)3 oxidation process. Al2(SO4)3 reagent was used to activate S2O82− 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 persulfate under the effect of pH was discussed by Furman [21]. Sulfate radical can be initiated at high pH (Eq. 4), 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].

\[ S_2O_8^{2−} + OH^− \rightarrow HSO_4^{−} + SO_4^{2−} + 1/2 O_2 \]  

(3)

The results of thirty-run obtained from face-centered composite design (FFCD) 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 NH3–N) and presented in Table 4. The effect of persulfate dosage (X1) 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 X1 and X2, X3 and X4, and X1 and X4 was significant as presented in Table 4. Significant interaction was exhibited between persulfate and pH, and between Al2(SO4)3 and pH while other two-factor interactions were insignificant (Table 4).

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

| Variables                  | Symbol | Range and levels |
|----------------------------|--------|------------------|
| Persulfate dosage          | X1     | Low level (-1)   |
|                            |        | Center (0)       |
|                            |        | High level (+1)  |
| Al2(SO4)3 dosage           | X2     | 1 ml             |
|                            |        | 5.5 ml           |
|                            |        | 10 ml            |
| pH                        | X3     | 3                |
|                            |        | 6                |
|                            |        | 9                |
| Reaction time              | X4     | 30               |
|                            |        | 105              |
|                            |        | 180              |

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

| Coded variable | Actual variable | Responses |
|----------------|-----------------|-----------|
| Al2SO4 Persulfate pH RT | Al2SO4 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 | 9 30 64.24 54.5 31.2 |
| 0 -1 0 0 | 5.5 1 6 105 56.8 39.78 31.07 |

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

| Color removal% (R-square = 0.904) | Source | Squares | Mean Square | DF | P-value F | Value | P-value |
|-----------------------------------|--------|--------|-------------|----|-----------|--------|---------|
| Model 896.92                    | 14     | 64.07  | 10.09       | < 0.0001 | 0.901 | < 0.0001 |
| $X_1$ 8.83                      | 1      | 8.83   | 1.39        | 0.2565 | 0.901 | < 0.0001 |
| $X_2$ 154.41                    | 1      | 154.41 | 24.32       | 0.0002 | 0.901 | < 0.0001 |
| $X_3$ 3.71                      | 1      | 3.71   | 0.58        | 0.4565 | 0.901 | < 0.0001 |
| $X_4$ 193.45                    | 1      | 193.45 | 30.47       | < 0.0001 | 0.901 | < 0.0001 |
| $X_5^2$ 0.58                    | 1      | 0.58   | 0.091       | 0.7665 | 0.901 | < 0.0001 |
| $X_6^2$ 245.70                  | 1      | 245.70 | 38.71       | < 0.0001 | 0.901 | < 0.0001 |
| $X_7^2$ 101.10                  | 1      | 101.10 | 15.93       | 0.0012 | 0.901 | < 0.0001 |
| $X_8^2$ 27.31                   | 1      | 27.31  | 4.30        | 0.0557 | 0.901 | < 0.0001 |
| $X_9X_2$ 9.33                   | 1      | 9.33   | 1.47        | 0.2441 | 0.901 | < 0.0001 |
| $X_1X_5$ 51.84                  | 1      | 51.84  | 8.17        | 0.0120 | 0.901 | < 0.0001 |
| $X_2X_6$ 53.36                  | 1      | 53.36  | 8.41        | 0.0110 | 0.901 | < 0.0001 |
| $X_3X_7$ 113.00                 | 1      | 113.00 | 17.80       | 0.0007 | 0.901 | < 0.0001 |
| $X_4X_8$ 16.61                  | 1      | 16.61  | 2.62        | 0.1266 | 0.901 | < 0.0001 |
| $X_5X_6$ 0.45                   | 1      | 0.45   | 0.071       | 0.7939 | 0.901 | < 0.0001 |
| Residual 95.22                  | 15     | 6.35   |             |       | 0.901 | < 0.0001 |
| 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)

| COD removal% (R-square = 0.901) | Source | Squares | Mean Square | DF | P-value F | Value | P-value |
|---------------------------------|--------|--------|-------------|----|-----------|--------|---------|
| Model 2206.30                   | 14     | 157.59 | 9.82        | < 0.0001 | 0.901 | < 0.0001 |
| $X_1$ 84.41                     | 1      | 84.41  | 5.26        | 0.0367 | 0.901 | < 0.0001 |
| $X_2$ 1.60                      | 1      | 1.60   | 0.100       | 0.7564 | 0.901 | < 0.0001 |
| $X_3$ 52.87                     | 1      | 52.87  | 3.29        | 0.0896 | 0.901 | < 0.0001 |
| $X_4$ 97.44                     | 1      | 97.44  | 6.07        | 0.0263 | 0.901 | < 0.0001 |
| $X_5^2$ 52.770                  | 1      | 52.770 | 32.88       | < 0.0001 | 0.901 | < 0.0001 |
| $X_2^2$ 211.90                 | 1      | 211.90 | 13.20       | 0.0025 | 0.901 | < 0.0001 |
| $X_2^2$ 414.18                 | 1      | 414.18 | 25.80       | 0.0001 | 0.901 | < 0.0001 |
| $X_2^2$ 11.19                   | 1      | 11.19  | 0.074       | 0.7889 | 0.901 | < 0.0001 |
| $X_2X_3$ 266.67                 | 1      | 266.67 | 16.61       | 0.0010 | 0.901 | < 0.0001 |
| $X_2X_4$ 169.78                 | 1      | 169.78 | 10.58       | 0.0054 | 0.901 | < 0.0001 |
| $X_3X_4$ 153.02                 | 1      | 153.02 | 9.53        | 0.0075 | 0.901 | < 0.0001 |
| $X_3X_5^2$ 17.89               | 1      | 17.89  | 1.11        | 0.3078 | 0.901 | < 0.0001 |

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The normal probability plots for COD, color and NH$_3$–N removal is demonstrated in Figure 1. Generally, most of the points follow the straight line for COD color and ammonia. A significant interaction between persulfate and Al$_2$SO$_4$ 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 describes the relationship between the independent variables and the responses.

### Table 1: Regression Analysis

| 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   | 0.0010  |
| $X_1X_2$| 52.27         | 1  | 52.27       | 3.15    | 0.0963  |
| $X_1X_3$| 117.29        | 1  | 117.29      | 7.06    | 0.0179  |
| $X_1X_4$| 34.22         | 1  | 34.22       | 2.06    | 0.1716  |
| $X_2X_3$| 22.28         | 1  | 22.28       | 1.34    | 0.2648  |
| $X_2X_4$| 127.92        | 1  | 127.92      | 7.70    | 0.0141  |
| 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    | 1244.07      | 29 |            |         |         |

### Figure 1: Design Expert plot; Predicted and actual standardized residual for (A) COD, (B) color (C) NH$_3$–N, removal
The second-order polynomial model for COD, color, and NH3-N removals are given in Eqs. 4-6, respectively.

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

(4)

\[
\text{Color} = 71.37 + 0.69X_1 + 2.93X_2 - 0.45X_3 + 3.28X_4 - 0.40X_1^2 - 9.77X_2^2 + 6.22X_3^2 + 3.22X_4^2 + 0.76X_1X_2 - 1.80X_1X_3 - 1.83X_1X_4 - 2.66X_2X_3 - 1.02X_2X_4 - 0.17X_3X_4
\]  

(5)

\[
NH_3-N = 34.60 - 0.33X_1 - 1.04X_2 - 2.81X_3 - 3.92X_4 + 8.47X_1^2 - 5.78X_2^2 - 2.31X_3^2 - 8.62X_4^2 + 1.62X_1X_2 - 1.81X_1X_3 - 2.71X_1X_4 - 1.46X_2X_3 - 1.18X_2X_4 + 2.83X_3X_4
\]  

(6)

where \( Y_1, Y_2 \), and \( Y_3 \) represent the COD removal, color removal, and ammonia (NH₃), respectively.

The second-order polynomial models explained most of the variation in the COD, color, and NH₃-N data since the coefficients of determination \( R^2 \) for the model is high and close to 1. The values of \( R^2 \) for COD, color, and NH₃-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 associated with the variable, positive coefficient tells us the ability of the variable to increase the response with high setting 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 NH₃-N removals, showing the behavior of pH and persulfate (a) and Al₂SO₄ (b). For COD, color, and NH₃-N removals revealed a clear picture for maximum removal for all selected responses. Furthermore, maximum percentage of removal for COD, color, and NH₃-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, Al₂SO₄, pH, and reaction time to produce maximum removals for COD, color, and NH₃-N. The optimum solution (maximum removals) for advanced oxidation process was found at persulfate 10 ml, Al₂SO₄ 5.5 ml, pH 5, and RT 105 min with removals of 68%, 82% and 48% for COD, color, and NH₃-N, respectively.

To evaluate the effectiveness of simultaneous S₂O₅²⁻ and Al₂SO₄ oxidation, leachate sample was treated with S₂O₅²⁻ only and Al₂SO₄ 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 Al₂SO₄
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](image)

Figure 3: Three-dimensional response surface showing the effect of persulfate/Al$SO_4$ on (A) COD (B) color (C) and NH$_3$-N removal at 5.5 ml of persulfate and 105 min reaction time.

![Figure 4](image)

Figure 4: Comparison the performance of persulfate, Al$SO_4$, and combined persulfate/Al$SO_4$ for COD, color and NH$_3$-N removal.

4. CONCLUSION

In the current study, the performance of employing Al$SO_4$ 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$SO_4$ 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$SO_4$ 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.

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