Stabilized leachate treatment by using combination of struvite precipitation and coagulation-flocculation methods: RSM optimization

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Abstract. Leachate is a form of pollution from landfills with pollutants in organic and inorganic forms. Stabilized leachate is characterized by the presence of organic materials with high ammoniacal nitrogen concentrations (>400mg/L), high chemical oxygen demand (COD) concentrations (<4000mg/L) and the ratio of BOD/COD is low (<0.1). In this study, struvite precipitation used to remove high ammonia content and coagulation-flocculation using poly-aluminium chloride (PAC) as coagulant agent and cationic polymer as flocculant agent used for COD and colour removal. Response surface methodology (RSM) is applied to determine the optimum parameters and interaction effects of the four main factors that influence the efficiency of treatment used; coagulant dosage, flocculant dosage, pH and Mg-N-P ratio on COD, NH$_3$-N and colour removal. Optimum parameters obtained from the study were the coagulant dosage of 2250 mg/L, flocculant dosage of 14 mg/L, pH 7, and Mg: N: P ratio 1:1. The combination of struvite precipitation and coagulation-flocculation method results a percentage of removal of COD 48.6%, NH$_3$-N 92.8% and color 98.8%.

1. Introduction
Solid waste management involves six main stages namely waste production control, storage, collection, transfer and transportation, solid waste processing and disposal in principle best practices for public health, economy, engineering, conservation, aesthetics and environment [1]. The ineffectiveness of solid waste management causes the problem of pollution of groundwater and surface water, in turn affecting human health and aquatic life. Therefore, the development of integrated and sustainable solid waste management is very important to reduce the level of pollution.

One of the important aspects of municipal solid waste management is leachate management. Leachate is a blackish-brown liquid produced by the diffusion of water through solid waste that carries with it all impurities resulting from the decomposition activity of the solid waste either in dissolved or suspended solids [1]. Leachate contains high levels of pollutants such as ammonia, organic and inorganic substances, heavy metals, soluble solids and colloids as well as various pathogens that have the potential to contaminate groundwater and surface water [2 - 4].

The leachate management and treatment system is still new and studies are underway to produce an effective treatment management. Therefore, the use of proper methods is necessary and important to treat leachate. The implementation of the most appropriate technique for leachate treatment depends on the leachate characteristics [5]. The state of the organic matter present, the age and structure of the landfill also influence the effectiveness of the treatment methods used [1]. The content of different...

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compositions requires different methods of treatment. Commonly used methods are biological preparations and physical-chemical preparations. Biological processing is often used to process young leachate containing high BOD concentrations due to its reliability, simplicity and cost effectiveness [6,7]. However, biologically processed are only effective for young leachate and less effective for stable leachate containing stable organic matter that is difficult to decompose. High ammonia nitrogen content in stable leachate inhibits the effectiveness of biological treatment methods [8].

For stable leachate processing, physical-chemical methods are more effective. Among the commonly used methods are coagulation-flocculation and chemical precipitation. The coagulation-flocculation method is a simple and commonly used method of treating water and wastewater and is further applied for leachate treatment. These coagulation-flocculation preparations are very effective in removing suspended solids, colloids and CODs [2,9], but less effective for the removal of ammonia nitrogen (NH₃-N).

Since single processing is not able to process leachate effectively, another method used in this study is the chemical precipitation method of struvite to remove of ammonia nitrogen content. Combined processing is the best way to process suitable leachate. Therefore, the use of a combination of coagulation-flocculation methods and struvite precipitation methods was selected in this study. The aims of this study were to determine the effectiveness of a combination of leachate treatment by chemical precipitation (struvite) and coagulation-flocculation methods and to determine the optimal parameters for the combination of struvite feeding method and coagulation-flocculation method using surface response method (RSM).

2. Materials and methods

2.1. Sampling method
Leachate sampling was conducted at Jeram domestic solid waste landfill, in Kuala Selangor. It is approximately 45 km from Shah Alam City. The sample taken was a 7-year-old leachate sample. The samples were placed in a 10-liter plastic container and taken to the laboratory and stored in a cool room at 4°C. The sample will be removed first at room temperature 2 hours before the analysis is carried out.

2.2. Struvite precipitation and coagulation-flocculation treatment
This struvite precipitation test was performed using a 500 mL beaker. The required chemicals to be weighed are di-sodium hydrogen phosphate (Na₂HPO₄·12H₂O) and magnesium sulfate (MgSO₄·7H₂O). 250 mL of leachate sample was placed in a 500 mL beaker. Leachate pH was determined by adding NaOH solution (1M) or H₂SO₄ solution (1M) to obtain the desired pH. The jar test stirrer was first turned on at a speed of 40 rpm before adding magnesium sulfate and followed by di-sodium hydrogen phosphate solutions. The speed of the jar test stirrer was increased to 150 rpm. Stirring is done for 15 minutes to allow all chemicals to dissolve and react with leachate. The jar test stirrer was stopped and the solution was left for 15 minutes.

This experiment was repeated by changing the pH of the leachate to the desired level by adding NaOH solution (1M) or H₂SO₄ solution (1M). The concentration of leachate-containing ammonia nitrogen before and after treatment was taken 3 times to obtain accurate data. Once all the optimal parameters were identified, the struvite precipitation test was performed first followed by coagulation-flocculation experiments. Determination of COD (mg / L), ammonium nitrogen (mg / L) and colour (PtCo) for raw leachate and treated leachate by combination method was tested 3 times to obtain accurate readings.

2.3. Analytical methods
The Nessler method is used to determine the ammonia nitrogen content in leachate based on the Standard Test Method on Water and Wastewater 4500-NH₃ B and C [10]. The COD test is determined by the colorimetric closed reflux method according to Standard Method of Test on Water and Wastewater using
DRB/200 reactor produced by HACH. Colour determination was determined using the Platinum-Cobalt standard method [11].

2.4. Design of experiment

The central composite design (CCD), which is one of the experimental designs in the response surface methodology (RSM), has been selected for the optimization of important variables in the experiments conducted such as coagulant dose, flocculants dose, Mg: N: P and pH ratio. Parameters were analyzed as a response were COD, ammonia nitrogen and colour. Since different variables are usually expressed in different units and have different variation limits, the significance of their effects on reactions can only be compared after they have been encoded. For statistical calculations, the Xi variable was coded as xi according to the following equation (1):

\[ X_i = \frac{x_i - x_{i,0}}{\delta x_i} \]  

where Xi represents the coded value of the independent variable i; xi is the non-coded value of the independent variable i; x {\text{0}} is the median of xi and Δxi step change data [12]. Δxi step changes are defined in equation (2):

\[ \delta x_i = \frac{\max(x_i) - \min(x_i)}{2} \]  

Flocculants dose (x1), coagulant dose (x2), pH (x3) and Mg: N: P ratio (x4) were selected as four independent variables; COD, NH3-N and colour were selected as dependent variables. The reaction variables were assembled by the second order model in the form of a quadratic polynomial equation, as stated in equation (3):

\[ Y_m = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 + \sum_{i<j} b_{ij} X_i X_j \]  

where i is a linear coefficient, j is a quadratic coefficient; b is the regression coefficient and k is the number of factors studied and optimized in the experiment. The interactive effect of the independent variable on the dependent variable is a two-dimensional plot contour illustration for this experiment. The coded and non-coded values were listed in table 1.
Table 1. The values of coded and non-coded factors for each experiment (Exp.) where $X_1$ and $x_1$ represent Coagulant, $X_2$ and $x_2$ represent Flocculants, $X_3$ and $x_3$ represent pH, $X_4$ and $x_4$ represent Mg-N-P ratio for coded (X) and non-coded value (x) respectively.

| Exp. | $X_1$ | $X_2$ | $X_3$ | $X_4$ | $x_1$/ppm | $x_2$/ppm | $x_3$/pH | $x_4$/ppm |
|------|-------|-------|-------|-------|------------|------------|----------|-----------|
| 1    | 1     | -1    | 1     | -1    | 3000       | 6.5        | 10       | 0.6       |
| 2    | -1    | -1    | 1     | 1     | 0          | 6.5        | 10       | 1.6       |
| 3    | -1    | -1    | -1    | -1    | 0          | 6.5        | 4        | 0.6       |
| 4    | 1     | -1    | -1    | 1     | 3000       | 6.5        | 4        | 1.6       |
| 5    | 0     | 0     | 0     | 2     | 1500       | 14         | 7        | 0.1       |
| 6    | 1     | 1     | 1     | 1     | 3000       | 21.5       | 10       | 1.6       |
| 7    | -1    | -1    | -1    | 1     | 0          | 6.5        | 4        | 1.6       |
| 8    | 0     | 0     | 0     | 0     | 1500       | 14         | 7        | 1.1       |
| 9    | 0     | 0     | 0     | 2     | 1500       | 14         | 7        | 2.1       |
| 10   | 0     | 0     | 0     | 0     | 1500       | 14         | 7        | 1.1       |
| 11   | 1     | 1     | -1    | -1    | 3000       | 21.5       | 4        | 0.6       |
| 12   | 0     | 0     | 0     | 0     | 1500       | 14         | 7        | 1.1       |
| 13   | 0     | -2    | 0     | 0     | 1500       | 0          | 7        | 1.1       |
| 14   | -1    | 1     | -1    | -1    | 0          | 21.5       | 4        | 0.6       |
| 15   | 0     | 0     | 0     | 0     | 1500       | 14         | 7        | 1.1       |
| 16   | -1    | 1     | 1     | -1    | 0          | 21.5       | 10       | 0.6       |
| 17   | 0     | 0     | 0     | 0     | 1500       | 14         | 7        | 1.1       |
| 18   | -2    | 0     | 0     | 0     | 0          | 14         | 7        | 1.1       |
| 19   | 1     | 1     | -1    | 1     | 3000       | 21.5       | 4        | 1.6       |
| 20   | 0     | 2     | 0     | 0     | 1500       | 29         | 7        | 1.1       |
| 21   | -1    | 1     | 1     | 1     | 0          | 21.5       | 10       | 1.6       |
| 22   | -1    | 1     | -1    | 1     | 0          | 21.5       | 4        | 1.6       |
| 23   | 2     | 0     | 0     | 0     | 4500       | 14         | 7        | 1.1       |
| 24   | 1     | -1    | 1     | 1     | 3000       | 6.5        | 10       | 1.6       |
| 25   | 0     | 0     | 2     | 0     | 1500       | 14         | 13       | 1.1       |
| 26   | 0     | 0     | -2    | 0     | 1500       | 14         | 1        | 1.1       |
| 27   | -1    | -1    | 1     | -1    | 0          | 6.5        | 10       | 0.6       |
| 28   | 1     | -1    | -1    | -1    | 3000       | 6.5        | 4        | 0.6       |
| 29   | 1     | 1     | 1     | -1    | 3000       | 21.5       | 10       | 0.6       |
| 30   | 0     | 0     | 0     | 0     | 1500       | 14         | 7        | 1.1       |
| 31   | 0     | 0     | 0     | 0     | 1500       | 14         | 7        | 1.1       |
3. Results and discussion

3.1. X-ray diffraction test

After the completion of the combined treatment of struvite precipitation and the coagulation-flocculation process on the leachate, the resulting precipitate is taken to determine the presence of minerals especially struvite that may be produced after treatment. The determination of the minerals in the resulting precipitate was tested using X-ray diffraction test. The presence of struvite can be ascertained based on the spectrum obtained in figure 1.

The angle of the deflection test has been set at 2-theta angle. Based on the X-ray diffraction tests performed, the presence of struvite minerals or also known as magnesium ammonium phosphate (MAP) can be determined in the treatment sludge. Figure 1 shows the spectrum of X-ray diffraction tests in which the highest peaks indicate the presence of struvite minerals. Other minerals also identified are rhoeite, Na2SO4 and thenardite, (Mg4Ti2)Ca2(Al4Si2O20). The presence of struvite in the resulting precipitate indicates that the content of ammonium ion (NH4+) in the leachate has reacted with magnesium ion (Mg2+) and phosphate ion (PO43-) added during the treatment process and formed the struvite mineral MgNH4PO4·12H2O. The chemical reaction of struvite formation is represented in equation (4):

\[
\text{Mg}^{2+}(\text{ak}) + \text{PO}_4^{3-}(\text{ak}) + \text{NH}_4^+(\text{ak}) \rightarrow \text{MgNH}_4\text{PO}_4(\text{p})
\]

![Figure 1. X-ray diffraction spectrum for leachate sludge.](image)

3.2. Optimization of COD removal

A total of thirty-one experiments were conducted to obtain the effectiveness of COD removal for the combination of struvite precipitation treatment and coagulation-flocculation. The quadratic regression model obtained is stated in equation (5):

\[
Y_{\text{COD}} = 2131.9 - 0.35X_2 + 1.84X_3 - 145.52X_1 - 265.38X_4 - 0.07X_1^2 + 13.33X_2^2 - 25.04X_3^2 - 0.01 X_2X_1 + 0.06X_2X_4 -1.3X_3X_1 + 7.87X_3X_4 + 5.33X_1X_4
\]
The results of ANOVA for the quadratic model formulating the model used for COD removal analysis are significant. ANOVA shows that the second order model is very much in line with the COD response test data. For COD reduction, it can be seen that the linear effects of coagulant dose and pH are significant. The interaction between coagulant dose and pH is very significant, while the interaction between other factors is insignificant for COD removal. Based on the quadratic regression model, the positive and negative values of the regression coefficient affect the effectiveness of variables to get rid of COD, whereas while positive values indicate the ability of variables to increase the effectiveness of removal, while negative values indicate the ability of variables to reduce the effectiveness of COD removal. The 3-Dimensional (3D) contour plot for COD in Figure 2 shows the effect of pH interaction and coagulant dose on COD removal. From the plot, it can be seen that COD removal is influenced by the interaction between pH and coagulant dose. Meanwhile, other factors respond individually.

Analysis of variance (ANOVA) was used to analyze the data and obtain the interaction between the four variables (coagulant dose, flocculants dose, Mg: N: P, pH ratio) on COD removal using statistical tests from Minitab 16. Response Surface Methodology (RSM) is used to assess the effect variables used on COD removal. Lack of fit (LOF) F - test was used to check whether the regression function matched the data. Quadratic regression indicates that this model is significant, due to the P LOF value (> 0.05). This indicates that there is a correlation between the COD variables and parameters. The value of the correlation coefficient (R² = 0.682) indicates that only 32% of the total variation cannot be explained by the empirical model. Figure 2 A-D show that the COD removal is obtained by setting one variable while the other two variables are varied within the desired experimental range. The 2-dimensional contour plot (2D) shows that all plots have a relatively large curvature. The optimal conditions for COD removal are at the heart of the design boundary, where it indicates that there is an interdependent interaction between the three factors.

Based on the experiments conducted, there is a significant interaction between pH and coagulant dose. The interaction between the coagulant dose and the pH is due to the presence of anions from the surface agent in the leachate which has a negative charge on the alkaline pH. While other factors such as Mg: N: P ratio and flocculant dose are less significant for COD removal. The contour plot of COD removal as in Figure 2 shows that the optimum pH is in the range of pH 5 to pH 9. COD removal is high when the coagulant dose is in the range of 1800 mg / L to 3500 mg / L. In the process of coagulation-flocculation, the aggregation of colloidal particles occurs through charge neutralization and sweep floc mechanism. In general, pH 6 to pH 8 is an ideal condition for the formation of amorphous aluminum hydroxide [Al(OH)₃] when aluminum ions are used as coagulants. The formation of the amorphous Al(OH)₃ causes the removal of organic matter through a sweep floc mechanism through adsorption on the precipitation of Al(OH)₃.

Based on the combined treatment experiments used, the results show that the percentage range of COD removal is 5.2% - 48.6%. This indicates that the maximum COD removal is 48.6%. Therefore, the most optimal COD removal is at pH 7.
3.3. Optimization of ammonia removal

A total of thirty-one experiments were conducted to obtain the effectiveness of NH$_3$-N removal for the combination of struvite precipitation treatment and coagulation-flocculation. The quadratic regression model obtained is shown in equation (6):

$$Y_{NH3-N} = 502.099 - 0.067X_3 + 22.894X_3 + 27.537X_1 + 570.705X_4 + 0.277X_3^2 - 6.115X_1^2 - 305.131X_3^2 - 0.006X_3X_3 + 0.017X_3X_1 + 0.102X_3X_4 - 0.861X_3X_1 - 6.833X_3X_4$$  \hspace{1cm} (6)

The ANOVA results for the quadratic model formulating the model used for the analysis of ammonium nitrogen (NH$_3$-N) removal are significant. ANOVA shows that the second order model is very much in line with the ammonia nitrogen removal experimental data. For the reduction of NH$_3$-N, it can be seen that the linear effects of Mg: N: P and pH ratios are significant. The interaction between Mg: N: P and pH was very significant, while the interaction between other factors was insignificant for NH$_3$-N removal.

Based on the quadratic regression model (figure 5), the positive and negative values of the regression coefficient affect the effectiveness of the variables for removal of NH$_3$-N, whereas positive values indicate the ability of the variables to increase the effectiveness of removal, while negative values indicate the ability of variables to reduce the effectiveness of NH$_3$-N removal. The 3-Dimensional (3D)
contour plot (figure 3A-D) for NH$_3$-N shows the effect of pH and Mg-N-P ratio on NH$_3$-N removal. From the plot, it can be seen that NH$_3$-N removal is influenced by the interaction between pH and Mg: N: P ratio. Meanwhile, other factors respond individually.

Analysis of variance (ANOVA) was used to analyze the data and obtain the interaction between the four variables (coagulant dose, flocculants dose, Mg: N: P, pH ratio) on NH$_3$-N removal using statistical test from Minitab 16. Response Surface Methodology (RSM) was used to evaluate the effect of the variables used on NH$_3$-N removal. The value of the correlation coefficient ($R^2 = 0.728$) indicates that only 27% of the total variation cannot be explained by the empirical model. Figure 3 show that the removal of NH$_3$-N is obtained by setting one variable while the other two variables are varied within the desired experimental range. For 2-dimensional (2D) contour plots, the optimal conditions for NH$_3$-N removal are at the center of the design boundary, where it indicates that there is interdependent interaction between the three factors.

In general, the effectiveness of NH$_3$-N removal is determined by the quantity of struvite formed after treatment performed on leachate as stated in equation (7).

$$\text{NH}_4^+ + \text{Mg}^{2+} + \text{PO}_4^{3-} + 6\text{H}_2\text{O} \rightarrow \text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O} \quad (7)$$

The interaction between pH and Mg:N:P ratio is significant. The effectiveness of ammonia removal increases with increasing pH i.e. effective removal at alkaline pH. This is due to the acidic pH of pH (1-6), the presence of hydrogen ions (H$^+$) interferes with the formation of phosphate (PO$_4^{3-}$) as indicates in equation (8).

$$\text{H}_3\text{PO}_4 \rightarrow \text{H}_2\text{PO}_4^- + \text{H}^+$$
$$\text{H}_2\text{PO}_4^- \rightarrow \text{HPO}_4^{2-} + \text{H}^+$$
$$\text{HPO}_4^{2-} \rightarrow \text{PO}_4^{3-} + \text{H}^+ \quad (8)$$

The presence of hydrogen ions promotes the formation of other acids when the phosphate from the coagulant used i.e. di-sodium hydrogen phosphate-12-hydrate ($\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$) reacts with the leachate hydrogen ion. Therefore, the optimum pH for NH$_3$-N removal is in the pH range (8-13) based on figure 3.
Figure 3. Contour plot of ammonia nitrogen (NH$_3$-N) concentration (mg/l) for factors of Mg-N-P ratio, pH, coagulant dose and flocculant dose.

3.4. Optimization of ammonia removal

A total of thirty-one experiments were conducted to obtain the color removal effectiveness for the combination of struvite precipitation treatment and coagulation-flocculation. The quadratic regression model obtained is stated in equation (9):

\[
Y_{\text{color}} = 10641.1 - 2.9X_2 - 133.7X_3 - 442.1X_4 - 5683.9X_4 - 0.7X_1^2 + 37.8X_1^2 + 1464.7X_2^2 + 0.5X_3X_4 + 1.5X_2X_4 + 73.3X_2X_4 - 3.3X_1X_4
\]  

(9)

Based on the quadratic regression model (figure 5), the positive and negative values of the regression coefficient affect the effectiveness of variables to remove color, whereas positive values indicate the ability of variables to increase removal effectiveness, while negative values indicate the ability of variables to reduce color removal effectiveness.

The results of ANOVA for the quadratic model formulating the model used for color removal analysis are significant. ANOVA shows that the second order model is very much in line with the color removal experiment data. Based on the results of a combined study of struvite precipitation treatment and coagulation-flocculation, almost all factors influence color removal. All interactions between Mg:N:P and pH ratios, Mg:N: P ratios and coagulant doses, Mg: N: P ratios and flocculants doses, pH
and coagulant doses, pH and flocculants doses, as well as coagulant and flocculants dose shows significant interaction. However, the interaction between Mg:N:P and pH is most significant.

Analysis of variance (ANOVA) was used to analyze the data and obtain the interaction between the four variables (coagulant dose, flocculants dose, Mg:N:P ratio, pH) on color removal using statistical tests from Minitab 16. Response Surface Methodology (RSM) was used to evaluate the effect of the variables used on NH$_3$-N removal. Lack of fit (LOF) test - F was used to check whether the regression function matched the data. The quadratic regression indicates that this model is significant, due to the P LOF value (>0.05). This indicates that there is a correlation between the variables on color removal. The value of the correlation coefficient ($R^2 = 0.789$) indicates that only 21% of the total variation cannot be explained by the empirical model. Figure 4 shows that color removal is obtained by setting one variable while the other two variables are varied in the experiment. For 2-dimensional (2D) contour plots, the optimal conditions for colour removal are at the center of the design boundary, where it indicates that there is interdependent interaction between the three factors.

**Figure 4.** Contour plot of color for factors of Mg-N-P ratio, pH, coagulant and flocculants dose.
Figure 5. Contour plot of ammonia nitrogen (NH$_3$-N) concentration (mg/l) for factors of Mg-N-P ratio, pH, coagulant dose and flocculant dose.
3.5. Optimum plot for COD, ammonia and color removal

Figure 6 shows the optimal plot of the combined factors for COD, ammonia and colour. The optimum values of coagulant dose, flocculants dose, pH and Mg-N-P ratio are 2250 mg / L, 14 mg / L, 7 and 1.1 respectively for maximum removal of COD, ammonia and colour. While the expected elimination results for COD, NH3-N and color are 952 mg / L, 854 mg / L and 706 mg / L, respectively.

![Figure 6. Optimal plot of COD, NH3-N and color removal.](image)

| Optimal Factor | Coag1 | Flocc1 | pH1 | RatioMgN |
|----------------|------|-------|-----|----------|
|                | (2250.0) | [14.0] | [7.0] | [1.10] |
| Composite Desirability (Cur) | High | Low |
| Color Minimum | y = 706.6832 | d = 0.00000 |
| COD Minimum | y = 952.5378 | d = 0.00000 |
| Ammonia Minimum | y = 854.2949 | d = 0.00000 |

4. Conclusions

The results of the study found that the removal of the most harmful pollutants in the leachate was successfully removed using struvite precipitation method and coagulation-flocculation method. The pollutants tested in this research focused on 3 main parameters namely COD, ammonia nitrogen and color. There are 4 optimal factors studied in this research, namely pH, coagulant dose, flocculants dose and Mg: N: P ratio. The optimum pH obtained in this study is at pH 7, the optimal coagulant dose (PAC) is at a concentration of 2250 mg / L, the dose of flocculants (cationic polymer) at a concentration of 14 mg / L and the optimal Mg: N: P ratio is at a ratio of 1: 1: 1. The maximum removal of ammonium nitrogen, COD and color nitrogen was 92.8%, 48.6% and 98.8% respectively based on the optimal factors obtained through the application of Response Surface Methodology (RSM).

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