OPTIMIZATION OF STRUVITE PRECIPITATION FOR LANDFILL LEACHATE TREATMENT

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Abstract: Sanitary landfill is the most preferred municipal solid waste disposal method. The production of highly polluted leachate is a major disadvantage of sanitary landfills. In this study, optimization of struvite precipitation to remove ammonium from landfill leachate was conducted by using Response Surface Methodology and central composite design. Optimum struvite precipitation conditions were determined based upon 11 runs performed in central composite design. A second-order polynomial functional model was fitted well to the results. The statistical analysis showed that two independent variables which are molar rates of Mg/N and N/P had significant effects on the ammonium removal efficiency. Maximum ammonium removal efficiency was 99.8% at a molar rate of 1.20 for Mg/N and 1.27 for N/P for a constant 9.2 pH value. The obtained results revealed that struvite used as pre-treatment in anaerobic process can be modelled by using response surface methodology. And also, response surface methodology can be used to optimize required ammonium removal efficiency for lower Mg/N and N/P molar ratio which affects the performance of pre-treatment method that designed for an anaerobic process having 300:5:1 ratio for COD/N/P.

Keywords: Central Composite Design (CCD), Landfill, Leachate, MAP, Response Surface Methodology (RSM), Struvite

Sızıntı Suyu Arıtımında Strüvit Çöktürme Optimizasyonu

Öz: Evsel katı atıkların bertarafında düzenli depolama yöntemi sıklıkla tercih edilmektedir. Çok kirli sızıntı suyu oluşumu, düzenli depolama tesislerinin en önemli dezavantajıdır. Bu çalışmada, Cevap Yüzey Yöntemi ve Merkezi Kompozit Tasarımı kullanılarak sızıntı suyundan amonyum giderimine yönelik strüvit çöktürme optimizasyonu yapılmıştır. Optimum strüvit çöktürme şartlarının belirlenmesi için 11 adet deneysel çalışma yapılmıştır. Sonuçlar ikinci dereceden polinom fonksiyonel model ile iyi uyum göstermiştir. İstatistiki analizler, Mg/N ve N/P molar oranı bağımsız değişkenlerinin amonyum giderim verimi üzerine önemli etkisinin olduğunu ortaya koymuştur. En yüksek amonyum giderim verimine %99,8 olarak; 9,2 sabit pH değerinde, Mg/N için 1,2 ve N/P için 1,27 molar oranlarında ulaşılmıştır. Elde edilen sonuçlar, anaerobik proseste ön arıtım olarak kullanılan strüvitin Cevap Yüzey Yöntemi kullanılarak modellenebildiğini ortaya koymuştur. Ayrıca Cevap Yüzey Yöntemi; KOİ/N/P için 300:5:1 oranına sahip bir anaerobik proses için tasarlanmış olan ön arıtım performansını etkileyen daha düşük Mg/N ve N/P molar oranı için gerekli amonyum giderme verimliliğini optimize etmek için kullanılabilir.

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1. INTRODUCTION

Sanitary landfill is the most preferred municipal solid waste (MSW) disposal method due to its low cost and easily disposable procedure (Aziz et. Al. 2010). However, the production of highly polluted leachate is a major disadvantage of sanitary landfills (Wiszniowski et al. 2007).

Leachate contains chemical impurities such as refractory organic compounds, ammonium (NH_4^+), phosphate (PO_4^{3-}), sulfide (S^{2-}), heavy metals, and chloride (Erkan and Apaydin, 2015).

Treatment of landfill leachate having high organic and inorganic contents requires combined techniques using modular or multistage units based upon targeted pollutants (Aygun et al. 2012). Therefore, leachate characteristics need to be identified to select the appropriate treatment methods (Durmusoglu and Yilmaz, 2006; Argun et al. 2017). Physical (Welander and Henrysson, 1998), chemical (Aneggi et al. 2012; Subramaniam et al. 2017; Yilmaz et al. 2010; Huang et al. 2016) and biological methods (Agdag and Sponza, 2005; Castillo et al. 2007) are usually used together for leachate treatment to achieve discharge standards. When these methods are not enough, leachate recirculation of treated landfill leachate can offer operational advantages such as enhanced microbiological treatment of leachate, improved landfill stabilization, and volume reduction of leachate by evaporation or absorption by refuse.

Anaerobic treatment can be successfully applied for removal of organic matter while it is inefficient for ammonium removal. High concentrations of ammonium have adverse effect on biological treatment systems due to its inhibitory effects on the methanogenic stage, resulting in reduced methane production (Zhang et al. 2009). Thus, pretreatment of excess ammonia from landfill leachate has crucial importance.

Air stripping, membrane separation processes (Brown et al. 2013; Ferraz et al. 2013; Rizkallah et al. 2013) and chemical precipitation (Quan et al. 2010; Zhang et al. 2011; Kabdasli, et al. 2008) are commonly used process for reducing of the excessive amount of ammonia in aqueous phase. Carbonate scaling in air stripping process creates an operational problem during the pretreatment steps. Therefore, some alternative techniques of ammonium nitrogen removal from leachate need to be studied (Campos et al. 2013; Li and Zhao. 2002; Zhang et al. 2011; Tonetti et al. 2016). As a chemical precipitation technique MAP is a promising and successful treatment option (Chen et al. 2010; Yetilmezsoy and Sapci-Zengin, 2009; Farrow et al. 2017). Ammonia removed from the leachate by struvite precipitation (MAP), having an inhibitory effect on the methane bacteria (Di Iaconi et al. 2010), can be used in agricultural field as fertilizer (Uysal and Kuru, 2013).

Precipitated white crystalline MAP is formed from the reaction of equal molar concentrations of magnesium, ammonium and phosphorus given in the following equation (1).

$$Mg^{2+} + NH_4^+ + PO_4^{3-} + 6H_2O \rightarrow MgNH_4PO_4.6H_2O \downarrow \quad K_{ÇÇ} = 2.5x10^{-13} \ (25 \ ^0C) \ (1)$$

pH is the main driving force behind of MAP precipitation reaction and the changes in pH of the solution affect the solubility products or MAP crystal formation (Gunay et al. 2008; Kabdasli et al. 2017). While MAP formation is inhibited by hydrogen ions at the low pH values, $Mg_3(PO4)_2$ might be formed instead of MAP at the high pH values (Zhang et al. 2009), leading to dropping down ammonium removal efficiency of the system in either case.

MAP precipitation efficiency also depends on magnesium, phosphate, and ammonium concentrations in aqueous phase. Li et al. (1999) reported that ammonium nitrogen content in the leachate could be quickly reduced from 5618 to 112 mg/L when the $Mg:NH_4^+:PO_4^{3-}$ molar ratio was equal to 1:1:1 (Ozturk et al. 2005). This molar ratio has been used as a reasonable value for MAP precipitation in previously published journal articles. While MAP precipitation

can be successfully applied for 91 % ammonium removal in the leachate, organic fraction of nitrogen removal is inefficient for this process. Ozturk et al. (2005) found that MAP precipitation was more effective for removal of NH_3–N than that of the organic compounds. Another study conducted by Calli et al. (2005) found that 3260 mg/L initial concentration of NH_3–N was removed at 98% by precipitation but only 20% of COD removal was achieved. Therefore, COD removal during the MAP precipitation may be attributed to the co-precipitation of MAP with side-products and other impurities present in the leachate. Because the total COD was not significantly reduced by the MAP precipitation, an additional biological treatment process may be needed for removal of residual COD (Yetilmezsoy and Sapci-Zengin, 2009).

Operational conditions of MAP precipitation for phosphate removal from artificial wastewater such initial pH and molar ratio of reagent was optimized by using Response Surface Methodology (RSM) in a recent study (de Luna et al. 2015). If wastewater having high ammonium concentrations needs to be treated by anaerobic methods, pretreatment has to apply to obtain the required effluent concentrations.

MAP precipitation can be an alternative pretreatment method. However, there is an information gap of operation conditions on ammonium removal by MAP precipitation from landfill leachate. The novelty of this research is primarily an optimization of promising MAP precipitation techniques useful as pretreatment method for landfill leachate. In this study, Central Composite Design (CCD) and RSM used to optimize the effects of stoichiometric rates of Mg/N and P/N on NH_4^+-N removal at constant 9.2 pH value from landfill leachate.

2. MATERIAL AND METHODS

2.1. Leachate Sampling and Analytical Methods

Konya, having the population of approximately 2 million people, is located in the central part of Turkey. Leachate samples were collected from the active detention pond in municipal landfill site of Konya. The samples, taken from 5 years aged pond, were stored in plastic containers and kept in 4 $^{\circ}$C until required analysis.

All chemicals which are analytical grade used in this research were supplied by Merck. pH of system was adjusted by using either 6M HCl or NaOH. Batch experiments were performed in jar test equipment (Velp Scientifica, FC4S) for total of 55 minutes each; 5 min at 120 rpm for rapid mixing, 20 min at 45 rpm for slow mixing and subsequently 30 min for settling.

All experimental runs were performed under laboratory temperature and pressure conditions. COD, BOD_5, pH, Sulphate, Phosphate-phosphorus, and Ammonium-nitrogen were analysed in the laboratory based on Standard Methods given as SM 5220 D, SM 5210 D, SM 4500-H^+ B, SM 4500-SO_4^{-2} E, SM 4500-PO_4-P E and SM 4500-NH_4-N B-F, respectively. pH measurements were done by using the WTW Multiparameter 340i. All of the colorimetric determinations were made with a DR 5000 UV-Vis Spectrophotometer.

pH values for ammonium removal were set as 9-9.5 in previously published journal articles (Booker et al. 1999; Li and Zhao, 2002; Zhang et al. 2009; Turker and Celen, 2007). However, optimum pH value was reported as 9.2 for ammonium removal (Ozturk et al. 2005). Therefore, all experiments were conducted at pH 9.2 in this study.

2.2. Experimental Design and Data Analysis

RSM consists of a group of mathematical and statistical techniques used in the development of an adequate functional relationship between input variables (X) and responses as output variables (Y). It is also a designed regression analysis meant to predict the value of a dependent variable based on the controlled values of the independent variables (Lee et al, 2006). In recent years, RSM has been applied to optimize and evaluate the interactive effects of

independent factors in different treatment systems for leachate treatment (Ghafari et al. 2009; Kabuk et al. 2014).

The statistical design of experiments and data analysis was conducted by using the Design Expert Software (version 8.0.7.1). In this research, to optimize the two most important operating variables of sutrivite precipitation such as Mg/N and P/N molar ratios at constant pH of 9.2 was used by CCD and RSM to maximize ammonium removal efficiency.

MAP precipitation was carried out at different predicted initial conditions for determining optimum input data for the model. In this respect, effect of $Mg:NH_4^+:PO_4$ molar ratio ranging from 0.5:1.0:0.8 to 2.0:1.0:1.4 at pH of 9.2 was determined on laboratory scale. Sodium hydrogen phosphate (Na_2HPO_4) and magnesium chloride ($MgCl_2$) was used for external sources of phosphorus and magnesium, respectively.

Response surface methodology postulates the functional relationship between the controllable input parameters and the obtained response surfaces. To evaluate our experimental data, the response variable was fitted by a second order model in the form of the following quadratic polynomial equation (2).

$$Y_i = \beta_0 + \beta_1 X_{1i} + \beta_2 X_{2i} + \beta_{12} X_1 X_{2i} + \beta_{11} X^2_{1i} + \beta_{22} X^2_{2i} + \varepsilon_i \qquad (2)$$

where i=1, 2; y_i is the dependent variable; X_{1i}, X_{2i} are the independent variables; β_i are the regression coefficients and e is the random error. The result of model was focused on verifying the influence of individual parameters on ammonium removal efficiency.

In the present work, CCD with three replicates of the center point was used to find the relationship between the response functions and variables. A total of 11 experiments were performed. Mg/N molar rates (A) and N/P molar rates (B) were set at five coded value levels: −1.5 (minimum), −1, 0 (central), +1, and +1.5 (maximum). Experimental range and levels of the independent variables used in RSM are presented in Table 1.

Table 1. Experimental range and levels of the independent variables used in RSM

Independent process variables code	Real values of coded levels				
	-1.5	-1	0	1	1.5
Mg/N molar rates	0.5	0.75	1.25	1.75	2
N/P molar rates	0.8	0.9	1.1	1.3	1.4

3. RESULTS AND DISCUSSION

3.1. Fitting the Models

Traditional way of optimization is not sufficient to express the combined effects of all operating parameters. Thus, the present study used RSM to optimize each input parameters in order to determine the combined effects on dependent variables. Actual design parameters of experiments and their responses as ammonium removal efficiency for MAP precipitation are given in Table 2.

Analysis of variance (ANOVA) was used for graphical analysis of the data to obtain the interaction between the process variables and the responses. The goodness of the fit polynomial model was expressed by the coefficient of determination, R^2; and its statistical significance was checked by the Fisher's F-test in the same software. Model terms were evaluated by the p-value (probability) with 95% confidence level. Three-dimensional plots and their respective contour plots were obtained for ammonium nitrogen removal based on the effects of two factors, Mg/N and N/P molar rates, at five levels.

ANOVA was used to assess the significance of each term in the fitted model. Backward elimination procedure of hierarchical terms was followed to find the best fit. Backward elimination procedure was initialized including all predictors in the model. The least significant variable was eliminated at each step, and then the model was refitted. Backward elimination procedure was finalized when all the remaining variables produced Fisher's F-test statistics with less than 0.10 p-values.

Table 2. The actual design of experiments and responses for MAP precipitation

Run	Mg/N (x_1)	N/P (x_2)	NH_4-N (%)
1	1.75	0.90	97.34
2	1.25	1.40	98.50
3	2.00	1.10	92.08
4	0.50	1.10	87.58
5	1.25	1.10	99.59
6	1.25	1.10	99.60
7	1.25	0.80	98.26
8	0.75	1.30	96.94
9	1.75	1.30	95.37
10	1.25	1.10	99.61
11	0.75	0.90	89.72

R^2 measures the proportion of variation for the response explained by the model. Regression analysis of mathematical model output to the experimental data was carried out to seek an optimal range for the response. ANOVA result of dependent parameter (response) is given in Table 3.

Table 3. ANOVA results for response parameters

Response	F-value	p-value	R^2	Adj R^2	AP	SD	CV
NH_4-N removal	51.29	< 0.0001	0.9716	0.9526	20.258	0.92	0.96

Predicted response function (ammonium nitrogen removal efficiency) was found as the following second-order polynomial equation under actual experimental conditions. (Eq (3))

$$Y_i = 34.20 + 69.72X_{1i} + 32.02X_{2i} - 22.98X_1X_{2i} - 16.57X^2_{1i} \qquad (3)$$

That the interaction term is antagonistic which means the slope of the response function against one of the predictors decreases for higher levels of the other predictor. Due to the interaction term, it is not possible to separate the effects of X_{1i} and X_{2i} on Y_i. Thus, the effects of Mg/N and N/P on ammonium removal efficiency cannot be separately interpreted.

The number of degrees of freedom (DoF) in the model affects the significance of the F-value. As shown in Table 4, the model to predict the removal of ammonium nitrogen efficiency was significant at the 5% confidence level since p-value was below 0.05.

F-test shows that the models had high F-value (a very low p-value) indicating that the models were highly significant for ammonium removal efficiency. A high coefficient of R^2 provides a satisfactory adjustment of the quadratic model to our experimental data. The value of Adj R^2 for Y was 0.9526, showing that less than 5.0% of the total variation was not demonstrated by the fitted model. This also proved that the models were highly significant, showing a good correlation between the observed and predicted data.

Relatively low values of Coefficient of Variance (CV) observed in this study show a better precision and reliability of the experiments. Adequate precision (AP) is 20.258 for response it means that due to the ratios greater than 4 this indicates adequate for model discrimination.
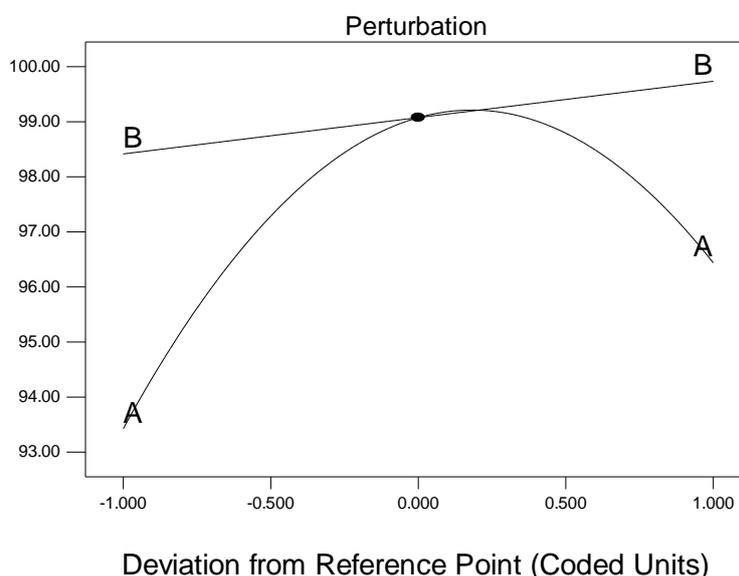
The *p*-values for ammonium nitrogen removal efficiency given Table 4 is used as a tool to check the significance of each term and the interaction strength between each independent variable. Higher F-value and smaller *p*-values are more significant on the model.

Table 4. ANOVA table for fitted model term

Source	Sum of Squares	Degrees of Freedom	Mean Square	F-value	*p*-value
X_1	19.28	1	19.28	22.76	0.0031
X_2	3.70	1	3.70	4.37	0.0816
X_1X_2	21.12	1	21.12	24.92	0.0025
X_{12}	129.72	1	129.72	153.12	< 0.0001

Obtained results, given in Table 4, indicated that some of variables (X_1, X_2, X_1X_2 and X_{12}) affected the ammonium nitrogen removal efficiency. The X_{12} (Mg/N) was the major influent for removal of ammonium nitrogen efficiency due to the low *p*-value and high F value.

Two factors (A and B) at a particular point in the design space were compared in the perturbation plot shown in Fig. 1. The perturbation plot at the center point (Mg/N: 1.25 and N/P: 1.10) was obtained to show the relative effect of the two chosen variables as 'one factor at a time' on ammonium nitrogen removal. As it can be clearly seen in Figure 1, Mg/N (A) has higher influential effect (steepest slope) on ammonium nitrogen removal efficiency than that of the N/P (B).
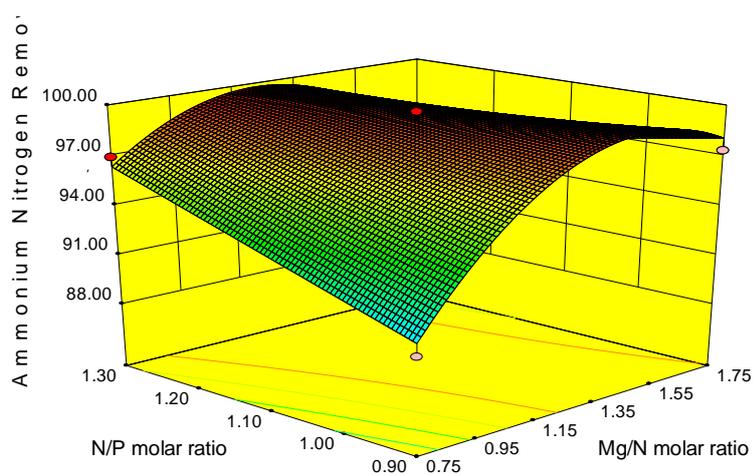

Figure 1:
General figure Perturbation Plot, A (Mg/N) and B (N/P) for ammonium nitrogen removal

### 3.2. Analysis of Response Surface

The relationship between response and experimental levels of two variables as well as their interactions for ammonium nitrogen removal efficiency within the experimental ranges is shown in Figure 2. The shapes of the contour plots could be circular or elliptical. While circular

contour plot shows that the interactions between the corresponding variables are negligible, elliptical shape indicates that the interactions are significant (Zhong and Wang, 2010).

Figure 2 showed the response surface and counter plots at various Mg/N and P/N molar ratio. The response curves demonstrated that higher ammonium nitrogen removal efficiency was observed at higher N/P molar ratio. The response curves were comparatively smooth at low Mg/N molar ratio, indicating the limited effect by the Mg/N molar ratio increases when N/P molar ratio was increased from 0.90 to 1.30. However, the ammonium nitrogen removal efficiency decreased with the further increase of Mg/N molar ratio. Higher ammonium removal efficiencies were observed at relatively higher N/P molar ratio and also at central value of Mg/N molar ratio.

a)
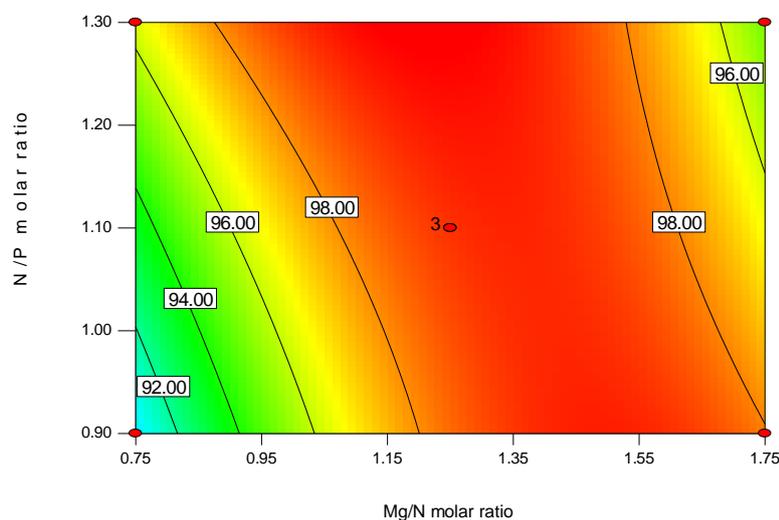
b)

Figure 2:
The effect of Mg/N and N/P on ammonium nitrogen removal (pH=9.2)
a. Response surface b. Counter plots

3.3. Process Optimization

The main problem of MAP precipitation is consumed high amount of phosphate and magnesium salts, resulting in high operational cost. Thus, to reduce of phosphate and magnesium consumptions during MAP formation is an important step and MAP chemicals usage needs to be optimized.

Obtained model can be used as determination of the optimum values of independent variables for ammonium nitrogen removal by MAP precipitation. The optimum values are at where all parameters simultaneously meet the targeted removal point.

Optimization procedure was conducted using numerical optimization option in the software. In optimization, the desired goal for the response was chosen as maximum and the variables of process parameters were selected to be within the in range. Selection constraints for each parameter were summarized in Table 5.

Table 5. Selection constraints for each parameter in optimization

Parameter	Goal	Upper Limit	Lower Limit	Importance
Mg/N molar ratio	In range	0.75	1.75	3
N/P molar ratio	In range	0.90	1.3	3
NH_4-N (%)	Maximize	87.6	99.6	3

The desirability function can be used to combine multiple responses into one response called the "desirability function" by choice of value from 0 (one or more independent variables are unacceptable) to 1 (all independent variables are on target). In this study, desirability function value was found as close to 1 for the optimum conditions.

Table 6 shows some characterization of raw leachate and after sutrivite precipitation process. For anaerobic treatment process COD/N/P molar ratio is usually set to 300:5:1 (Yilmaz et al. 2012). Application of enhanced Mg^{2+} or PO_4^{3-} may increase ammonium removal efficiency. On the other hand, PO_4^{3-} overdose will increase effluent phosphate concentration as well as operational cost.

Table 6. Characterization of the landfill leachate before and after MAP precipitation

Parameter	Raw leachate	After MAP precipitation	Change (%)
pH	7.77	9.2	+ 18.4
NH_4-N (mg/L)	3890	7.78	- 99.8
PO_4-P (mg/L)	23.3	10.4	- 55.4
COD (mg/L)	27545	20768	- 24.6
BOD (mg/L)	17500	12860	- 26.5
SO_4^{-2} (mg/L)	1965	1156	- 41.2

In Table 6, it is clearly seen that to reach maximum ammonium removal efficiency, optimum COD and NH_4-N concentration will be 20768 mg/L, 7.78 mg/L, respectively, showing that COD was an important limiting factors to reach desired COD/N/P molar ratio. According to desired molar ratios, 346 mg/L of NH_4-N concentration in the system right after precipitation would be sufficient to overcome negative effects of NH_4-N concentration on process performance. Therefore, ammonium removal efficiency would be set to 91% in order to achieve this ratio. When the set value is used as 91% for ammonium removal efficiency in

optimization stage of RSM, stoichiometric ratio for Mg/N and N/P were found as 0.76 and 0.92, respectively.

MAP precipitation process using less MAP chemicals will be more desirable and affordable. This study confirmed that the process optimization had crucial importance on the less experimental runs and also on the quantity of the chemicals affecting the cost of precipitation.

4. CONCLUSION

The present paper focuses on the precipitation of MAP from landfill leachate having high ammonium content. RSM was used to optimize the process parameters such as Mg/P and N/P molar ratio effects on the ammonium removal efficiency. Ammonium removal efficiency can be predicted fitted model by a second order form of the quadratic polynomial equations consisting of independent variables with a high significant effect on response.

RSM can be used to optimize required ammonium removal efficiency for lower Mg/N and N/P molar ratio which affects the performance of pretreatment method designed for an anaerobic process having 300:5:1 ratio for COD/N/P. MAP precipitation process using less MAP chemicals will be more desirable and affordable. However, maximum ammonium removal efficiency was 99.8% at a molar rate of 1.20 for Mg/N and 1.27 for N/P for a constant 9.2 pH value in this study.

RSM can be applied for performance optimization of sutrivite precipitation to obtain an affordable and desirable solution as a pretreatment method prior to anaerobic treatment of landfill leachate.

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