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Optimization of process parameters in coagulation of landfill leachate by $Al_2(SO_4)_3$ and PACl

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Abstract

In this study, landfill leachate treatment by coagulation and optimization of process parameters were aimed. Alum ($Al_2(SO_4)_3$) and Poly Aluminum Chloride (PACl) were used as coagulants to remove total suspended solids (TSS) from landfill leachate, and coagulant dose, reaction time, and pH were optimized as process variables. The Box-Behnken, one of the response surface methodology designs, was used in modeling the coagulation process. The R^2 values were very high (>95%) for TSS removal and the models were sufficiently in good agreement with experimental results. The TSS removal efficiencies in coagulation processes with alum and PACl under optimum process conditions determined by the model were 62.1% and 76.4%, respectively while the experimental values under optimum operating conditions were 60.8% and 75.1% in alum and PACl coagulation processes, respectively. According to the results of the study, both coagulation processes were effective in TSS removal from landfill leachate, and response surface methodology is a useful tool for optimizing the treatment parameters. The removal efficiency of the coagulation process conducted with PACl is higher than that of the alum process. Thus, it can be inferred that PACl is more effective under optimized conditions in this study.

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1. Introduction

Landfilling is the most widely used method in the disposal of solid wastes all over the world. Leachate, which has a high pollution load, occurs through rainwater in landfill storage areas [1]. The content of the leachate has a highly variable structure in terms of pollutant types and concentrations. Leachate contains a high quantity of organic matter, ammonia, heavy metals, and a wide variety of toxic contaminants [2]. Factors affecting the leachate characteristics are the type and compression ratio of the waste, amount of precipitation, hydrology, design, and operating conditions of the site. Besides, the age of the landfill is one of the most affecting parameters of leachate content. Leachate that occurred approximately 10 years after storage has a stable characteristic and is characterized by very strong organic content, high ammonia concentration, and low biodegradability [3]. This leachate is stated as mature leachate and its biological treatment is almost impossible and poses a risk to the environment [3–5]. Efficient treatment of leachate should be conducted to lower the high concentrations of pollutants to acceptable levels for final discharge. Physicochemical methods are preferred for leachate with a low biochemical oxygen demand/chemical oxygen demand (BOD/COD) ratio and high concentrations of toxic components [6, 7].

Landfill leachate treatment is one of the most important issues in solid waste landfilling. Treatment of leachate with a complex structure is very difficult with a single method. Besides, an appropriate treatment method should be applied to the leachate, which has varying characteristics depending on the stored waste, compression degree, age, and design of storage area, season, and climate conditions. In the selection of the leachate treatment system, parameters such as the characteristics of the leachate, the storage age, discharge criteria, the efficiency of the treatment system, the leachate flow rate, investment and operating costs, the need for qualified personnel, and the land requirement should be considered [8]. Biological methods (aerobic, anaerobic), physicochemical methods (chemical treatment, oxidation, adsorption, reverse osmosis, ammonia stripping) and combined systems are among the processes applied in the treatment of leachate. The biological treatment process is usually the first step of this combination and is followed by physicochemical methods [9].

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Coagulation and flocculation are some of the most preferred physicochemical methods in leachate treatment [10–12]. Coagulation is a comparatively simple and controllable pre-treatment or post-treatment method in leachate treatment to provide more biodegradability [13]. However, optimization of process parameters such as pH, optimum coagulant dosage, reaction time, and selection of appropriate coagulant is essential in the design of a coagulation process in leachate treatment [14]. The traditional one factor at a time optimization method is both time and energy-consuming approach. It is also a lack of the evaluation of interactions between variables. Thus, a better alternative such as response surface methodology (RSM) comes into prominence. Box-Behnken experimental design is one of the RSM designs and it provides to determine the interactive effects of variables and their impact factor [15]. Among the RSM designs, the minimum number of experimental sets is provided by applying the Box-Behnken design [16, 17].

The purpose of this study was to investigate total suspended solids (TSS) removal from landfill leachate by chemical coagulation. In this study, the efficiency of two different coagulants, namely, Alum and Poly Aluminum Chloride were investigated. The effects of independent variables on the coagulation processes were optimized by the Box-Behnken experimental design, which provides optimum process conditions with a minimum number of the experimental run. In the coagulation processes conducted with Alum and Poly Aluminum Chloride, optimum values of initial pH, coagulant dose, and treatment time were determined for TSS removal from landfill leachate.

2. Materials and Methods

2.1. Landfill leachate

The landfill leachate samples used in the study were obtained in October 2020 from Odayeri Landfill Leachate Treatment Plant, İstanbul, Turkey. Wastewater samples were stored at + 4 ºC to prevent biological activity.

Table 1. Characteristics of landfill leachate used in this study

Parameter	Value
pH	8.09±0.015
COD, mg/L	13100±26.7
Conductivity, mS/cm	37.4±0.15
TSS, mg/L	1110±47.5

The wastewater characterization before and after treatment was carried out using methods recommended by APHA [18]. The characteristics of landfill leachate used in this study are given in Table 1.

2.2. Experimental design and analytical methods

The solutions (10 g/L) of alum ($Al_2(SO_4)_3$) and poly aluminum chloride (PACl) were prepared and used in the experiments. After pH adjustment with 6 N H_2SO_4 and 6 N NaOH solutions, 100 ml of wastewater sample was put into a 250 ml beaker and placed in the Jar-test apparatus. Then the coagulant dosage determined by the model was added to the wastewater. The samples were rapidly mixed at 200 rpm for 2 minutes. Slow mixing was conducted at the desired value of the model at 45 rpm. The samples were settled for 30 minutes after the reaction time ended. The supernatant was separated for TSS analysis. TSS analysis was carried out according to standard methods and the removal efficiency was calculated according to Equation 1.

$$RE = \frac{C_0 - C}{C_0} \times 100 \quad (1)$$

where C_0 and C (mg/L) were the TSS of the samples before and after the treatments, respectively.

Response surface methodology provides the optimization of process variables with a minimum number of experiments. In this study, the Box-Behnken design of RSM was applied for the modeling of the coagulation process for TSS removal in landfill leachate using alum and PACl coagulants. Design Expert 11.1.0.1 software was used for modeling process variables. The levels and ranges of the independent variables are given in Table 2.

15 experimental sets for each coagulant were conducted with three levels of RSM and three independent variables. The ranges for pH, dosage, and reaction time were 6-10, 1-5 g/L, and 15-45 minutes, respectively. The ranges and coded values were determined from the results of the preliminary studies.

Table 2. The levels and ranges of the variables of the experimental design matrix

Coagulant	Factor	Levels and ranges		
		-1	0	1
Alum	A-pH	6	8	10
	B-Dosage, g/L	1	3	5
	C-Reaction Time, min.	15	30	45
Coagulant	**Factor**	**Levels and ranges**		
		-1	0	1
PACl	A-pH	6	8	10
	B-Dosage, mg/L	1	3	5
	C-Reaction Time, min.	15	30	45

Equation 2 shows the interaction among the independent variables (pH, dosage, and reaction time) and the response (TSS removal). In this equation, Y is the response (TSS removal); b_0 is the constant; b_i, b_{ii}, and b_{ij} are the coefficients of the linear, quadratic, and interaction effects respectively; X_i and X_j are the independent variables; n is the number of independent variables, and e is the prediction error.

$$Y = b_0 + \sum_{j=1}^{n} b_j x_j + \sum_{j=1}^{n} b_{jj} x_j^2 + \sum_{i} \sum_{<j=2}^{n} b_{ij} x_i x_j + e_i \qquad (2)$$

3. Results and Discussion

The regression equations for TSS removal by coagulation using alum and PACl are presented in Equations 3 and 4, respectively. The signs of the coefficients can be used to identify the synergistic (positive sign) and antagonistic (negative sign) effects of the variables. In the coagulation process conducted with alum, TSS removal efficiency increases with the increase in dosage and reaction time while it decreases with increasing pH value. Besides, in the coagulation process conducted with PACl, TSS removal efficiency increases with the increased pH, dosage, and reaction time.

Alum;

$$TSS\ removal, \% = +43.87 - 3.90 \cdot A + 17.76 \cdot B + 3.50 \cdot C - 0.47 \cdot A \cdot B - 0.8869 \cdot A \cdot C + 1.55 \cdot B \cdot C + 5.86 \cdot A^2 - 13.1 \cdot B + 2.39 \cdot C^2 \qquad (3)$$

PACl;

$$TSS\ removal, \% = +65.69 + 3.58 \cdot A + 11.22 \cdot B + 6.99 \cdot C + 1.71 \cdot A \cdot B + 1.22 \cdot A \cdot C - 1.05 \cdot B \cdot C - 5.73 \cdot A^2 - 3.50 \cdot B^2 - 5.19 \cdot C^2 \qquad (4)$$

In the coagulation process applied for TSS removal from leachate, 15 sets of analyzes were performed for each coagulant. Experimental design matrix obtained for coagulation processes, removal efficiencies estimated using the model, and obtained from the experiments are given in Table 3. The TSS removal efficiencies from landfill leachate vary between 13.6-60.6% in the alum coagulation process while it ranged from 35.7 % to 76.2% in the PACl coagulation process. Normal probability plots drawn to check the normality of the data are given in Figure 1a and Figure 1c for alum and PACl, respectively.

Table 3. Box-Behnken Design matrix with predicted and actual TSS removal ratios in coagulation process with alum and PACl coagulants

Run	Factors			TSS removal with Alum, %		TSS removal with PACl, %	
	pH	Alum or PACl dosage, g/l	Reaction time, min	Actual	Predicted	Actual	Predicted
1	6	1	30	19.8	22.3	45.0	43.4
2	10	1	30	13.6	15.5	47.7	47.1
3	6	5	30	60.6	58.8	61.8	62.4
4	10	5	30	52.5	50.0	71.3	72.9
5	6	3	15	52.3	51.6	45.8	45.4
6	10	3	15	45.6	45.6	51.6	50.2
7	6	3	45	60.4	60.4	55.5	56.9
8	10	3	45	50.2	50.8	66.2	66.6
9	8	1	15	15.3	13.5	35.7	37.7
10	8	5	15	43.4	45.9	62.5	62.3
11	8	1	45	19.8	17.4	53.6	53.8
12	8	5	45	54.1	55.9	76.2	74.2
13	8	3	30	46.7	43.9	64.2	65.7
14	8	3	30	43.4	43.9	68.5	65.7
15	8	3	30	41.5	43.9	64.4	65.7

Whether the residuals show a normal distribution can be interpreted according to the straightness of the line on the plot [19–21]. The data show the normal distribution and the normality assumption was confirmed as can be seen from the figures. The removal efficiencies obtained from the experimental studies under the conditions determined by the model versus (vs.) the removal efficiencies estimated by the model are shown in Figures 1b and 1d for alum and PACl, respectively. The obtained lines and high R^2 values show the agreement between the experimental and estimated removal efficiencies.

Table 4. ANOVA results for TSS removal by alum coagulant

	Sum of Squares	Df	Mean Square	f	p
Model	3595.51	9	399.50	37.81	0.0005
A-pH	121.46	1	121.46	11.50	0.0194
B-Dosage, g/L	2522.64	1	2522.64	238.78	< 0.0001
C-Reaction time, min	97.77	1	97.77	9.25	0.0287
AB	0.8838	1	0.8838	0.0837	0.7840
AC	3.15	1	3.15	0.2978	0.6087
BC	9.55	1	9.55	0.9038	0.3854
A^2	126.82	1	126.82	12.00	0.0180
B^2	633.39	1	633.39	59.95	0.0006
C^2	21.01	1	21.01	1.99	0.2175
Residual	52.82	5	10.56		
Lack of Fit	38.98	3	12.99	1.88	0.3662
Pure error	13.85	2	6.92		
Cor Total	3648.33	14			
Std. Dev.	3.25				
Mean	41.28				
C.V. %	7.87				
R^2	0.9855				
Adjusted R^2	0.9595				
Adeq Precision	17.6895				

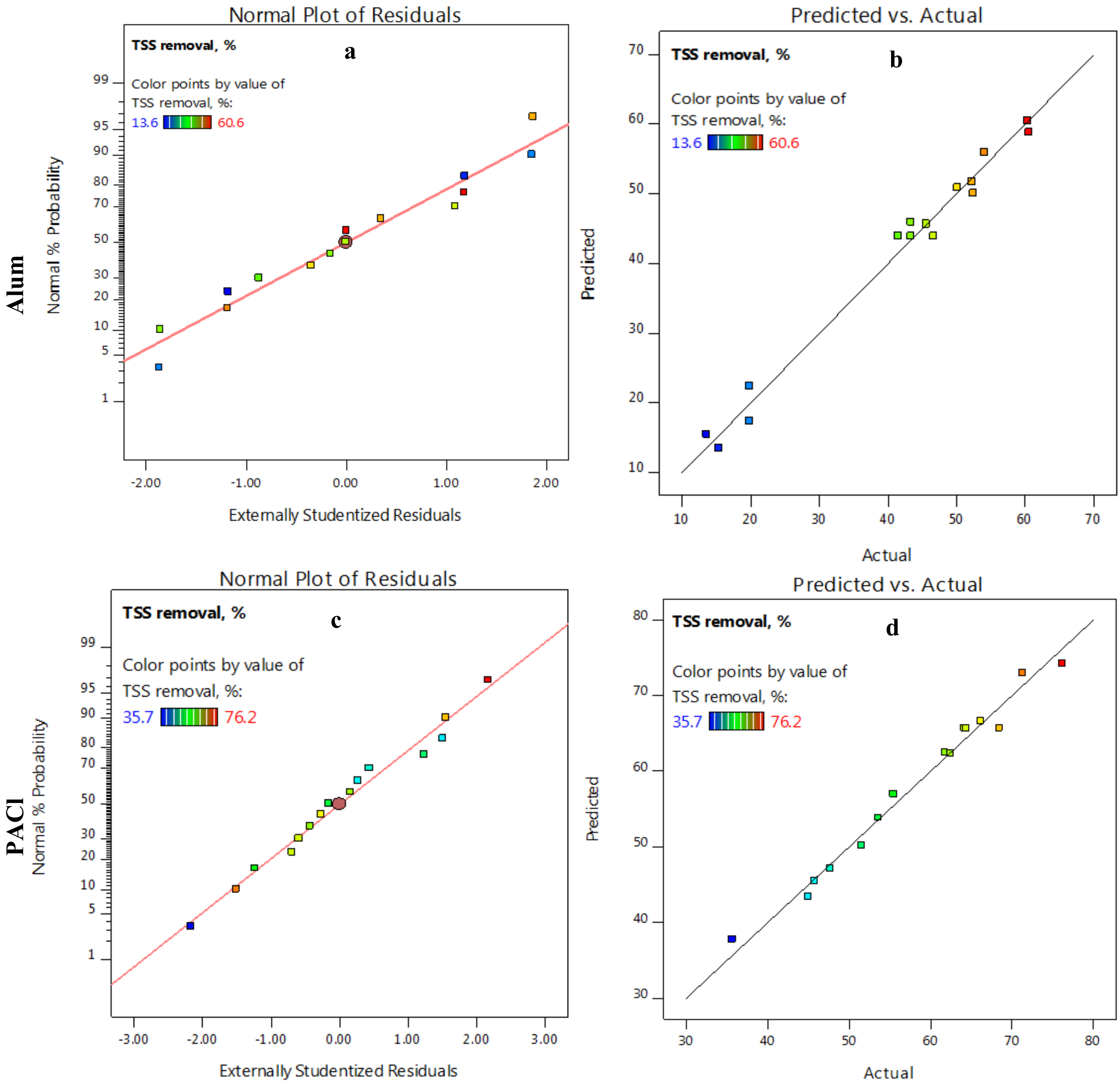

Figure 1. Normal probability plots and predicted versus actual values plottings for alum and PACl coagulants

The model was statistically evaluated by conducting the Analysis of Variance (ANOVA) analysis. The ANOVA results of the coagulation process conducted with alum and PACl are shown in Table 4 and Table 5, respectively. Various coefficients are calculated and evaluated to check the model suitability. The coefficients used for this purpose are coefficient of determination (R^2), adjusted R^2, the coefficient of variance (CV), and adequate precision (AP). The conformity of the model is checked by R^2 values and closeness to 1 indicates stronger and better predictive models. The R^2 values obtained for TSS removals indicate that most data variations can be explained by the model. Besides, the Adjusted R^2 values close to the R^2 indicate the significance of the model. Adjusted R^2 values in this study were found close to R^2 for both coagulants (Table 4 and Table 5). The CV value is an effective parameter used in evaluating the reproducibility feature of the model, and the CV value is required to be lower than 10 to interpret the model reproducible [22, 23]. Adequate precision value, which is used to measure the signal-to-noise ratio, is required to be 4 and above [24, 25]. As a result of the variance analysis, CV values obtained for TSS removal with both coagulants were found to be lower than 10 and adequate precision values were higher than four. These ratios also show that the models are reproducible for both processes.

Table 5. ANOVA results for TSS removal by PACl coagulant

	Sum of Squares	Df	Mean Square	f	p
Model	1754.97	9	195.00	31.62	0.0007
A-pH	102.67	1	102.67	16.65	0.0095
B-Dosage, g/L	1007.11	1	1007.11	163.31	< 0.0001
C-Reaction time, min	390.60	1	390.60	63.34	0.0005
AB	11.70	1	11.70	1.90	0.2269
AC	6.00	1	6.00	0.9733	0.3692
BC	4.41	1	4.41	0.7151	0.4363
A^2	121.11	1	121.11	19.64	0.0068
B^2	45.29	1	45.29	7.34	0.0423
C^2	99.35	1	99.35	16.11	0.0102
Residual	30.83	5	6.17		
Lack of Fit	19.23	3	6.41	1.10	0.5075
Pure error	11.60	2	5.80		
Cor Total	1785.80	14			
Std. Dev.	2.48				
Mean	58.00				
C.V. %	4.28				
R^2	0.9827				
Adjusted R^2	0.9517				
Adeq Precision	17.9593				

The effect of process variables on TSS removal by coagulation process is shown on 3-D plots in Figure 2. The pH value was selected between 6 and 10 in both alum and PACl coagulation processes. It was observed that the removal efficiency decreased as the pH value increased in the coagulation process carried out with alum. On the other hand, the removal efficiency increased as the pH value increased in the coagulation process conducted with PACl. The TSS removal efficiency increased with increasing coagulant dosage in both processes. The removal of pollutants in the coagulation process is explained by the charge neutralization mechanism, and the negative charges in the particles are neutralized by the addition of cationic coagulants [26]. As the age of landfill increases, the amount of negatively charged particles in the leachate increases, thus the amount of coagulant required to neutralize these particles increase [27]. The increase in TSS removal efficiency from landfill leachate in this study can be explained by this condition. In the coagulation process reaction time between 15 and 45 minutes was modeled. In both processes conducted using alum and PACl, the TSS removal efficiency was increased depending on the increase of treatment time.

The optimum values of process variables for alum and PACl coagulation processes to obtain maximum TSS removal efficiencies were determined by the models. Maximum TSS removal efficiencies were selected to determine the optimum conditions of the independent variables. The target values of three independent variables of both processes were selected within the range and the optimum conditions are given in Table 6. For the coagulation process conducted with alum, the optimum pH was 6.47, the dosage was 4.29 g/L and the reaction time was 41.83 minutes. The optimum conditions for the PACl coagulation process were as follows: pH 9.12, dosage 4.89 g/L, and the reaction time 37.9 minutes. Under optimum conditions, the TSS removal efficiencies estimated by the model were 62.1 % and 76.4 % for alum and PACl processes, respectively. The removal efficiencies obtained from the experimental studies conducted under optimum conditions to verify the model suitability and the accuracy of the optimization method were found to be 60.8 % and 75.1 % for TSS removal in alum and PACl processes, respectively. The proximity of the removal efficiencies estimated by the model to the obtained results of experimental studies confirms the suitability and accuracy of the approach in the models. The flow rate of landfill leachate in landfilling sites is closely related to precipitation, surface run-off, and infiltration of groundwater percolating through the landfill [5]. The fluctuation in the flow rate of landfill leachate may affect the coagulation process in terms of the amount of used coagulant. Thus, the changes in flow rate should be considered in the operation of the coagulation process.

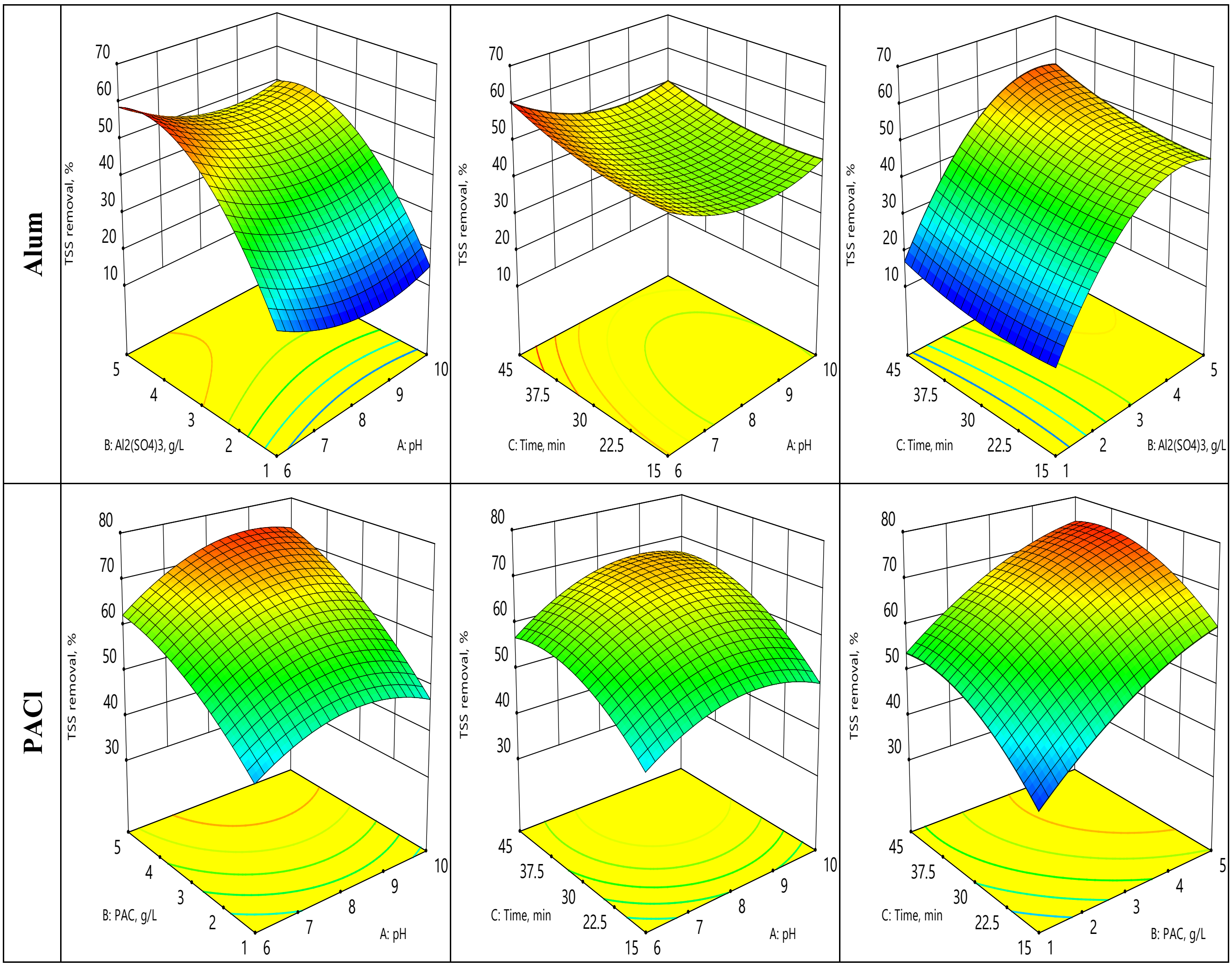

Figure 2. 3-D plots of the effects of independent variables on TSS removal with alum and PACl coagulants

Table 6. Optimum operating conditions and TSS removal efficiencies of alum and PACl

Factor	Optimum Conditions	
	Alum	PACl
pH	6.47	9.12
Reaction time, min	41.83	37.9
Dosage, g/L	4.29	4.89
Predicted removal efficiency, %	62.1	76.4
Experimental Removal Efficiency, %	60.8	75.1

3. Conclusions

This study investigated the optimization of process variables for TSS removal from landfill leachate by chemical coagulation. The Box-Behnken design was applied for modeling and optimization of the performance of the coagulation with alum and PACl coagulants. The optimum pH, reaction time, and coagulant dosage were determined to provide maximum TSS removal from leachate. The high correlation coefficient values show that the models were in good agreement with the experimental data for both coagulation processes. According to optimum conditions, optimum pH was 6.47, the dosage was 4.29 g/L and the required reaction time was 41.83 min for the coagulation process conducted with alum. Besides, the optimum conditions in the coagulation process conducted with PACl were pH 9.12, dosage 4.89 g/L, and reaction time 37.9 minutes. The experimental TSS removal efficiencies of coagulation with alum and PACl were 60.8 % and 75.1 %, respectively. The removal efficiencies showed that both coagulation processes were effective in TSS removal from landfill leachate. Besides, the results showed that under optimum conditions in this study PACl is more effective than alum in TSS removal from landfill leachate. As a result, coagulation can be applied as a pretreatment method for TSS removal from landfill leachate, and the response surface methodology is a useful tool for optimizing the process variables.

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Conflicts of interest

The author states no conflict of interests

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