



## Optimization of coagulation–flocculation process conditions using the central composite design for pretreatment of Moroccan landfill leachate

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### ABSTRACT

In this article, we used response surface methodology to determine the best conditions for leachate treatment using coagulation–flocculation (CF) with calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ). The leachate is collected in a public landfill of the Fez City in Morocco. Hence, the central composite design was employed to optimise the values of the input parameters, namely pH, calcium hydroxide (CH) concentration, fast stirring time and slow stirring time. Additionally, turbidity, absorbance and sludge volume, which all characterize the leachate treatment efficiency, are considered in this study as the PCC design responses. Using the investigated quadratic model, we were able to establish a perfect relationship between these responses and the four input parameters. In fact, the results of the analysis of variances ( $p$ -value < 0.05) show that the four factors, including CH quantity and pH, have a significant impact on the three responses. Moreover, with the help of the desirability function and the isoresponse curves, we succeeded in obtaining the optimal values for pH, CH Concentration and the two stirring times (speed and slow) of 11.5, 13 g L<sup>-1</sup>, 25 and 11.5 min, respectively. With the use of such optimal conditions allowed, in a control test, for a reliable and efficient pollutant elimination, as measured by turbidity, absorbance reduction at 254 nm, and sludge volume after treatment of the order of 12 NTU, 80%, and 48 ml L<sup>-1</sup>, respectively.

**Keywords:** Coagulation–flocculation; Response surface; Landfill leachate; Calcium hydroxide; Central composite plan

### 1. Introduction

Nowadays, landfill leachates (LL) are one of the most critical environmental issues faced by countries around the world [1,2]. They result from the percolation of rain water through layers of solid waste, to which is added water from biochemical processes in waste's cells and water that comes from wastes themselves [3]. They are therefore highly charged with organic and mineral matter, which requires treatment before discharge to the receiving environment [4].

Many studies showed that biological technologies have a cost-effective method to treat LL, especially with a combination between anaerobic and aerobic systems, but the large periods of hydraulic detention and the high volume of sludge produces represent a real limit for biological treatments [5]. LL components can be removed by various physicochemical processes. Advanced chemical oxidation by Fenton process [6] might be a good method to treat LL, but high process costs would be required [5]. A combination of electrocoagulation [7], adsorption [8], evaporation

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[9] and membrane processes [10] were used in large scale, but the cost was expensive, and it was not considered suitable for an industrial scale [5].

The coagulation–flocculation (CF) process is among the widely used methods of water and industrial wastewater treatment for its simplicity and low cost compared to other traditional techniques. Aluminum sulfate ( $\text{Al}_2(\text{SO}_4)_3$ ), poly-aluminum chloride ( $\text{Al}_n(\text{OH})_m\text{Cl}_{3n-m}$ ) and ferric chloride ( $\text{FeCl}_3$ ) are previously used as coagulants for LL pretreatment or in combination with biological methods [11,12]. The optimization of the coagulation–flocculation significant variables through the classical method involves the changing of one parameter at a time while fixing all other parameters at one level and studying the effect of the variable on the response. This is an extremely time-consuming, expensive, and complicated process for a multi-variable system. In this research, calcium chloride ( $\text{Ca}(\text{OH})_2$ ) was used for the treatment of landfill leachate in coagulation–flocculation process and the response surface methodology (RSM) was employed for the optimal experimental design of CF process to overcome of optimization parameters difficulty. The RSM technique can improve product yields and provide closer confirmation of the output response toward the nominal and target requirements [13,14]. The experiments were carried out by jar test which is usually employed to evaluate the treatment process efficiency. A preliminary study on the effect of coagulant concentration, pH, mixing speed, and the time, temperature, and settlement time on the coagulation–flocculation process was carried out in order to determine the most critical factors and their region of interest.

The main objective of this work was to optimize the coagulation–flocculation process and investigate the interactive effects of experimental chosen factors, including

coagulant concentration at  $\text{g L}^{-1}$ , pH, speed and slow agitation in minute. For this purpose, a Moroccan landfill leachate sample was selected as the target to be treated by the coagulation–flocculation process which was optimized by response surface methodology. The turbidity of treated water, the reduction absorbance at 254 nm and the sludge volume were chosen as the dependent output variables. The compromise optimal conditions for the three responses were also obtained using the desirability function approach and the design space.

## 2. Experimental

### 2.1. Materials and methods

Calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) from Sigma Aldrich, ACS reagent,  $\geq 95\%$  was used in powder as a coagulant. The landfill leachate used in this study was collected from the urban sanitary landfill of Fez City in Morocco, located at 15 km from Fez center ( $34^\circ 00' 16.6'' \text{ N}$ ,  $4^\circ 55' 44.5'' \text{ W}$ ). All samples were stored at  $4^\circ\text{C}$  in specific opaque bottles.

The CF experiments were carried out using the jar tests in 1 L beakers. The testing equipment is composed of four stirred reactors (Flocculator Fisher 1198). The coagulant was added with varying concentration from 7.3 to  $13.3 \text{ g L}^{-1}$ . The pH was adjusted to 9.5–11.5 by using 0.1 and 1 M HCl with GLP 22 CRISON pH-meter. The mixture was immediately stirred at two stages, speed agitation at 250 rpm from 5 to 25 min, followed by a slower stirring at 50 rpm from 10 to 30 min. Lastly, a settlement step was fixed at 2 h and the sludge volume in  $\text{mL L}^{-1}$  was estimated by using an Erlenmeyer of 1 L. Turbidity in nephelometric turbidity units (NTU) was measured with Hanna HI 88713 Turbidimeter and reduction absorbance at 254 nm using UviLine 9400 was calculated as follows:

$$\text{Removal absorbance \%} = \frac{\text{Initial absorbance} - \text{Residual absorbance after treatment}}{\text{Initial absorbance}} \times 100 \quad (1)$$

\*All analytical results were performed in triplicate.

### 2.2. Central composite experimental design

The central composite plan (CCP) was selected to optimize the values of the four independent parameters in coagulation–flocculation process using response surface methodology (RSM): pH ( $X_1$ ), coagulant concentration in  $\text{g L}^{-1}$  ( $X_2$ ), speed ( $X_3$ ) and slow ( $X_4$ ) stirring in minutes. Their range and levels are given in Table 1. In addition, turbidity (NTU), reduction absorbance at 254 nm % and the sludge volume ( $\text{mL L}^{-1}$ ) were chosen as the CCP design responses. The number of experiments used in this study was 28, calculated using Eq. (2):

$$N = 2k(k-1) + C_0 \quad (2)$$

where  $N$  is the number of experiments and  $k$  is number of the studied factors.

The MINITAB Trial software was used to create the experiments and conduct the statistical analysis in this study.

The response variable was related to the Factors by a second-order linear regression model ( $Y_m$ ) as follows:

$$Y_m = b_0 + \sum_{i=1}^k b_i X_i + \sum_{i \leq j} b_{ij} X_i X_j + \sum_{i=1}^k b_{ii} X_i^2 + \varepsilon \quad (3)$$

where  $Y_m$  is the response variable to be modeled (represented as  $Y_1$  for turbidity,  $Y_2$  for sludge volume and as  $Y_3$  for removal absorbance);  $X_i$  and  $X_j$  are the independent parameters which influence  $Y_m$ ;  $b_0$  is the constant,  $b_i$  is the linear coefficient,  $b_{ii}$  represents the quadratic coefficient and  $b_{ij}$  is the interaction coefficient. The number of the independent variables was represented by  $i$  and  $j$  and the total number of the optimized factors is represented by  $k$  and  $\varepsilon$  is the term error [15]. The actual design of this work is presented in Table 3. The quality of fit of the empirical model is evaluated by an analysis of variance via the  $F$ -test at the 5% significance level, by the coefficient of determination  $R^2 > 0.8$  and the value of the adjusted  $R^2$  [16]. The main, interaction and quadratic terms are evaluated by a student's  $t$ -test; the significance of a term is rejected if  $p$ -value is greater than 5% [17].

### 3. Results and discussion

#### 3.1. Landfill leachate characterization

A characterization focused on the important parameters used in this study (Landfill leachate of Fez City in Morocco) is summarized in Table 2. The turbidity in NTU and the absorbance at 254 nm, which represents the absorption wavelength of organic matter, replaces chemical oxygen demand (COD), showed high values and should be treated before a final discharge to the environment. Furthermore, the 28 trials of the CCP design were conducted in a randomized way to get results of the three responses, which are grouped in Table 3.

#### 3.2. Statistical analysis

The results of analysis of variance test for the second-order response surface model is provided in Table 4 for turbidity, sludge volume and reduction absorbance at 254 nm. The *F*-test indicates a highly significant result since the *p*-values of the three models were less than 0.01, indicating that the variation of the three responses is accurately related with the variation of the four input parameters. In addition, the goodness of fit of the models was checked by the coefficient of determination (*R*<sup>2</sup> and adjusted *R*<sup>2</sup>). Thus, turbidity and sludge volume showed almost the same value of the determination coefficient (*R*<sup>2</sup>) 0.88 and 0.91 respectively indicates that the models do not explain only 12% and 9% of the total variation. The value of the adjusted determination model coefficient of both responses: turbidity and sludge volume (adjusted *R*<sup>2</sup>) 0.74 and 0.82 are also high to confirm a high significance of the models. Results of analysis of variance (ANOVA) for absorbance removal showed that the model is significant. While the *R*<sup>2</sup> = 0.84 is high as compared to other models of turbidity and sludge volume, besides the *R*<sup>2</sup> = 0.66 value is better as compared to other models.

Moreover, Table 5 shows the significance of the coefficients in the second-order linear regression models evaluated by using the student's *t*-test. The coefficients with a *p*-value less than 0.05 reveal that the parameters have a significant effect. Furthermore, several studies have shown that when the *t*-value is higher and the *p*-value is lower, the corresponding factors are significant in the regression model. As can be observed in Table 5, for turbidity and sludge volume, the significant terms in the model were the main effect of pH and *T*<sub>1</sub> and the second-order effect of [concentration]<sup>2</sup>, [*T*<sub>2</sub>]<sup>2</sup> and [pH × *T*<sub>1</sub>]

respectively. For absorbance removal, the main effect of *T*<sub>1</sub> followed by pH and the second order effect is an interaction of [Concentration]<sup>2</sup> and between pH and *T*<sub>1</sub>.

Likewise, it was apparent that a long speed agitation *T*<sub>1</sub> of the mixture was more important than initial pH, in order to well mix and adjust the samples pH [18]. Results showed that *T*<sub>1</sub> has a negative effect for sludge volume and reduction absorbance at 254 nm. Moreover, pH showed a positive effect except for turbidity. Previous researchers had reported that pH significantly influenced turbidity, sludge volume and absorbance removal efficiencies in the coagulation–flocculation process [19–21]. Given that regardless the effect of pH, a formation of metallic hydroxides in alkaline condition could improve the turbidity and absorbance removals [22]. Meanwhile, the second order variables and interaction between [concentration × concentration] and [pH × *T*<sub>1</sub>] had a significant effect on turbidity and sludge volume, but the reduction absorbance at 254 nm was less affected by [concentration × concentration], the same phenomena was observed by Bouaouine et al. [22].

The following regression equations are the empirical models in terms of significant parameters for:

- (a) Turbidity = 46.07 – 16.53pH + 17.86*T*<sub>1</sub> + 49.03 [Concentration]<sup>2</sup> – 26.75[*T*<sub>2</sub>]<sup>2</sup> – 14.44 × [pH × *T*<sub>1</sub>]
- (b) Sludge volume = 40.61 + 7.4pH – 8.68*T*<sub>1</sub> – 22.6 [Concentration]<sup>2</sup> + 13.9[*T*<sub>2</sub>]<sup>2</sup> + 6.64[pH × *T*<sub>1</sub>]
- (c) Reduction absorbance = 65.78 + 8.42pH – 11.68*T*<sub>1</sub> + 8.41[pH × *T*<sub>1</sub>] – 17.56 [Concentration]<sup>2</sup>

The equations can be used to express prediction of turbidity, sludge volume and reduction absorbance at 254 nm responses based on the dependent variables

Table 2  
Characterization of Fez City landfill leachate (LL)

Parameters	Values*
Color	Dark brown
Temperature (°C)	27 ± 2
Conductivity (mS cm <sup>-2</sup> )	3.3 ± 0.2
pH	8.1 ± 0.3
Turbidity (NTU)	560 ± 20
Absorbance (254 nm)	30.7 ± 0.4

\*Results were performed in triplicate.

Table 1  
Levels of the variables in the central composite plan

Parameters	Symbols	Levels		
		-1	0	+1
pH	<i>X</i> <sub>1</sub>	9.5	10.5	11.5
Coagulant concentration, (g L <sup>-1</sup> )	<i>X</i> <sub>2</sub>	7.3	10.3	13.3
Speed stirring, (min)	<i>X</i> <sub>3</sub>	5	15	25
Slow stirring, (min)	<i>X</i> <sub>4</sub>	10	20	30

Table 3  
Central composite design matrix and response results of 28 randomized runs

Run	Coded factors				Response		
	pH	Coagulant concentration (g L <sup>-1</sup> )	Speed stirring (min)	Slow stirring (min)	Turbidity (NTU)	Sludge volume (mL L <sup>-1</sup> )	Absorbance removal at 254 nm
1	-1	-1	-1	-1	23.12	45	73.2
2	+1	-1	-1	-1	2.06	55	98.1
3	-1	+1	-1	-1	7.35	55	94.7
4	+1	+1	-1	-1	33.19	40	63.4
5	-1	-1	+1	-1	90.2	14	42.5
6	+1	-1	+1	-1	26.46	42	70.4
7	-1	+1	+1	-1	31.98	40	65.2
8	+1	+1	+1	-1	25.67	43	71.9
9	-1	-1	-1	+1	10.42	50	92.5
10	+1	-1	-1	+1	0.97	63	99.5
11	-1	+1	-1	+1	1.47	60	98.7
12	+1	+1	-1	+1	4.61	55	96.3
13	-1	-1	+1	+1	93.4	10	40.3
14	+1	-1	+1	+1	24.19	45	71.8
15	-1	+1	+1	+1	102.6	10	40.7
16	+1	+1	+1	+1	3.52	60	97.5
17	-1	0	0	0	69	33	54.78
18	+1	0	0	0	5.58	54	95.1
19	0	-1	0	0	115	11	38.31
20	0	+1	0	0	89.6	20	50.12
21	0	0	-1	0	25.3	39.6	59.41
22	0	0	+1	0	37.7	41.8	67.7
23	0	0	0	-1	23.37	53	63.91
24	0	0	0	+1	29.66	51	70.81
25	0	0	0	0	33.97	45	72.1
26	0	0	0	0	34.60	42	70.5
27	0	0	0	0	36.84	46	71.5
28	0	0	0	0	35.7	44.5	73

Table 4  
ANOVA of quadratic models of turbidity, sludge volume and absorbance removal

Source	Turbidity			Sludge volume			Absorbance removal		
	DF	MS	F	DF	MS	F	DF	MS	F
Model	14	1,817.4	6.6	14	394.55	9.79	14	562.22	4.73
Error	3			3			3		
<i>p</i> -value	0.08%			0.01%			0.4%		
<i>R</i> <sup>2</sup>	0.88			0.91			0.84		
Adjusted <i>R</i> <sup>2</sup>	0.74			0.82			0.66		

\*DF (Degree of freedom): an estimate of the number of independent categories in a particular statistical test or experiment;

\*MS (Mean square): the mean square of a set of values is the arithmetic mean of the squares of their differences from some given value, namely their second moment of that value.

Table 5

Estimated values of the regression coefficients and their significance in the second-order multiple linear regression models for turbidity, sludge volume and absorbance removal

Term	Turbidity			Sludge volume			Absorbance removal		
	Coefficient	<i>t</i> -value	<i>p</i> -value	Coefficient	<i>t</i> -value	<i>p</i> -value	Coefficient	<i>t</i> -value	<i>p</i> -value
Constant	46.07	8.06	0.000	40.61	18.50	0.000	65.78	17.45	0.000
pH	−16.53	−4.24	<b>0.001</b>	7.40	4.95	<b>0.0003</b>	8.42	3.28	<b>0.006</b>
Conc.	−5.09	−1.31	0.21	3.04	2.03	0.063	3.43	1.34	0.2
$T_1$	17.86	4.58	<b>0.0005</b>	−8.68	−5.80	<b>0.0001</b>	−11.68	−4.55	<b>0.0005</b>
$T_2$	0.09	0.02	0.98	1.32	0.88	0.39	4.15	1.61	0.13
pH × Conc.	5.80	1.40	0.18	−3.74	−2.35	0.035	−3.23	−1.18	0.26
pH × $T_1$	−14.44	−3.49	<b>0.004</b>	6.64	4.18	<b>0.0011</b>	8.41	3.09	<b>0.0087</b>
pH × $T_2$	−6.47	−1.57	0.14	3.76	2.37	0.034	4.66	1.71	0.11
Conc. × $T_1$	−5.40	−1.31	0.21	3.24	2.04	0.062	3.16	1.16	0.27
Conc. × $T_2$	1.32	0.32	0.75	0.11	0.07	0.94	0.51	0.19	0.85
$T_1$ × $T_2$	5.74	1.39	0.19	−2.51	−1.58	0.14	−4.20	−1.54	0.15
pH × pH	−15.98	−1.55	0.14	5.40	1.37	0.2	13.16	1.94	0.075
Conc. × Conc.	49.03	4.77	<b>0.0004</b>	−22.60	−5.72	<b>0.0001</b>	−17.56	−2.59	<b>0.022</b>
$T_1$ × $T_1$	−21.77	−2.12	0.05	5.75	1.45	0.17	7.97	1.17	0.26
$T_2$ × $T_2$	−26.75	−2.60	<b>0.022</b>	13.90	3.52	<b>0.004</b>	5.58	0.82	0.43

\**t*-value: value calculated by the ratio of two sample variances. The *T* statistic can test the null hypothesis: (1) that the two sample variances are from normal populations with a common variance; (2) that two population means are equal; (3) that no connection exists between the dependent variable and all or some of the independent variables.

\**p*-value: is associated with a test statistic. It is the probability, if the test statistic really were distributed as it would be under the null hypothesis, of observing a test statistic [as extreme as, or more extreme than] the one observed

### 3.3. Simulation of landfill leachate treatment using surface response methodology

Interpretation of the results has to start from the study of the Derringer's desirability function. The optimum conditions coagulation–flocculation process were also estimated by use of Derringer's desirability function:

$$D = [d_1 w_1 \times d_2 w_2 \times \dots \times d_n w_n]^{1/n} \quad (4)$$

where  $w_i$  is the weight of the response,  $n$  the number of responses, and  $d_i$  the individual desirability function for each response. In this study, all  $w_i$  values were set equal to 1. Derringer's desirability function (*D*) can take values from 0 to 1. A value close to unity indicates that the combination of the different criteria is matched in a global optimum [23]. According to Fig. 1 shows the desirability plots corresponding to turbidity, sludge volume and removal absorbance responses for landfill leachate treatment using calcium hydroxide (CH) as coagulant in coagulation–flocculation process. The optimal treatment conditions were obtained (pH: 11.5; coagulant concentration: 13 g L<sup>−1</sup>;  $T_1$ : 25 min;  $T_2$ : 11.5 min) with a maximal desirability value of 0.82, with a lower turbidity of 12.5 NTU, sludge volume of 48 mL L<sup>−1</sup> and a maximum reduction of absorbance at 254 nm (80%)

Contour plots of the response surface methodology (RSM) are drawn as a function of two factors at a time, holding all other factors at fixed levels. Those plots clarify both the main and the interaction effects of these two factors. The 3D surface graphs, pH vs. coagulant concentration (g L<sup>−1</sup>) in Fig. 2A–C, show at fixed speed and slow

agitation of 25 and 11.5 min respectively, that a significant interaction occurs between coagulant concentration and pH for turbidity, sludge volume and reduction absorbance at 254 nm as responses. It is clear from the figures that the turbidity and sludge volume reduce (less than 20 NTU and 50 mL L<sup>−1</sup>) and the absorbance at 254 nm reaches the maximum removal around 80%, at optimal conditions, where coagulant concentration is around 13 g L<sup>−1</sup> and for a pH of 11.5 which is in accordance with the results reported by [24,25]. On the other hand, those results are in contradicts with [26] who had showed a turbidity removal of 65% with chloride hydroxide (CH) for optimal conditions (pH: 8; concentration: 25 g L<sup>−1</sup>), approximately in neutral conditions with a high (CH) amount (two times more).

### 3.4. Optimization analysis

The overlay plot was generated by superimposing the contours for the multiple response surfaces. By determining the desired limits of the turbidity, sludge volume and reduction absorbance at 254 nm at fixed speed and slow stirring (25 and 11.5 min respectively), the white area of the overlay plot defined the permissible values of the dependent variables as shown in Fig. 3. The optimum values of the test variables in actual were as follows; coagulant concentration (from 10 to a maximum of 13 g L<sup>−1</sup>), pH (from 10 to 11.5) while the responses predicted were turbidity at 10 NTU, sludge volume of 40 mL L<sup>−1</sup> and reduction absorbance at 254 nm of 80%.

Table 6 presents a verification of the results using the set of optimized factors accomplished by performing the

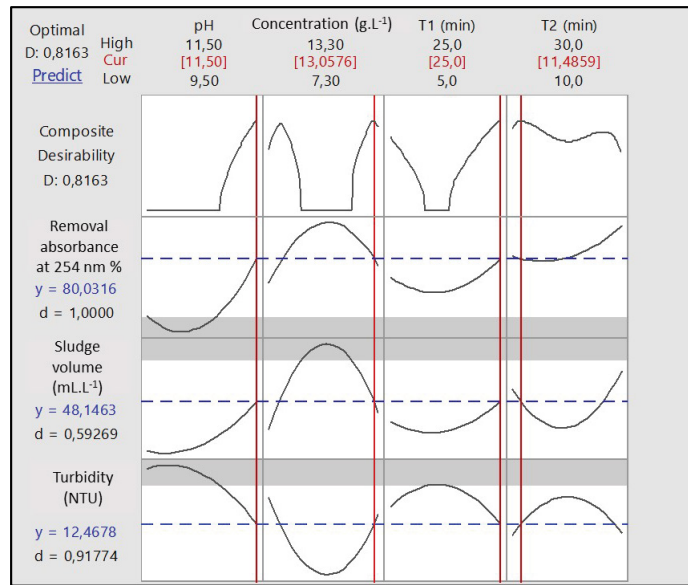


Fig. 1. Desirability plots of landfill leachate (LL) treatment responses using central composite plan (CCP).

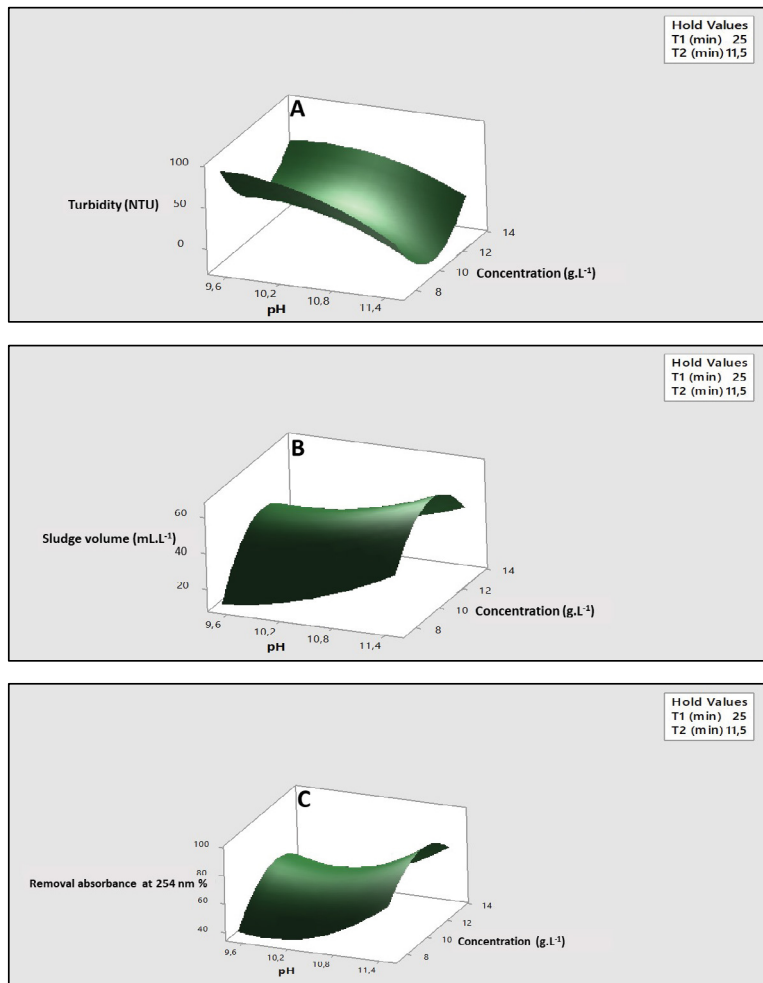


Fig. 2. 3D surface graph of (A) turbidity, (B) sludge volume and (C) reduction absorbance at 254 nm showing the effect of coagulant concentration in g L<sup>-1</sup> and pH at fixed speed and slow agitation.

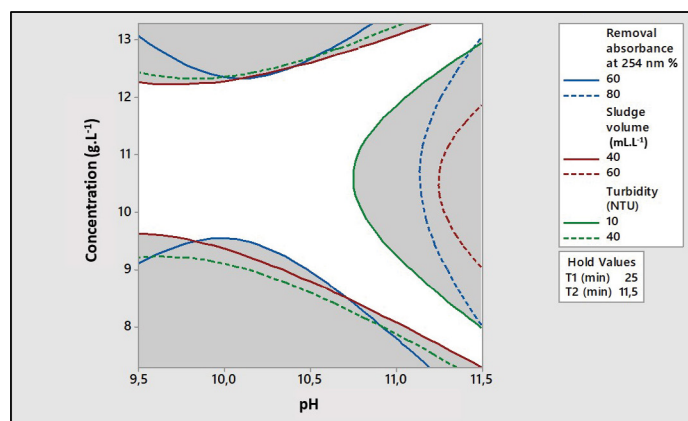


Fig. 3. Overlay plot for turbidity, sludge volume and removal absorbance for landfill leachate treatment by optimal conditions of pH and calcium hydroxide concentration.

Table 6

Simulated and experimental values of landfill leachate responses after optimal conditions treatment

Optimal conditions	Turbidity (NTU) value		Sludge volume (mL L <sup>-1</sup> ) value		Reduction absorbance at 254 nm	
	Experimental	Simulated	Experimental	Simulated	Experimental	Simulated
pH = 11.5						
CH concentration: 13 g L <sup>-1</sup>						
Speed stirring: 25 min	20.3 ± 5	12.5	52 ± 10	48	74 ± 3	80
Slow stirring: 11.5 min						

\*Experiments were conducted in triplicate.

experiments incorporating the optimized variables (pH: 11.5; calcium hydroxide (CH) concentration: 13 g L<sup>-1</sup>) at fixed speed and slow stirring at 25 and 11.5 min respectively. The average turbidity, sludge volume and reduction absorbance at 254 nm values obtained through the experiment was 20.3 ± 5 NTU, 52 ± 10 mL L<sup>-1</sup> and 74% ± 3 respectively. These experimental findings were in close agreement with the model prediction (Table 6). Let's note that effluent treatment at a basic pH of 11.5 increases the metal hydroxide deposits.

#### 4. Conclusion

The surface response methodology was employed in this study to adjust the experimental settings and improve the coagulation–flocculation process for treating Fez landfill leachate (LL). Thanks to a quadratic model relating the analyzed responses and the four optimized parameters, this research, which was carried out at the laboratory level using a central composite design and a smaller number of experiments, was able to get relevant results.

The best regression coefficients ( $R^2$ ) were obtained firstly for turbidity and sludge volume (0.88), followed by reduction absorbance at 254 nm (0.78). Using the desirability function and iso-response curve, we were able to demonstrate that the optimized values of the input parameters, namely pH, calcium hydroxide concentration, slow and fast stirring rates, had a clear influence on reducing turbidity, sludge volume to 20.3 ± 5 NTU and 52 ± 10 mL L<sup>-1</sup> respectively, while maximizing the reduction in absorbance at 254 nm to 74 ± 3%.

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