ISSN: 2277-3754
ISO 9001:2008 Certified
International Journal of Engineering and Innovative Technology (IJEIT)
Volume 4, Issue 3, September 2014

Optimized physico-chemical treatment of a fresh leach ate using a rejection of steel industry

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Abstract-Acoagulation flocculation process was used to treat fresh leachate landfill of city of Mohammedia with a combined Steel Industry Wastewater rich in ferric chloride (SIWW) (and polymer. A 4^2 central composite experimental design and response surface methodology were employed to evaluate and optimize the reagents dosage and to achieve a compromise between efficiency and operational costs. The influence of pH was also evaluated to determine the most suitable pH condition. The best regression coefficients (R^2) were obtained for chemical oxygen demand (COD) removal, biological oxygen demand (BOD_5) removal and turbidity removal, reaching values of 0.9452, 0.9333 and 0.9696 respectively. The most significant factors in the analysis of variance (ANOVA) in this study were pH and SIWW dosage for COD removal, BOD_5 removal and turbidity removals. However, flocculant dosage was not most significant factor. Multiple response optimizations fits the optimum values of the factors and the responses as 40 mL/L of coagulant and 12 mL/L of polymer and 52.93, 76.51 and 80.11 % of COD, BOD_5 and turbidity removal respectively at pH=6.

***Keywords*-Steel Industry; Wastewater valorization; physicochemical process; Fresh leachate landfill; Response surface methodology; central composite experimental design.**

I. INTRODUCTION

Land filling is one of the least expensive methods for disposal of solid waste. It is reported that about 90% of municipal solid waste (MSW) is disposed in open dumps and landfills unscientifically, creating problems to public health and the environment [1]. Leachates may contain large amounts of organic matter (biodegradable, but also refractory to biodegradation), where humic-type constituents consist an important group [2], as well as ammonia-nitrogen, heavy metals, chlorinated organic and inorganic salts [3]. Landfill leachates have been identified as potential sources of ground and surface waters contamination, as they may percolate through soils and sub soils. Leachates present considerable variations chemical composition [4-6]. If landfills are not properly managed, these can cause uncontrolled gaseous and liquid emissions. The landfill can be classified into three categories based on age: young, medium and old. Normally land filling commenced within 5 years is termed as young age landfill. It consists of large amount of biodegradable matters and a higher COD value of 20000 mg/l. [7] The leachate characteristics vary with time and from site to site because it depend on type of wastes disposed, rainfall, age of the landfill and design of the landfill etc [8]. The characteristics of the landfill leachates can usually be represented in terms of the parameters such as COD, BOD5, ratio of BOD5/COD, colour, pH, temperature, alkalinity, oxidation reduction potential and heavy metals [9]. The treatment processes used for landfill leachates often involve a combination of appropriate techniques. Coagulation/flocculation is an essential process in water and in industrial wastewater treatment. Several studies have been reported on the examination of coagulation–flocculation for the treatment of landfill leachates, aiming at performance optimization, i.e. selection of the most appropriate coagulants and flocculants determination of experimental conditions, assessment of pH effect and investigation of flocculant addition [10]. Coagulation and flocculation is a relatively simple technique that may be ployed successfully in treating old landfill leachates [11-13]. The advantages of the proposed physico-chemical method for the treatment of leachates are mainly simplicity, low cost, good removal efficiencies and easy onsite implementation. This method could be used for pre- or post-leachate treatment in combination with biological treatment process. As a result of the apparent inability of the method for sufficient pollutant removal, the cost of the high chemical dosages that are required, and the associated problems of the chemical sludge that is generated, it could be suggested that no single leachate treatment method, biological or physicochemical, is able to produce an effluent with acceptable quality, and that both approaches should be appropriately combined. The main objective of coagulation and flocculation process is remove turbidity of organic compounds and heavy metals from the leachate. Several authors have used response surface methodology and optimization to improve the coagulation–flocculation processes of wastewater of different origins [14-20]. These authors agree that the type and dosage of coagulant and flocculant reactants are decisive to the success of the coagulation–flocculation process. The aim of this study is to evaluate and optimize variables for using of Steel Industry Wastewater for a physico-chemical water treatment process of leachate resultant from compacting of solid waste (Mohammedia, Marocco). A statistically analyze experimental data is must enable a compromise between efficiency and operational costs of a real industrial process.

II. MATERIALS AND METHODS

A. Sample collection

The leachate studied is a fresh leachate resulting from compaction of solid waste in Mohammedia city (Marocco); it is recovered from a reservoir which is in the dump trucks. To make a collection of leachate, four

Garbage Compactor Trucks were chosen at random containing each approximately 5 tones of solid. The quick dump compactor consists of a single box-body loading hopper. The solid waste is loaded with note containers while waste compaction is by means of a compacting shovel actuated by a pair of double acting hydraulic cylinders. The box has a useful volume of 5 to 10 m³. The central and front parts form the loading volume, while the rear part of the inclined plate 6mm loading plane, is the loading hopper. The anterior chamber is equipped with a leachate collection system, so that no loss can occur in the corners and after shots of the driver brakes. It is possible to easily drain through a ball valve 2 "½. Using this faucet is made to recover the leachate in a can of 35 liter.

B. Chemicals and Materials

The physico-chemical characteristics of raw leachate produced by compaction of solid waste are reported in Table 1.

Table 1: The characteristics of raw leachate

Parameter	Value
pH	4.45
Conductivity (ms/cm)	11.57
Turbidity (NTU)	4000
Colour	1.5
COD (mg/L)	57 600
BOD_5 (mg/l)	6 800
Phenol (mg/L)	182
Surfactant (mg/L)	35.6
Settled volume (ml/l)	16
MES (mg/l)	6530

The Steel Industry Wastewater (SIWW) was taken from Maghreb steel (Morocco)) is rich in $FeCl_3$ and was used as a coagulant in this study. The characteristics of SIWW are given in Table 2. This rejection was valorized as coagulant in the treatment of leachate.

Table 2: Main characteristics of SIWW

Parameter	Value
FeCl3 (g/l)	101,3
Conductivity (ms/cm)	20,2
pH value	1,40

The flocculant used is an anionic polymer 35 %; its trade name is Hymoloc DR3000. The characteristics of this flocculant are shown in table 3.

Table 3: Characteristics of polymer Hymoloc DR3000

Appearance Milky	Value
Parameter	Weight High
Density	35%
Viscosity	3.0-4.1
pH	<600cp
Cationicity	1.2 g/cm3
Molecular	White Liq.

The experimental set-up used for the coagulation–flocculation experiments at laboratory scale consisted of a Jar-test device (Jar Test Flocculator FC -6S Velp Scientific) in which six stirring blades were connected to a motor that operated under adjustable conditions. The system permitted the experiments to be performed with ease and the different variables affecting the removal of suspended fat and organic matter to be interpreted such as pH, stirring time and speed, retention time or reactant concentrations.

C. Analytical procedure

The pH is one of the most restrictive parameters in the coagulation step and affects the hydrolysis equilibrium produced by the presence of the coagulant agent [21]. The pH of leachate was adjusted by addition of NaOH (40 %) to a desired value in the range of 5 – 7.7. Coagulant dosages (SIWW) varied in the range of 26–54 mL/L (equivalent to 2634 –5470 mg Fe^{3+}/L), while flocculant dosages (Hymoloc DR3000 0.3%) ranged from 5 to 19 mL/L. Sixteen experiments were carried out. After the addition of coagulant, the leachate was stirred at 160–180 rpm for 10 min. The flocculant was then added and the medium stirred at 40–50 rpm for 20 min. Samples were taken from the supernatant and analyzed after leaving the medium to stand for two hours. The samples of leachate were measured using a turbidimeter (Model HACH 2100N TURBIDIMETER). COD was determined by titrimetric method as described in standard Methods [22] and BOD_5 was determined by BOD meter. Removal efficiency of COD, BOD_5 and turbidity were obtained according to the formula given below:

$$\text{Removal } \% = \left(1 - \frac{C}{C0}\right) \times 100 \qquad \textbf{(1)}$$

Were C_0 and C are the initial and final concentration values.

D. Experimental design

A central composite rotatable design for *k* independent variables was employed to design the experiments [23] in which the variance of the predicted response, $\hat{Y}$, at some points of independent variables, *X*, is only a function of the distance from the point to the design centre. These designs consist of a 2^k factorial (coded to the usual ±1 notation) augmented by $2{*}k$ axial points ($\pm\alpha$, 0, 0), (0, $\pm\alpha$, 0), (0, 0, $\pm\alpha$), and 2 centre points (0, 0, 0) [24]. The value of α for rotatability depends on the number of points in the factorial portion of the design, which is given in Eq. (**2**):

$$\alpha=(N_F)^{1/4} \qquad \textbf{(2)}$$

Where N_F is the number of points in the cube portion of the design ($N_F = 2^k$, *k* is the number of factors). Since there are three factors, the N_F number is equal to 2^3 (=8) points, while α is equal to $(8)^{1/4}$ (=1.682) according to Eq. (**2**).

In this study, the responses were COD removal (Y_{COD}), BOD_5 removal (Y_{BOD5}) and turbidity removal (Y_{TUR}) of landfill leachate. Each response was used to develop an empirical model that correlated the response to the coagulation processes activated variables using a second-degree polynomial equation as given by Eq. (**3**) [25]:

$$\hat{Y} = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 \quad \mathbf{(3)}$$

Where β_o the constant coefficient, β_i the linear coefficients, β_{ij} the cross-product coefficients and β_{ii} the quadratic coefficients. The software JMP® 10 was used for the experimental design, data analysis, model building, and graph plotting.

III. RESULTS AND DISCUSSION

A. Development of the regression model equation

Preliminary experiments were carried out to screen the appropriate parameters and to determine the experimental domain. From these experiments, the effects of initial pH of leachate (X1), coagulant dosage in mL/L (X2) and flocculent dosage in mL/L (X3) are investigated on three responses: COD removal, BOD_5 removal and turbidity removal. The parameter levels and coded values were given in Table 4.

Table 4: Study field and coded factors.

Natural variables (X_j)	**Unit**	**Coded variables X_1, X_2, X_3***				
		A	**-**	**0**	**+**	**A**
x_1= initial pH	-	4,31	5	6	7	7,68
x_2= Coagulant dosage	mL/L	26,5	32	40	48	53,4
x_3= Flocculent dosage	mL/L	5,3	8	12	16	18,7

* The coded values $X_j = \pm 1$ are obtained by equation: $X_j = (x_j - \bar{x}_j)/\Delta$

The coefficient of determination R^2 in this study were relatively high, indicating a good agreement between the model predicted and the experimental values. Meanwhile, adjusted R^2 permitting for the degrees of freedom associated with the sums of the squares is also taken into account in the lack-of-fit test, which should be an approximate value of R^2. When R^2 and adjusted R^2 differ dramatically, there is a good chance that insignificant terms have been included in the model [26]. As shown in Table 5, the two R^2 values were not significantly different.

Table 5: Regression coefficient R^2 and adjusted R^2

	COD removal (%)	**BOD_5 Removal (%)**	**Turbidity Removal (%)**
R^2	0.94	0.93	0.97
R^2_{adj}	0.86	0.83	0.92

The experimental design matrix, the corresponding experimental parameters and response value were shown in Table 6.

Table 6: Experimental design and results for leachate removal.

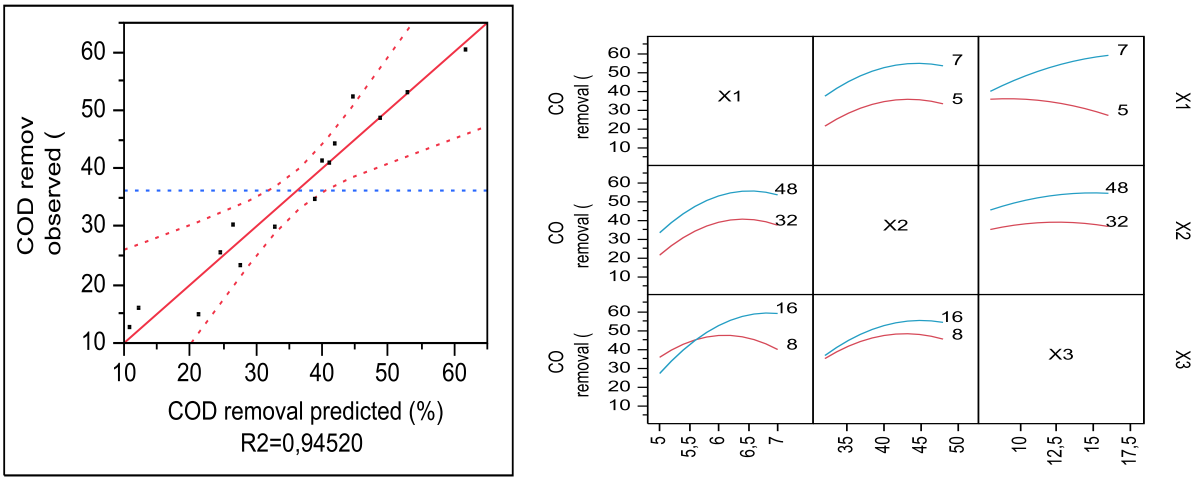
Configuration	**X_1**	**X_2**	**X_3**	**COD removal (%)**	**BOD_5 removal (%)**	**Turbidity removal (%)**
− − −	5	32	8	25	62	71
− − +	5	32	16	16	57	76
− + −	5	48	8	30	65	72
− + +	5	48	16	23	61	79
+ − −	7	32	8	30	65	73
+ − +	7	32	16	44	72	74
+ + −	7	48	8	35	67	87
+ + +	7	48	16	60	79	84
a00	4,32	40	12	12	56	59
A00	7,68	40	12	41	70	70
0a0	6	26,5	12	15	56	81
0A0	6	53,4	12	52	76	87
00a	6	40	5,3	41	70	80
00A	6	40	18,7	49	73	80

0	6	40	12	53	76	80
0	6	40	12	53	76	80

B. COD removal

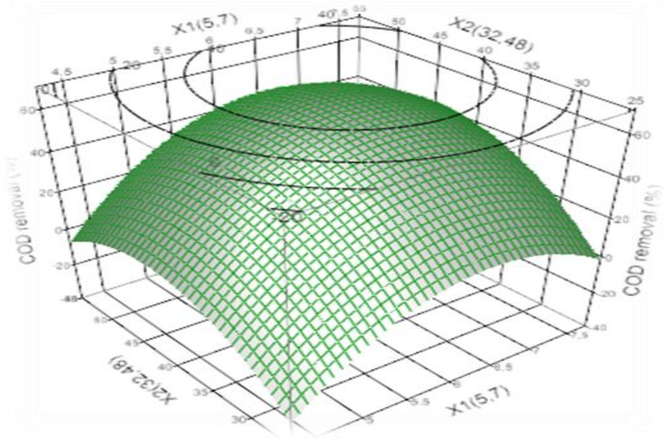
The regression coefficient (R^2=0.94) indicates the sample variation explained by the model. The plots of the experimental value versus the predicted value for COD removal are shown in Fig. 1 (a). The experimental values are distributed relatively near to the straight line. The analysis of variance test (ANOVA) for the response surface model is provided in Table 7. Since the p-value for the model was lower than 0.05, there was a statistical relationship between COD removal and the selected variables at a 95% confidence level. As can be observed in the ANOVA table (Table 7) and the main interactions plotted in Fig. 1 (b), pH and coagulant dosage was the most significant factors. However, COD removal response was found at a pH near neutrality and a high level of coagulant dosage led to a high COD removal percentage and a low COD removal percentage at acidic pH. Fig. 1 (b) shows the interaction between X1, X2 and X3 for COD removal. The interaction between this factors causes the most significant variation in COD removal as can be observed, for example, if we set the coagulant dosage and flocculant dosage at 40 and 16 mL/L respectively, DCO removal can be achieve 27 % at pH equal at 5, and 59 % at pH equal at 7, this last percentage may increase to 62 % if coagulant dosage is increased to 45 mL/L. These results show that the coagulation–flocculation mechanism differs depending on the pH value and dosage of coagulant. Several studies have reported the examination of this process for the treatment of industrial wastewater, especially with respect to performance optimization of coagulant/flocculant, determination of experimental conditions, assessment of pH and investigation of flocculant addition [27]. Fig. 1 (c) shows the three-dimensional response a surface which was constructed to show the effects of the coagulant dosage and the pH on the COD reduction of fresh leachate by coagulation processes with SIWW. The flocculant concentration was fixed at 12 mL/L. The optimum removal point (64.9 %) obtained at around dose 47 mL/L and initial pH 7.33. The decline in removal efficiencies is observed when moving away from this point, implying that neither increase nor decrease in any of the tested variables is desired.

Fig. 1 (a): Experimental values versus predicted values for the COD removal. (b): Main interactions plot for COD removal. (c): Response surface plots for COD removal as a function of pH (X1) and coagulant dosage (X2) at flocculant dosage equal at 12 mL/L.



(a)

(b)



(c)

Table 7: ANOVA for COD removal response surface models.

Source	Degrees of freedom[b]	Sum of squares[c]	F-value[d]	Rapport t	p-value[e]
X1(5,7)	1	1109,59	35,03	5,92	0,0010[a]
X2(32,48)	1	665,06	20,99	4,58	0,0038[a]
X3(8,16)	1	94,08	2,97	1,72	0,1356
X1*X2	1	9,05	0,28	0,53	0,6121
X1*X3	1	384,61	12,14	3,48	0,0131[a]
X2*X3	1	26,39	0,83	0,91	0,3965
$X1^2$	1	838,75	26,48	-5,15	0,0021[a]
$X2^2$	1	464,26	14,66	-3,83	0,0087[a]
$X3^2$	1	84,90	2,68	-1,64	0,1527

[a] Significant at the 95% confidence level.

[b] Degrees of freedom: an estimate of the number of independent categories in a particular statistical test or experiment.

[c] Sum of squares: the sum of squares is a mathematical approach to determining the dispersion of data points. The sum of squares is used as a mathematical way to find the function which best fits (varies least) from the data.

[d] F-value: value calculated by the ratio of two sample variances. The F 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.

[e] p-Value: 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 actually observed.

C. BOD_5 removal

As COD results, The diagnostic plots of predicted versus observed values of BOD_5 removals (Fig. 2 (a)) illustrated that actual values were distributed near to the straight line, which indicated the model corresponded well with the measured values. Accordingly, this plot showed a sufficient agreement between observed values of BOD_5 removals and the values obtained from the model.

The BOD_5 permitted the development of mathematical equations where each response (Y) was estimated as a function of X_1, X_2 and X_3, and computed as the sum of a constant, three linear effects (terms in X_1, X_2 and X_3), three quadratic effects (X_1^2, X_2^2 and X_3^2), and three interactions effect (X_1X_2, X_1X_3, X_2X_3) according to Eq. (3). The result for response BOD_5 analyzed by analysis of variance (ANOVA) to estimate the goodness of fit has been summarized in Table 8.

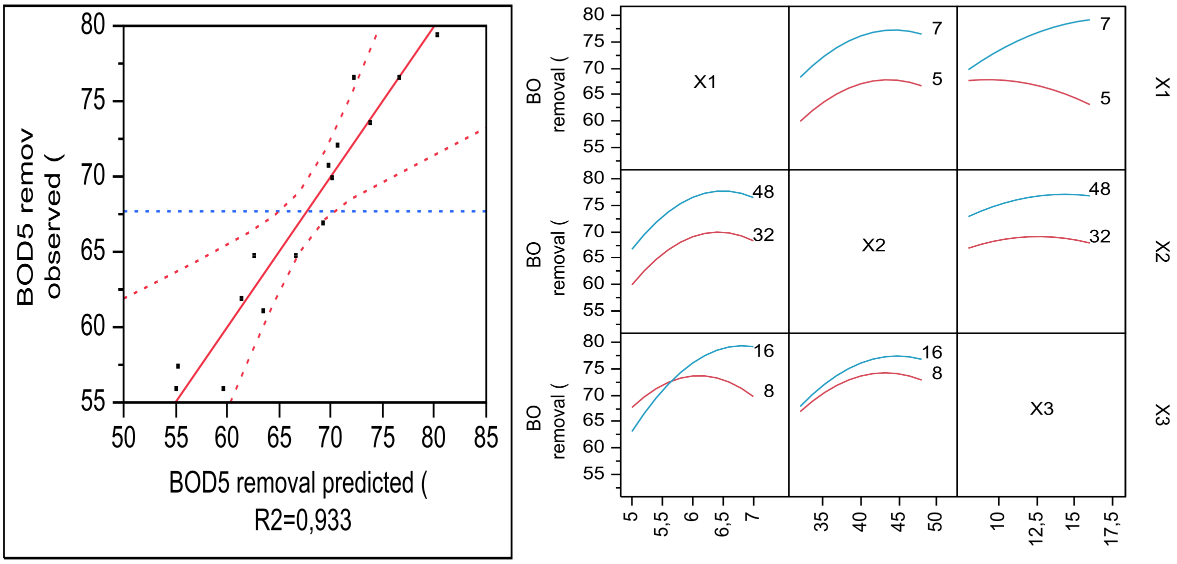
Table 8: ANOVA for BOD_5 removal response surface models.

Source	**Degrees of freedom[b]**	**Sum of squares[c]**	**F-value[d]**	**Rapport t**	**p-value[e]**
X1(5,7)	1	279,06621	27,3179	5,23	0,0020*
X2(32,48)	1	189,02336	18,5036	4,30	0,0051*
X3(8,16)	1	20,43218	2,0001	1,41	0,2070
X1*X2	1	1,06580	0,1043	0,32	0,7577
X1*X3	1	97,58045	9,5522	3,09	0,0214*

Source	**Degrees of freedom**[b]	**Sum of squares**[c]	**F-value**[d]	**Rapport t**	**p-value**[e]
X2*X3	1	4,32180	0,4231	0,65 	0,5395
$X1^2$	1	135,58152	13,2721	-4,68 	0,0108*
$X2^2$	1	104,51114	10,2306	-3,57 	0,0186*
$X3^2$	1	25,78362	2,5240	-1,59 	0,1632

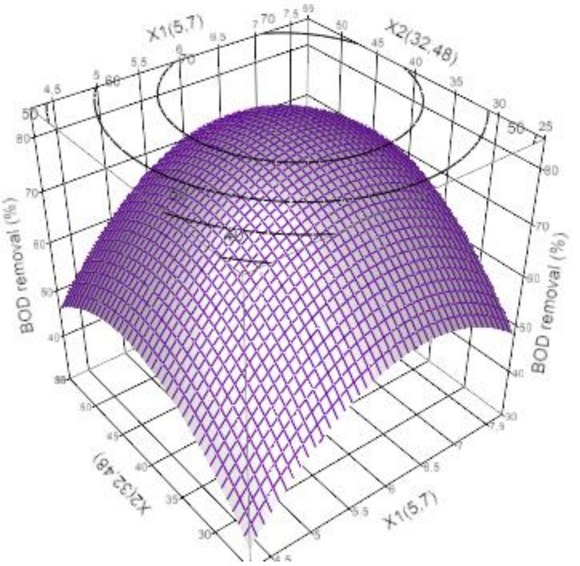
Data listed in this table indicated that X_1, X_2, X_1X_3, X_1^2 and X_2^2 resulting from ANOVA analysis in terms of coded variables were significant at 95% confidence level, with p-values of regression <0.05. As COD removal, the factors influences the elimination of BOD_5 are pH and dosage coagulant. At pH near to neutrality and dosage coagulant equal at 48 mL/L there is a high value of percentage removal of BOD_5 (77.5%). (Fig.2 (b)). As illustrated in Figs. 1 & 2 (c), the three-dimensional (3D) surface plot is approximately symmetrical in shape with circular contours. The response of BOD_5 removal plot show clear peak, which indicate that the optimum conditions for maximum value of the response are determined by dose coagulant and initial pH inside the design boundary. The two-dimensional representation of the response on the dose and initial pH upper surface present concentrically closed curves whose centers represent the optimum conditions. Figs. 2 (c) demonstrate that the optimum removal point (81.25%) obtained at around dose 45.7 mL/L and initial pH 7.07. The decline in removal efficiencies is observed when moving away from this point, implying that neither increase nor decrease in any of the tested variables is desired.

Fig. 2 (a): Experimental values versus predicted values for the BOD_5 removal. (b): Main interactions plot for BOD_5 removal. (c): Response surface plots for BOD_5 removal as a function of pH (X1) and coagulant dosage (X2) at flocculant dosage fixed at 12 mL/L.



(a)

(b)



(c)

D. Turbidity removal

The results central composite experimental design and response surface model in the form of analysis of variance for turbidity are shown in Table 9. The analysis shows that the model is highly significant as the p-value was lower than 0.05. Hence, there is a statistical relationship between turbidity removal and the selected variables at a 95% confidence level. As can be observed in Table 9, the significant terms in the model were X_1 of pH, X_2 of coagulant dosage, X_1X_2, X_1X_3 X_1^2 and X_2^2. Other model terms were not significant. Fig. 3(b)

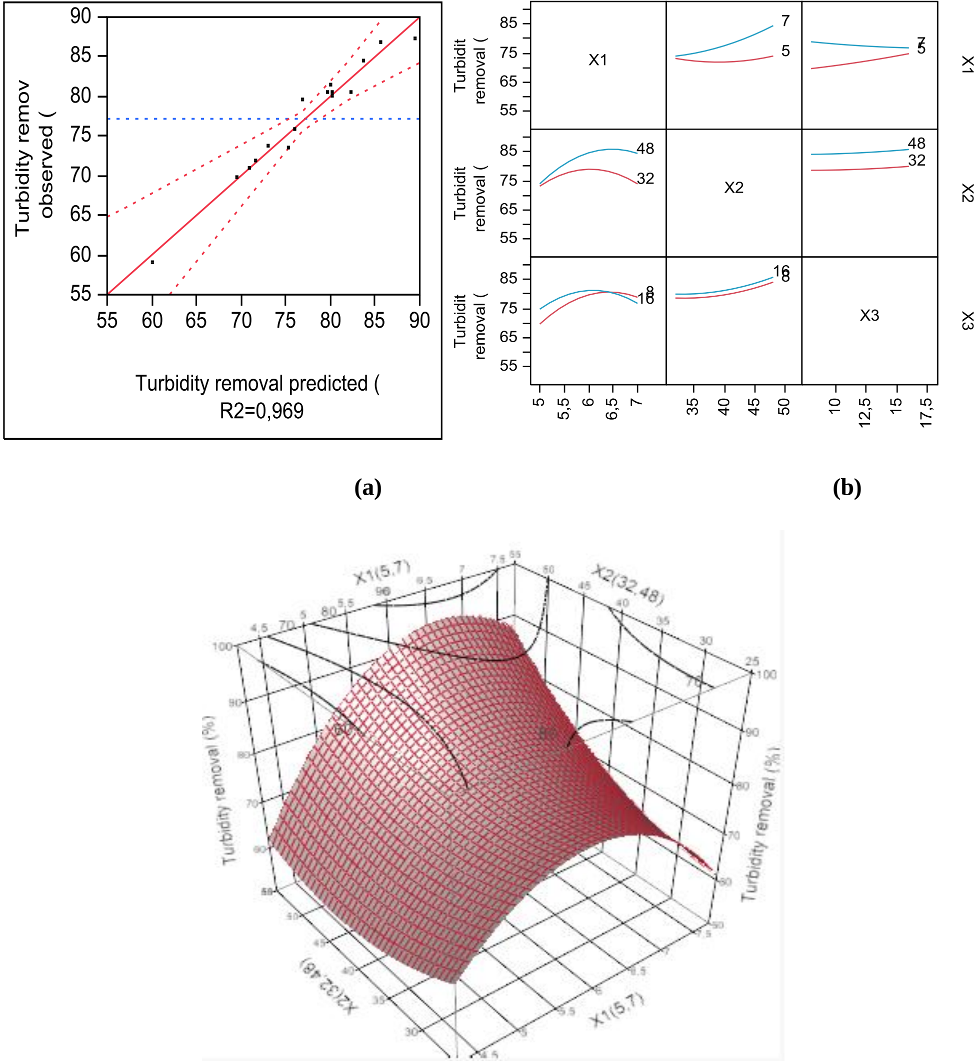
Table 9: ANOVA for turbidity removal response surface models

Source	Degrees of freedom[b]	Sum of squares[c]	F-value[d]	Rapport t	p-value[e]
X1(5,7)	1	107,29176	26,7815	5,18	0,0021*
X2(32,48)	1	108,60617	27,1096	5,21	0,0020*
X3(8,16)	1	7,84386	1,9579	1,40	0,2113
X1*X2	1	46,60951	11,6344	3,41	0,0143*
X1*X3	1	26,10031	6,5150	-2,55	0,0433*
X2*X3	1	0,05951	0,0149	0,12	0,9070
$X1^2$	1	444,55745	110,9678	-8,24	<,0001*
$X2^2$	1	26,18743	6,5367	2,52	0,0431*
$X3^2$	1	0,91071	0,2273	0,48	0,6504

The goodness of fit of the model for turbidity removal was evaluated by the regression coefficient R^2=0.97 The 96.96 % sample variation observed for turbidity removal was attributed to the variables selected (pH, coagulant and flocculant dosages), while the model did not explain 3.04 % of the variations.

Another way to assess the goodness of fit of the model is by plotting the experimental values versus the predicted values for turbidity removal. Fig. 3 (a) shows these plots. As can be seen, the model approximately total represents the experimental data over the range studied. The plot shows the best fit as it may also be observed by the regression coefficient. Fig. 6 shows 3D response surface plots for turbidity removal. The best results for turbidity removal were obtained at very high coagulant dosage levels and at pH near at neutrality as can be observed by the saddle shape at dosage flocculant fixed at 12 mL/L. To maximize turbidity removal, a single response optimization was carried out as explained in the section on COD removal.

Fig. 3 (a): Experimental values versus predicted values for the turbidity removal. (b): Main interactions plot for turbidity removal. (c): Response surface plots for turbidity removal as a function of pH (X1) and coagulant dosage (X2) at flocculant dosage fixed at 12 mL/L.



(a)

(b)

(c)

E. Model validation

To further validate the models under even higher reactant dosages, an additional experiment (7 of pH, 40 mL/L of coagulant, equivalent to 4052 mg Fe^{3+}/L, and 12.0 mL/L of flocculant) was performed. The responses are listed in Table 10 along with the predicted measured results.

Table 10: Validation of the models

	COD removal (%)			BOD_5 removal (%)			Turbidity removal (%)		
	X_1	X_2 (ml/l)	X_3 (ml/l)	X_1	X_2 (ml/l)	X_3 (ml/l)	X_1	X_2 (ml/l)	X_3 (ml/l)
Optimum X_i	7.33	47	20.9	7.07	45.7	18.5	6.15	32.4	9.5
Optimum response predicted (%)	64.9			81.2			78.7		
Optimum response experimental (%)	62.8			77.9			77.4		
Validation of the models at pH= 6, at 40 mL/L of coagulant and 12 mL/L of flocculant									
Optimum response predicted (%)	52.9			76.5			80.1		
Optimum response experimental (%)	50.9			73.2			78.8		

As can be seen in the table, the three responses were close to the responses that were estimated using response surface methodology. The experimental results were quite similar to the predicted results when the models to higher factor levels as can be observed. Therefore, it can be concluded that the models accurately represent COD, BOD5 and turbidity removals over the experimental range studied.

Table 10 shows the optimum values for the responses and the factors. The values were calculated by means of the desirability function and the models obtained using response surface methodology. The validation of this model show the optimum values of the factors and the responses were 6 of the pH, 40 mL/L of coagulant (equivalent to 4052 mg Fe3+/L), 12 mL/L of flocculant and 52.93, 76.51 and 80.11 % of COD, BOD5 and turbidity removal, respectively. The optimum dose of a coagulant or flocculant is defined as the value above which there is no significant difference in the increase in removal efficiency with a further addition of coagulant or flocculant.

IV. CONCLUSIONS

A 4^2 central composite experimental design and response surface methodology were used to optimize the coagulation–flocculation process of leachate resultant from compacting solid waste of city Mohammedia for reducing the number and cost of experiments and improving the process at industrial scale. The best regression coefficients (R^2) were obtained for COD, BOD_5 and turbidity at variable pH (5-7.7): 0.9452, 0.9333 and 0.9696, respectively. Coagulant dosage and pH seems to be the most significant factors in the soluble removal of COD, BOD_5 and turbidity. For flocculant dosage, however, the trend is not given that this factor influences in treatment of fresh leachate (for a low concentration of flocculant). Multiple response optimization allowed the coagulant and flocculant dosages to be minimized, while maximizing the COD, BOD_5 and turbidity removal percentages. The pH was also evaluated to determine the most suitable condition for the coagulation–flocculation process of operational, economic and post-treatment factors. The validation of this model show the optimum values of the factors and the responses were 6 of the pH, 40 mL/L of coagulant (equivalent to 4052 mg Fe^{3+}/L), 12 mL/L of flocculant and 52.93, 76.51 and 80.11 % of COD, BOD_5 and turbidity removal, respectively.

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