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Ceramic industry wastewater treatment by chemical coagulation process: a statistical optimization of operating parameters

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Abstract

This study deals with chemical oxygen demand (COD) removal from ceramic industry wastewater by chemical coagulation using alum and ferric chloride ($FeCl_3$) as coagulants. The study also focuses on the capillary suction time (CST) of sludge samples which is an important sludge dewatering parameter. Response surface methodology (RSM) approach was employed to evaluate the effects and interactions of the operating variables and to optimize the performance of the process. Significant quadratic polynomial models were obtained (R^2 = 96.26% for alum and R^2=89.15% for $FeCl_3$ for COD removal; R^2=96.6% for alum and R^2=90.9% for $FeCl_3$ for CST of sludge, respectively). Alum was found more effective coagulant for ceramic industry wastewater treatment as compared with $FeCl_3$. Numerical optimization based on desirability function was employed; in a 36 min trial 95.2% of COD removal was achieved at alum dosage of 3.3 g/L and pH 5. The optimization study shows that the minimum CST of sludge was found 17.4 s at alum dosage of 5 g/L and pH 5 in a reaction time of 16 min. The results indicate that the RSM is suitable for the design and optimization of chemical coagulation process using alum as a coagulant for the treatment of ceramic industry wastewater.

Keywords: Ceramic industry wastewater, chemical coagulation, response surface method, optimization

1. INTRODUCTION

Ceramics are produced using bricks, clay and sand as raw metarial in ceramic production industry [1]. Different kinds of soil and chemicals are used in ceramic industry processes [2]. Ceramic production steps such as preparation of clays slips and clay bodies for shaping, wet beneficiation or grinding processes require high amount of water. Depending on the applied techniques and the water pressure, approximately 5 L/t of product water is used [3]. Therefore this industry produces a significant amount of wastewater that contains organic and inorganic pollutants, even quantity of used water can be reduced when recycling on the process after settling process [1-3]. High amount of total suspended solids (TSS) and total dissolved solids (TDS) presence in wastewater because of high mineral ingredient of produced materials. Solids concentrations are generally between 1000 to 20000 mg/L for TSS and between 300 to 1000 mg/L for TDS, respectively [2, 4]. Ceramic industry wastewaters also contain organic compounds and have significant COD concentrations (between 500 to 1200 mg/L for a typical ceramic industry wastewater). On the other hand, wastewaters may contain heavy metals such as boron (B), lead (Pb), aluminium (Al), iron (Fe),

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copper (Cu), cadmium (Cd), manganese (Mn) and zinc (Zn) in low concentrations [2-4]. Boron is widely used to improve mechanical strength of ceramics in the ceramic and therefore, the main heavy metal is boron and its concentration in ceramic wastewater is up to 15 mg/L. A low concentration of Boron and other heavy metals in ceramic wastewater may have toxic effect on plant and human [1].

Ceramic industry wastewater is generally treated by chemical coagulation process to remove TSS [5-8]. Arslan et al. [5] investigated physicochemical treatability of tile industry wastewater. In the study, different coagulants ($Al_2(SO_4)_3$•$18H_2O$, $FeCl_3$•$6H_2O$ and Agrofloc 100) were used and TS and TSS removal efficiencies were investigated. They found high removal efficiencies for all coagulant matter [5]. On the other hand adsorption process is applied the removal of heavy metals in ceramic industry wastewater [9]. The boron and TSS removals were investigated by adsorption-flocculation mechanism using palm oil boiler bottom ash and polimer by Chong et al. [9]. Boron removal efficiency was found 80% under the optimum conditions using palm oil ash as a coagulant. On the other hand, the combined cationic and anionic polymers were used to treat the ceramic wastewater under the optimum conditions and the boron and TSS removal were found 80% and 99.75%, recpectively [9].

The present study investigated the treatment efficiency of the chemical coagulation process using $Al_2(SO_4)3$•$18H_2O$ and $FeCl_3$•$6H_2O$ as coagulants on ceramic industry wastewater. The process efficiency is evaluated through analyzing its effect on different operating parameters (pH, coagulant dosage and reaction time) in the aim to find the most adequate and favorable operating conditions. The operating parameters were chemical coagulation on COD and turbidity removal efficiencies and capillary suction time (CST) of sludge. The Response Surface Method (RSM) approach using Central Composite Design (CCD) was used to develop a mathematical model and to study the interactive effects of operating parameters.

2. MATERIALS AND METHODS

2.1. Ceramic industry wastewater characterization

Wastewater used in this study was taken from ceramic production for pool base cover in Istanbul. The characteristics of the wastewater were shown in Table 1. As seen from the Table 1, ceramic industry wastewater included high amount of TSS. According to the ceramic wastewater characteristics, at least 78% COD removal and 99% TSS removal efficiencies are ensured for the treated wastewater direct discharge to receiving water body according to Water Pollution Control Regulation of Turkey (COD <80 mg/L; TSS<100 mg/L) (Ministry of Environment and Urbanization, MoEU) [10].

Table 1. Characterization of ceramic industry wastewater

Parameter	Value
Chemical oxygen demand (COD; mg/L)	365 ± 10
Total suspended solids (TSS; g/L)	285 ± 5
Chloride (mg/L)	1200 ± 20
Electrical conductivity (µS/cm)	545 ± 15
pH	8,83 ± 0,15

2.2. Experimental set-up and procedure

$Al_2(SO_4)_3$•$18H_2O$ and $FeCl_3$•$6H_2O$ salts were used as a coagulant matter in chemical coagulation process and a stock solutions of these cogulants (200 g/L) were prepared for the coagulation process for. 500 mL ceramic industry wastewater was used for each experimental test. In the first step of coagulation process, the pH of ceramic industry wastewater was arranged to desired value using 6N of sulphuric acid or sodium hydroxide (pH: 5, 7, 9) and then necessary amount of coagulant was added (between 1 to 5 g/L) using a stock solutions of coagulants. Catioanic polyelectrolyte was added (1 g/L) for enhancing the coagulation process in all experimental sets. The optimum concentration of cationic polyelectrolte was determined by preliminary experiments. After that, rapid mixing was performed via jar test equipment at 200 rpm for 3 min. After the rapid mixing, wastewater sample was gently stirred at 30 rpm for a desired reaction time (15 to 45 min) in each experimental test and then the sample was settled for 3 h. Approximately 200 mL of the supernatant sample was taken for further analysis.

2.3. Design of experiments and data analysis

CCD was used for the RSM in order to design the set of experiments for coagulation prosess in this study. RSM is primarily a particular set of statistical and mathematical methods for experimental design, models buildings, determining the effect of variables, and investigating the optimum operating conditions [11-13]. Statgraphics Centurion XVI.I software programme was used for the statistical experimental design and analysis of data. The three most important operating variables, coagulant dosage (X_1), reaction time (X_2), and pH (X_3) in coagulation process were optimized. Each operating factor was coded at three levels between -1 and +1 for the ranges in the RSM approach. The levels and ranges of independent factors and experiment sets were shown in Table 2 and Table 3 for coagulation prosess, respectively. The responses of Y_1 and Y_2 are COD removal efficiency for alum and Fe_3Cl, Y_3 and Y_4 are CST of sludge for alum and Fe_3Cl, respectively in Table 3.

RSM makes it possible to represent operating parameters in quantitative form (Eq. (1)):

$$Y = f(X_1, X_2, X_3, \dots, X_n) \pm \varepsilon \quad (1)$$

Where Y is the dependent (response) parameter, f is the response function, ε is the experimental error and X_1, X_2, X_3,…., X_n are independent (operating) parameters. In the optimization process, the dependent parameters can be related to independent factors by linear or quadratic models. A quadratic model that includes the linear model is given in Eq. (2).

Table 2. Experimental range and levels of the operating variables for CC process

Factor	-1	0	+1
X_1: $FeCl_3 \cdot 6H_2O$ or $Al_2(SO_4)_3 \cdot 18H_2O$ dosage (g/L)	1	3	5
X_2: Reaction time (min)	15	30	45
X_3: pH	5	7	9

$$Y = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_{11}{X_1}^2 + \beta_{22}{X_2}^2 + \beta_{33}{X_3}^2 + \beta_{12}X_1X_2 + \beta_{13}X_1X_3 + \beta_{23}X_2X_3 \quad (2)$$

Where β is regression coefficients: the intercept (β_0), linear (β_1, β_2, β_3), interaction (β_{12}, β_{13}, β_{23}) and quadratic coefficients (β_{11}, β_{22}, β_{33}).

Analysis of variance (ANOVA) was used to obtain the interaction between the independent and dependent variables. R^2 was used to evaluate the quality of the fit polynomial model. Model statistical significance was also checked by the Fisher F-test by ANOVA. The model terms were utilized by the P value (probability) with 95% confidence level.

3. Table 3. Experimental conditions and experimental values of the responses in coagulation process

Run	X_1	X_2	X_3	Y_1*	Y_2*	Y_3*	Y_4*
1	-1	-1	0	50,5	87,7	57,2	95,4
2	1	-1	0	96,5	92,1	28,9	73,1
3	-1	1	0	64,9	94,7	46,4	102,5
4	1	1	0	81,0	89,5	23,9	56,6
5	-1	0	-1	80,7	54,4	41,7	88,2
6	1	0	-1	91,2	64,1	17,9	55,9
7	-1	0	1	56,2	74,6	60,8	83
8	1	0	1	66,8	10,6	28,5	65,1
9	0	-1	-1	90,4	22,0	30,4	58,9
10	0	1	-1	99,5	50,0	33,5	79,6
11	0	-1	1	90,5	79,8	40,9	63
12	0	1	1	86,8	30,7	35,4	73,2
13	0	0	0	94,6	45,1	47,8	69,1
14	0	0	0	94,4	46,4	48,8	68
15	0	0	0	94,2	44,1	52,7	67,9

* Y_1: COD removal efficiency for alum

*Y_2: COD removal efficiency for Fe_3Cl

*Y_3: CST of sludge for alum

*Y_4 : CST of sludge for Fe_3Cl

4. RESULTS AND DISCUSSION

In this study, the relationship between the three variables (coagulant dosage, reaction time, and, initial pH) and the two important process responses (COD and CST of sludge) for the coagulation process was analyzed using RSM. COD removal efficiency for alum (Y_1: %), COD removal for alum for Fe_3Cl (Y_2: %), CST for alum (Y_3: s), and CST for Fe_3Cl (Y_4: s) were considered to be response variables (Table 3). TSS of supernatant was not optimized as all samples TSS values were found over 99% for alum and $FeCl_3$.

4.1. Optimization of COD Removal

Based on the experimental design results, the regression equations with the coded variables obtained for describing the COD removal efficiencies from ceramic industry wastewater by coagulation process using alum and Fe_3Cl are presented in the following Equations (3-4). In this equations, the coefficients with one factor (X_1, X_2, or X_3) represent the effects of the linear main factor, the coefficients with two factors (X_1X_2, X_1X_3, X_2X_3) and second order terms (X_1^2, X_2^2 or X_3^2) represent the interaction between the two factors and the quadratic effects. The positive sign in front of the coefficients indicates a synergistic effect, whereas the negative sign indicates an antagonistic effect [14,15]. On the basis of the coefficients in Eq. (3) it can be seen that COD removal efficiencies increase with an increase of coagulant dosage, reaction time and initial pH when alum is used as a coagulant. On the other side, only the pH of wastewater has positive effect on COD removal efficiency when $FeCl_3$ is used as a coagulant.

$$Y_1, \% = 7,49058 + 42,9632*X_1 + 1,8446*X_2 + 0,892694*X_3 - 4,98359*X_1^2 - 0,274429*X_1*X_2 - 0,0547945*X_1*X_3 - 0,00885591*X_2^2 - 0,0606164*X_2*X_3 - 0,248145*X_3^2 \quad (3)$$

$$Y_2, \% = -286,031 - 7,11301*X_1 - 0,801826*X_2 + 102,873*X_3 + 6,38185*X_1^2 - 0,0803653*X_1*X_2 - 4,60274*X_1*X_3 + 0,0900761*X_2^2 - 0,642922*X_2*X_3 - 4,96062*X_3^2 \quad (4)$$

The significance of regression parameters was evaluated using ANOVA and the ANOVA results of quadratic models are shown in Table 4. The results show a high reliability in the estimation of COD removal efficiencies for both coagulants (R^2 values of 96.26 and 89.15 for alum and Fe_3Cl). It can be said that only 3.74% (alum) and 11% ($FeCl_3$) of the variability in the responses were not explained by these models in coagulation process. As seen from Table 4, the model F-values of 14.32 and 4.56 implied significant models for COD removal efficiencies for alum and $FeCl_3$, respectively. It is note that the large F-value shows a high significance of the corresponding term. It is also note that the model terms are significant when Prob. > F values less than 0.05. Whereas the values greater than 0.1 indicate that the model terms are not significant [16, 17]. The Model Prob.>F values of COD removal efficiency is less than 0.05 for only alum and it is said that the prediction of the model is significant.

Table 4. ANOVA results of the predicted response surface quadratic model of COD removal

Coagulant	R^2	Adj.R^2	SS	MS	F-value	Prob.>F
Alum	96.26	89.54	3179.20	340.05	14.32	0.0046
$FeCl_3$	89.15	69.62	10018.45	992.39	4.56	0.0546

Table 5. ANOVA results for the response surface quadratic model of COD removal for alum

Factor	SS	Df	MS	F-value	Prob.>F
X_1	632.35	1	632.31	26.63	0.0036
X_2	7.75	1	7.75	0.33	0.5924
X_3	666.62	1	666.62	28.08	0.0032
X_1^2	1467.24	1	1467.24	61.80	0.0005
X_1X_2	271.12	1	271.12	11.42	0.0197
X_1X_3	0.19	1	0.19	0.01	0.9318
X_22	14.66	1	14.66	0.62	0.4676
X_2X_3	13.23	1	13.23	0.56	0.4890
X_3^2	3.64	1	3.64	0.15	0.7116
Total error	118.71	5	23.74		
Total (corr.)	3179.20	14			

*SS: sum of squares, Df: degree of freedom, MS: mean square

The ANOVA tables obtained from the response surface quadratic models of COD removal efficiency for alum are shown in Table 5. The ANOVA results showed that coagulant dosage (X_1), pH (X_3), the quadratic terms of coagulant dosage (X_1^2), and the interaction between coagulant dosage and reaction time (X_1X_2) have significant effects on COD removal efficiency ($P<0.05$).

Figure 1 shows the effects of responses variables on COD removal efficiency. As can be seen from Figure 1a, a slight increase in COD removal was observed with an increase in the coagulant dosage. A similar interaction effect was shown between coagulant dosage and pH (Figure 1b). It can be also said that COD removal efficiencies increased with the increasing reaction time (Figure 1c).

Optimized conditions for COD removal are presented in Table 6. The optimum conditions were 3.3 for coagulant dosage, 36 min for reaction time, and 5 for pH. Under these conditions, optimum COD removal efficiency of model prediction was 100%. The additional coagulation process was conducted at these optimum conditions in order to control the optimization and it was obtained 95.2% of COD removal efficiency from the laboratory experiment. This result indicates that the prediction for the COD removal model was in the confidence interval.

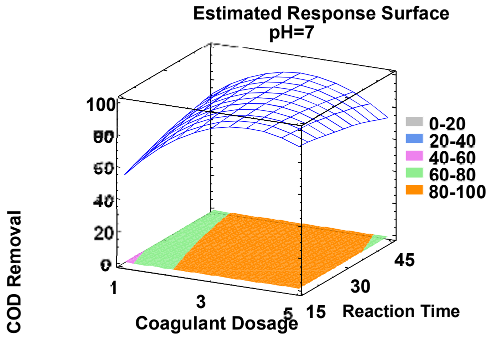
Table 6. Optimum operating conditions and comparison of model prediction result with experimental result for COD (alum as a coagulant)

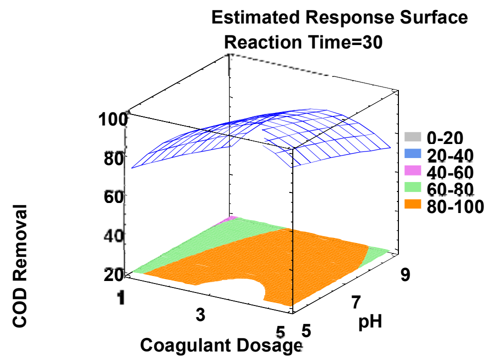
Dosage (g/L)	Time (min)	pH	Model prediction result (%)	Experimental results (%)
3.3	36	5	100	95.2

4.1. Optimization of Capillary Suction Time of Sludge

Sludge dewataring generally remains the most difficult and elusive of Environmental Engineering challenges and there are not easy and reliable methods to evaluate releasing water from the sludge. The two commonly used and well-known methos are spesific resistance to filtration (SRF) and capillary suction time (CST). SRF is inconvenient and time-consuming method, whereas CST is a quick and easy method for determining sludge dewaterability. The CST is measured by recording the time in seconds (s) for the water draining from the sludge poured into the central funnel to travel a set distance. The CST test is a precise measure of the rate at which water is released from a sludge matrix and can be used as a substitute for SRF to assess sludge dewaterability [13, 18, 19].

(a)





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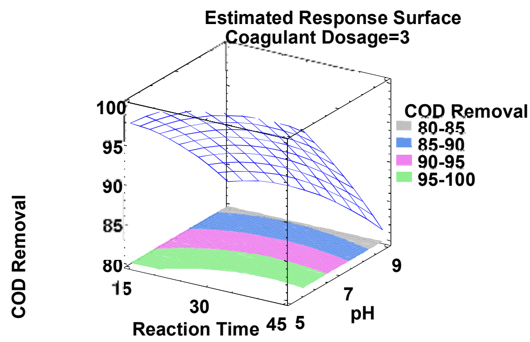


(b)

Figure 1. Response surface graphs of COD removal for alum (a) coagulant dosage vs. reaction time. (b) coagulant dosage ratio vs. pH. (c) reaction time vs. pH

In this study, CST values of sludge were measured after the coagulation process for both coagulants. The regression equations with the coded variables obtained for describing the CST values of sludge from ceramic industry wastewater by coagulation using alum and $FeCl_3$ are presented in Equations (5-6). On the basis of the coefficients in Eq. (5-6) it can be seen that CST values for both alum and $FeCl_3$ increase with increase in reaction time and pH. Whereas, CST values increase with increase in alum dosage but they decrease with increase FeCl3 dosage in coagulation process.

$$\text{CST, s (Alum)} = -102.775 + 1.95625*X_1 + 1.91722*X_2 + 35.4104*X_3 - 1.06146*X_1^2 + 0.0483333*X_1*X_2 - 0.53125*X_1*X_3 - 0.028537*X_2^2 - 0.0716667*X_2*X_3 - 2.07396*X_3^2 \quad (5)$$

$$\text{CST, s } (FeCl_3) = 53.6937 - 21.2563*X_1 + 0.156111*X_2 + 14.9208*X_3 + 2.24271*X_1^2 - 0.196667*X_1*X_2 + 0.9*X_1*X_3 + 0.0204259*X_2^2 - 0.0875*X_2*X_3 - 1.06354*X_3^2 \quad (6)$$

The ANOVA analysis indicated that all independent variables and their interactions had significant effect on the models. The quadratic model statistical results for CST of sludge are summarized in Table 7. The results show a high reliability in the estimation of CST of sludge for boht coagulant (R^2 values of 96.6% and 90.9%). It can be said that only 3.4% and 9.9% of the variability in the responses for CST values were not explained by the models in coagulation process with alum and $FeCl_3$. The model F-values implies that the models are significant for CST. The associated Prob>F value is used to estimate whether statistical significance is large enough [20, 21]. The values of Prob. > F less than 0.05 indicates that the terms are significant in all the models (All response's Prob. > F values less than 0.05).

The ANOVA tables obtained from the response surface quadratic models for CST are shown in Tables 8. The ANOVA results showed that the alum dosage (X_1), pH (X_3), the quadratic terms of reaction time (X_2^2) and pH (X_3^2) have significant effects on CST of sludge in coagulation process. On the other hand, only $FeCl_3$ dosage has significant effect on CST values of sludge.

The effects of variables on CST of sludge can be seen in Figure 2 and 3 for alum and $FeCl_3$, respectively. As can be seen in Figure 2 and 3, all the plots have clear peaks at the optimum operating conditions indicating that the maximum values of the responses are attributed to all the variables in the design space.

The effects of operational parameters for the minimization of CST values of sludge are shown in Table 9. Under the optimum conditions, the minimum CST values of sludge of model predictions for alum and $FeCl_3$ were 17 s and 53 s. According to the experimental results conducted at optimum conditions, the CST values were found as 17.4 s and 56.2 s. The optimum CST were determined as 97.7% and 94.3% by model prediction and laboratory experiment, respectively. It is quite clear that the experimental values for CST of sludge were found to be consistent with the predicted one.

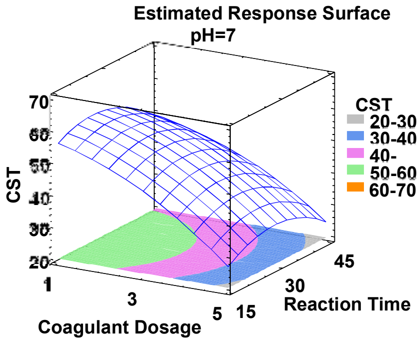
Table 7. ANOVA results of the predicted response surface quadratic model of CST

Coagulant	R^2	Adj.R^2	SS	MS	F-value	Prob> F
Alum	96.6	90.4	2229.56	15.35	15.58	0.0037
$FeCl_3$	90.9	74.5	2734.12	49.87	5.53	0.0370

Table 8. ANOVA results for the response surface quadratic model of CST for alum and $FeCl_3$ (*SS: sum of squares, Df: degree of freedom, MS: mean square)

	Factor	SS	Df	MS	F-value	Prob.>F
Alum	X_1	1428.45	1	1428.45	93.03	0.0002
	X_2	41.40	1	41.40	2.70	0.1615
	X_3	221.55	1	221.55	14.43	0.0126
	X_1^2	66.56	1	66.56	4.33	0.0918
	X_1X_2	8.41	1	8.41	0.55	0.4925
	X_1X_3	18.06	1	18.06	1.18	0.3276
	X_2^2	152.22	1	152.22	9.91	0.0254
	X_2X_3	18.49	1	18.49	1.20	0.3225
	X_3^2	254.11	1	254.11	16.55	0.0097
	Total error	76.77	5	15.35		
	Total (corr.)	2229.56	14			
$FeCl_3$	X_1	1752.32	1	1752.32	35.14	0.0019
	X_2	57.78	1	57.78	1.16	0.3309
	X_3	0.36	1	0.36	0.01	0.9355
	X_1^2	297.142	1	297.14	5.96	0.0586
	$X1X_2$	139.24	1	139.24	2.79	0.1556
	X_1X_3	51.84	1	51.84	1.04	0.3547
	X_2^2	77.99	1	77.99	1.56	0.2664
	X_2X_3	27.56	1	27.56	0.55	0.4907
	X_3^2	66.82	1	66.82	1.34	0.2993
	Total error	249.36	5	49.87		
	Total (corr.)	2734.12	14			

(a)



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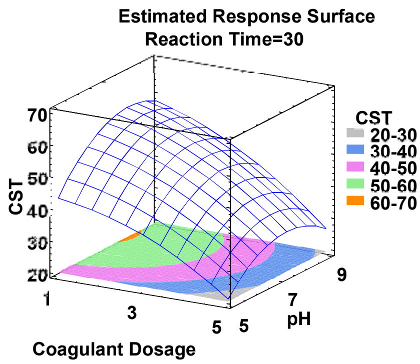

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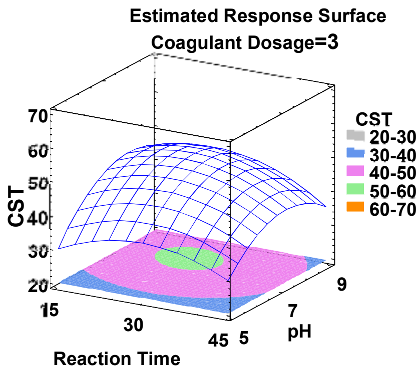
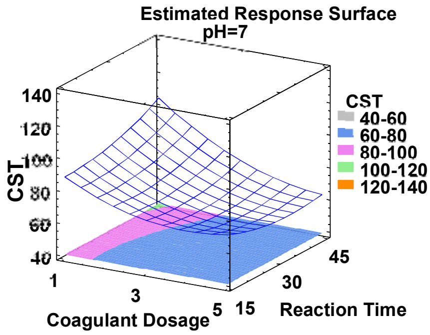


Figure 2. Response surface graphs of CST for alum (a) coagulant dosage vs. reaction time. (b) coagulant dosage ratio vs. pH. (c) reaction time vs. pH



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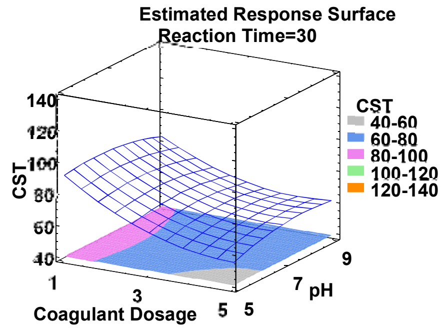

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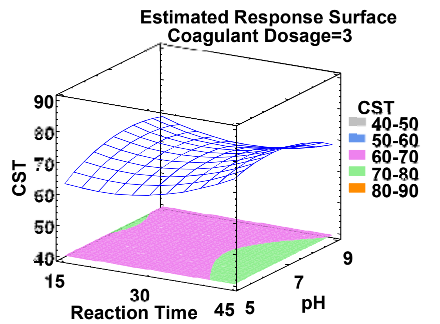

Figure 3. Response surface graphs of CST for $FeCl_3$ (a) coagulant dosage vs. reaction time. (b) coagulant dosage ratio vs. pH. (c) reaction time vs. pH

(a)

Table 9. Optimum operating conditions and comparison of model prediction result with experimental result for CST values of sludge

	Dosage (g/L)	Time (min)	pH	Model prediction result (s)	Experimental results (%)
Alum	5	16	5	17	17.4
$FeCl_3$	5	28	5	53	56.2

5. CONCLUSION

In this study, the chemical coagulation process was used for the ceramic industry wastewater treatment. Alum was found an effective parameter for the removal of COD and mimimum value of sludge CST. The treatment efficiency was found to be a function of the alum dosage, reaction time and initial. Within the coagulation process, the COD removal efficiencies, and CST of waste sludge were determined. The RSM approach was also applied to find the optimum operating conditions for these responses. Although TSS of ceramic industry wastewater was significantly high, TSS removal was not optimized because of over the 99% TSS removal efficiencies for all experimental studies. According to the ANOVA results, the RSM could be used to navigate the design space with high regression coefficient value above 90.9% for all the responses. The COD removal efficiency at optimum operating parameters were found to be 95.2% for alum. The effluent COD cocentration was determined as 17.5 mg/L. The results obtained indicated that the discharge limit standards for ceramic industry wastewater given in the Water Pollution Control Regulation of Turkey could easily be met (Water Pollution and Control Regulation, 2004). On the other hand CST of waste sludge was found 17.4 s when the alum was used as a coagulant matter at optimum operating conditions and this result indicate that the waste sludge is easily dewatered by sludge dewatering processes such as filter press and belt filter.

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