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Application of response surface methodology for optimization of chemical coagulation process to treat rice mill wastewater

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ABSTRACT

In this present study, chemical coagulation method (CC) using ferric chloride was used to treat the rice mill wastewater and the effects of initial pH, ferric chloride dose and agitation speed on the removal of COD, turbidity and total suspended solids were investigated using Box-Behnken response surface design. The experimental results show that CC could be effectively reduced the COD (91%), turbidity (87%) and TSS (83 %) at the optimum conditions of pH 7, ferric chloride dose of 2.5 g/l, and agitation speed of 30 rpm respectively. The experimental results were in reasonable agreement with the predicted values. These results show that, effectiviness of CC as an effective pre-treatment in wastewater treatment.

KEYWORDS

Rice mill wastewater;
Chemical coagulation;
Ferric chloride;
Box-Behnken design.

INTRODUCTION

Rice mill industry wastewater is recognized as one of the toxic wastewater, which contains huge amount of organic and inorganic matters with the yield of 1.0–1.2 l/kg of rice[1]. Dischrage of this wastewater into the ecological system without pretreatment causes the harmful effects on receiving natural environmental sources such as water bodies, land and human being. Therefore, there is a critical need to find out the proper treatment technique to treat rice mill industry wastewater interms of removal efficiency of toxic organic matters as well as economic feasibility to implement in large scale application[2]. From the literature survey it was found that very limited studies on the treatment of rice processing industry wastewatere were reported. Rajesh et al.[1] studied the treatment of rice mill industry wastewater using two stage upflow anaerobic sludge blanket (UASB) bioreactor and found that the percentage of BOD and COD removal were 89 and 78 respectively under the optimum conditions. Manogari et al.[2] investigated the treatment of rice mill industry wastewater using pseudomonas sp. and found that the percentage of COD and BOD removal were 86.44 and 55.34 respectively after 24 hours by immobilized packed bed system. Manaswini Behera et al.[3] have demonstrated the performance of microbial fuel cells (MFC) made of earthen pot and a proton exchange membrane for treatment of rice mill wastewater and found that higher COD removal efficiency (>75 %). But, these treatment techniques were not sufficient to treat rice proceesing industry wastewater effectively due its complicated treatment setup, longer retention time and limited amount of removal efficiency of toxic pollutants[3].

Various advanced wastewater treatment techniques such as chemical coagulation, electrocoagulation, fenton, adsorption, fluidized bed reactor, electrochemical oxidation, electro-fenton, membrane filtration, photo-fenton, anaerobic process and ozonation treatment process have been proposed to treat various industrial wastewaters[4-13]. Among these technologies, chemical coagulation has many advantages such as cost-effective, high removal efficiency, no need of additional separation method, simple process, small treatment time and easy to handle, over other treatment techniques[14]. Moreover, this process can be directly applied to wastewaters to remove organic matters together with suspended solids, without being affected by the toxicity in the wastewater. This method consists in destabilizing colloids, aggregating and binding them together into flocculates; the resulting flocs can finally be removed either by sedimentation or by precipitation[15]. Destabilization involves first an increase of ionic strength which promotes double-layer compression, and the neutralization of the particle surface charge by adsorbing counter-anions, using the addition of chemicals called coagulants. Mean while, many factors can influence its efficiency, such as the coagulant type and coagulant dosage, pH, mixing, temperature and retention time[16]. Optimization of these factors significantly increase the process efficiency. In conventional multi-factor experiments, optimization is usually carried out by varying single factor while keeping all other factors constant at a specific set of environment. It is not only time-consuming, but also usually incompetent of realization the true optimum due to ignoring the relations among variables[17]. On the other hand, the response surface methodology (RSM) has been projected to determine the influences of individual factors and their interactive effects. The RSM is a statistical technique for designing experiments, building models, evaluating the effects of several factors, and searching optimum conditions for desirable responses[18]. With RSM, the interactions of possible influencing parameters on treatment efficiency can be evaluated with a limited number of planned experiments. This method has been widely used for optimization of various wastewater treatment techniques such as electrocoagulation, adsorption, electro-Fenton and electro-oxidation[19].

However, to our best knowledge, no publications are available on the treatment of rice mill industry wastewater using chemical coagulation via response surface methology. Hence the objective of the present study has been made to investigate and optimize the individual and the interactive effect of process variables such as initial pH, ferric chloride dose and agitation speed on the maximum removal efficiency of chemical oxygen demand (COD), turbidity and total suspended solids (TSS) from rice mill industry wastewater using Box-Behnken response surface design coupled with Derringer's desired function methodology.

MATERIALS AND METHODS

Materials

Freshly collectd rice mill industry wastewater was used as raw material, which was collected from the local industry near Erode, TamilNadu, and were stored at 4 °C prior to the experiments. The characteristics of rice processing industry wastewater are shown in TABLE 1. Ferric chloride ($Fecl_3.6H_2O$) was purchased from Sigma chemicals, Mumbai. All the chemicals used in this study were analytical grade.

TABLE 1 : Characteristics of rice processing industry wastewater

Characteristics	Value
pH	5.1
COD (mg/l)	2154
BOD (mg/l)	986
Total suspended solids (mg/l)	744
Turbidity (NTU)	574
Conductivity (mS/cm)	0.09

Chemical coagulation

A conventional batch type chemical coagulation studies were carried out with varying coagulant dose in 100 ml of composite wastewater for different pH range (5 - 9). The samples were agitated for 3 min by varying agitation speed (20-40 rpm) using incubator shaker equipment and then samples were allowed to settle for one hour. Then supernatant portion of wastewater was used for determination of COD, turbidity and TSS. All the experiments were performed in three replicates to check the reproducibility.

Analytical methods

American public health association (APHA) standard methods were used to determine the wastewater characteristics such as initial pH, COD, turbidity, BOD and TSS. The removal efficiency was calculated using the following equation[20]

$$R = \frac{Y_0 - Y}{Y_0} \times 100 \qquad (1)$$

Where, R is removal efficiency (%), Y_0 and Y were initial and final values of COD, turbidity and TSS.

Experimental design

In this study, Box-Behnken response surface experimental design (BBD) with three factors at three levels was used to optimize and investigate the influence of process variables such as initial pH (5-9), ferric chloride dose (1 - 3 g/l) and agitation speed (20 – 40 rpm) on the treatment of rice mill industry wastewater. Process variables and their ranges were determined based on the single factor experimental analysis. After selection of process (independent) variables and their ranges, experiments were established based on a BBD and the complete design consists of 17 experiments with five centre points (used to estimate the experimental error). The total number of experiments was calculated from the following equation[21]

TABLE 2 : Ranges of independent variables and their levels

Variable (unit)	Symbol	Level		
		-1	0	1
Initial pH	X_1	5	7	9
Ferric chloride dose (g/L)	X_2	1	2	3
Agitation speed (Rpm)	X_3	20	30	40

$$N = 2K(K-1) + C_0 \qquad (2)$$

where, K is number of factors and C_0 is the number of central point. For predicting the optimal point after performing experiments, a second-order polynomial equation was fitted to correlate the relationship between independent variables and responses, which accounts for variations caused by linear, quadratic and interactive effect of the process variables. The mathematical form of second-order polynomial equation is given below[22]

$$Y = \beta_0 + \sum_{j=1}^{k}\beta_j X_j + \sum_{j=1}^{k}\beta_{jj}X_j^2$$

$$+ \sum_{i}\sum_{<j=2}^{k}\beta_{ij}X_iX_j + e_i \qquad (3)$$

where, Y is the response; X_i and X_j are variables (i and j range from 1 to k); β_0 is the model intercept coefficient; β_j, β_{jj} and β_{ij} are interaction coefficients of linear, quadratic and the second-order terms, respectively; k is the number of independent parameters (k = 3 in this

TABLE 3 : BBD and their experimental results

S.No	Initial pH (X_1)	Ferric Chloride dose (X_2)	Agitation speed (X_3)	COD Removal (Y_1)	Turbidity removal (Y_2)	TSS removal (Y_3)
1	5	1	30	65.54	61.28	55.48
2	9	1	30	45.84	40.57	35.48
3	7	3	40	74.85	70.56	65.86
4	5	2	20	57.84	53.84	48.58
5	9	3	30	78.24	73.98	68.45
6	7	2	30	90.04	86.22	81.48
7	7	1	20	50.24	45.94	40.58
8	7	2	30	90.04	86.22	81.48
9	9	2	20	55.38	51.76	45.86
10	7	3	20	76.98	73.46	68.54
11	7	1	40	69.48	64.87	61.24
12	9	2	40	59.54	55.88	50.42
13	5	3	30	74.28	72.04	66.52
14	7	2	30	90.04	86.22	81.48
15	7	2	30	90.04	86.22	81.48
16	5	2	40	71.54	65.48	56.94
17	7	2	30	90.04	86.22	81.48

TABLE 4 : Multi regression analysis of responses

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	Remarks
Sequential model sum of squares for COD removal						
Mean	88987	1	88987			
Linear	938	3	313	2	0.2463	
2FI	277	3	92	0	0.7582	
Quadratic	2315	3	772	516	< 0.0001	Suggested
Cubic	10	3	3	6366	< 0.0001	Aliased
Residual	0	4	0			
Total	92527	17	5443			
Sequential model sum of squares for turbidity removal						
Mean	79257	1	79257			
Linear	991	3	330	2	0.2353	
2FI	262	3	87	0	0.7817	
Quadratic	2385	3	795	261	< 0.0001	Suggested
Cubic	21	3	7	6366	< 0.0001	Aliased
Residual	0	4	0			
Total	82915	17	4877			
Sequential model sum of squares for TSS removal						
Mean	67517	1	67517			
Linear	946	3	315	1	0.2781	
2FI	260	3	87	0	0.8015	
Quadratic	2570	3	857	205	< 0.0001	Suggested
Cubic	29	3	10	6366	< 0.0001	Aliased
Residual	0	4	0			
Total	71322	17	4195			

Source	Std.Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	Remarks
Model summary statistics for COD removal						
Linear	14.150	0.265	0.095	-0.1127	3939	
2FI	15.251	0.343	-0.051	-0.6036	5677	
Quadratic	1.223	0.997	0.993	0.9527	167	Suggested
Cubic	0.000	1.000	1.000		+	Aliased
Model summary statistics for turbidity removal						
Linear	14.325	0.271	0.103	-0.1022	4032	
2FI	15.512	0.342	-0.052	-0.6159	5911	
Quadratic	1.745	0.994	0.987	0.9068	340	Suggested
Cubic	0.000	1.000	1.000		+	Aliased
Model summary statistics for TSS removal						
Linear	14.830	0.249	0.075	-0.1411	4341	
2FI	16.121	0.317	-0.093	-0.7144	6522	
Quadratic	2.045	0.992	0.982	0.8769	468	Suggested
Cubic	0.000	1.000	1.000		+	Aliased

study); and e_{-i} is the error. The detail methodology used in this study was reported in elsewhere[23,24]. All the statistical analyses were done with the help of Stat ease Design Expert 8.0.7.1 statistical software package (Stat-Ease Inc., Minneapolis, USA). After fitting the data to the models, the models were used for the construction of response surface contour plots to forecast the relationships between independent and dependent variables. After analyzing the polynomial equation depicting the effect of independent variables on the responses, optimization process was carried out by Derringer's desired function methodology in order to find out the ef-

TABLE 5 : ANOVA table for responses

Source	COD removal (%)		Turbidity removal (%)		TSS removal (%)	
	RC	*P* value	RC	*P* value	RC	*P* value
Model	90.04	< 0.0001	86.22	< 0.0001	81.48	< 0.0001
X_1	-3.78	< 0.0001	-3.81	0.0005	-3.41	0.0022
X_2	9.16	< 0.0001	9.67	< 0.0001	9.57	< 0.0001
X_3	4.37	< 0.0001	3.97	0.0004	3.86	0.0011
X_1X_2	5.92	< 0.0001	5.66	0.0003	5.48	0.0011
X_1X_3	-2.39	0.0059	-1.88	0.0681	-0.95	0.3838
X_2X_3	-5.34	< 0.0001	-5.46	0.0004	-5.84	0.0007
X_1^2	-15.44	< 0.0001	-15.61	< 0.0001	-16.80	< 0.0001
X_2^2	-8.63	< 0.0001	-8.64	< 0.0001	-8.20	< 0.0001
X_3^2	-13.53	< 0.0001	-13.87	< 0.0001	-14.23	< 0.0001
C.V. %	1.69		1.74		3.25	
AP	45.75		32.42		27.71	
R^2	0.9970		0.9942		0.9923	
Adj-R^2	0.9932		0.9867		0.9824	
Pre-R^2	0.9527		0.9068		0.8769	

fective operating conditions of chemical coagulation method.

RESULTS AND DISCUSSIONS

In this present study, three factors with three levels BBD was used to evaluate and optimize the chemical coagulation process variables on the responses such COD removal, turbidity removal and TSS removal. Chemical coagulation process variables and their ranges are shown in TABLE 2. A total number of 17 batch experiments including five centre points were carried out in triplicates using statistically deigned experiments and the results (means values) are shown in TABLE 3.

Experimental design analysis

The experimental data was analyzed by two different tests namely the sequential model sum of squares[25] and model summary statistics in order to obtain regression models and decide about the adequacy of various models (linear, interactive, quadratic and cubic) to represent the chemical coagulation treatment process effectively. The results are listed in TABLE 4. From the TABLE 4, it is found that, quadratic models are exhibited higher R^2, adjusted R^2, predicted R^2 and also having low *p*-values, when compared with other models. Therefore the quadratic model is chosen[26] to describe the effects of process variables on the treatment of rice processing industry wastewater using ferric chloride.

Mathematical model development

The results obtained from BBD experiments were evaluated by multiple regression analysis method and empirical relationship between the response and independent variables has been expressed by a second-order polynomial equation with interaction terms. Three empirical models were developed to understand the interactive correlation between the responses and process variables. The final model obtained in terms of coded factors is given below

$$\begin{aligned} \mathbf{Y_1} &= \mathbf{90.04 - 3.77X_1 + 9.16X_2 + 4.37X_3} \\ &\mathbf{+ 5.91X_1X_2 - 2.39X_2X_3 - 5.34X_2X_3 - 15.44X_1^2} \\ &\mathbf{- 8.63X_2^2 - 13.53X_3^2} \end{aligned} \qquad (5)$$

$$\begin{aligned} \mathbf{Y_2} &= \mathbf{86.22 - 3.81X_1 + 9.67X_2 + 3.97X_3} \\ &\mathbf{+5.66X_1X_2 - 1.88X_2X_3 - 5.46X_2X_3 -} \\ &\mathbf{15.61X_1^2 - 8.64X_2^2 - 13.874X_3^2} \end{aligned} \qquad (6)$$

$$\begin{aligned} \mathbf{Y_3} &= \mathbf{81.48 - 3.41X_1 + 9.57X_2 + 3.86X_3} \\ &\mathbf{-5.48X_1X_2 - 0.95X_2X_3 - 5.84X_2X_3 -} \\ &\mathbf{16.80X_1^2 - 8.20X_2^2 - 14.23X_3^2} \end{aligned} \qquad (7)$$

Where, Y_1, Y_2 and Y_3 are COD removal, turbidity removal and TSS removal respectively.

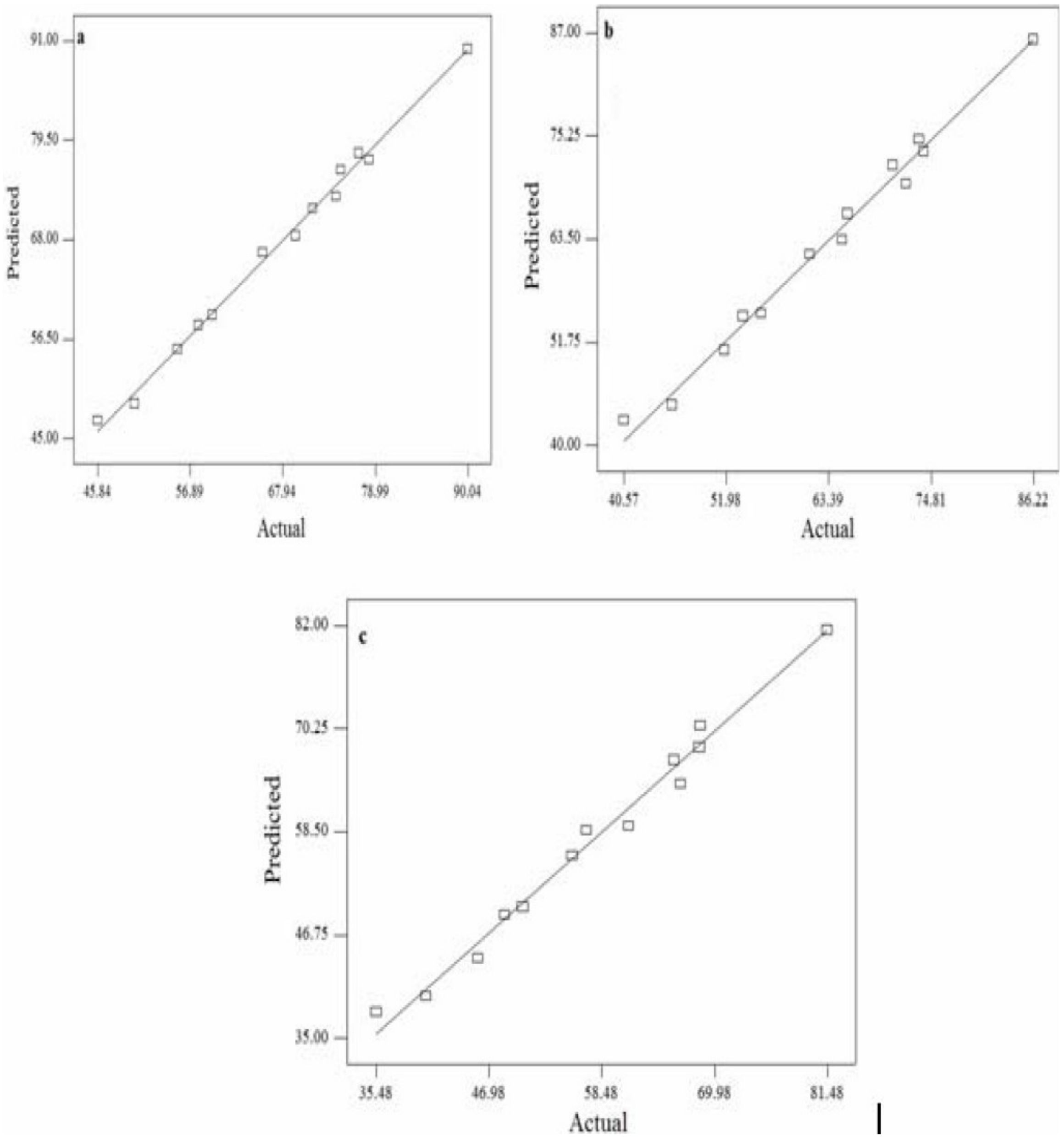

Figure 1 : Predicted versus actual for responses a) COD removal, b) Turbidity removal, c) TSS removal

Adequacy of models

The adequacy of developed mathematical models was evaluated by constructing diagnostic plots such as predicted versus actual and normal probability plots for the experimental data obtained from this study. Diagnostic plots such as predicted versus actual (Figure 1) help us to find out the relationship between predicted and experimental values and the data points on this plot lie very close to the diagonal line which indicates a good adequate agreement between experimental data and the data predicted by the developed models. Moreover, Normal probability plot (Figure 2) is a also suitable graphical method for judging residuals normality and they lie reasonably close on a straight line which confirms the normal distribution of the observed data and adequacy of the developed models.

Statistical analysis

Pareto analysis of variance (ANOVA) were used to analyze the BBD experimental data using *F* and *p*-values and it is shown in TABLE 4. The higher *F* values and lower *p*-values ($p < 0.0001$) of the developed mathematical models indicated that, the developed model was highly significant. The goodness of fit of the model was evaluated by the determination co-efficient

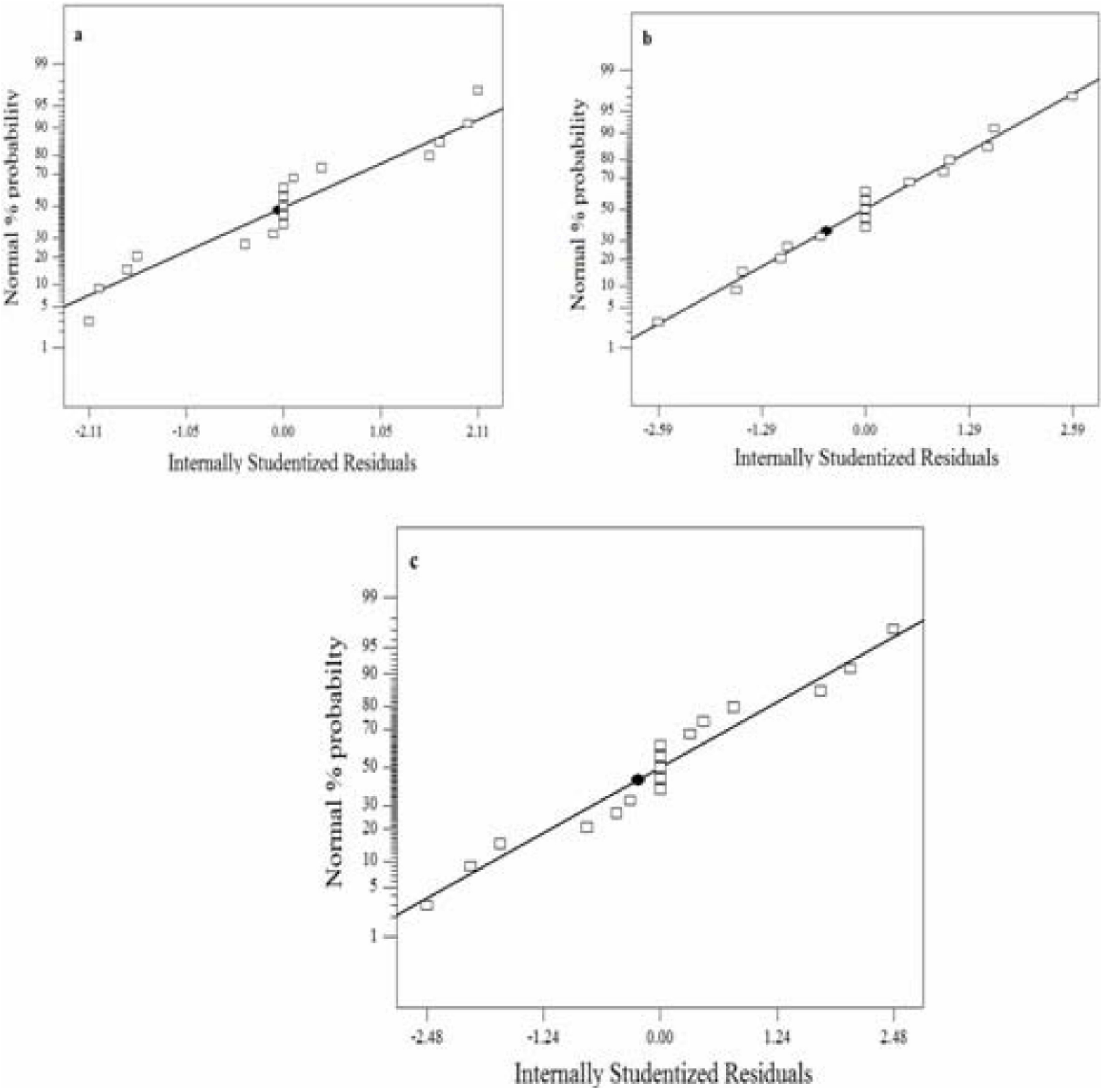

Figure 2 : Normal probability plots for responses a) COD removal, b) Turbidity removal, c) TSS removal

(R^2), adjusted determination co-efficient (R_a^2), predicted determination co-efficient (R_p^2) and co-efficient of variance (CV) and adequate precision (AP). The high R^2 values revealed that, the models are statistically significant. The value of and are in reasonable agreement with the developed models. Lower CV values and AP values clearly stated that, the deviations between experimental and predicted values.

Effect of process variables

Response surface contour plots were plotted from the developed models in order to study the individual and interaction effect among process variables on the responses and also used to determine the optimal condition of each factor for higher removal efficiency of COD, turbidity and TSS.

Effect of initial pH

pH is an important parameter influencing the performance of chemical coagulation process significantly. More over, the surface charge of the coagulating particle also varies with pH. The experiments were carried out at various initial pH and the results are shown graphically in Figures 3-5. From the figures, it is found that the percentage of COD, turbidity and TSS removal increased with increasing initial pH and reaches to a maximum level when pH is equal to 7. This is due to the formation of Fe(III) species in the form of Fe(OH)$_{3(s)}$ and later the percentage of COD, turbidity and TSS removal de-

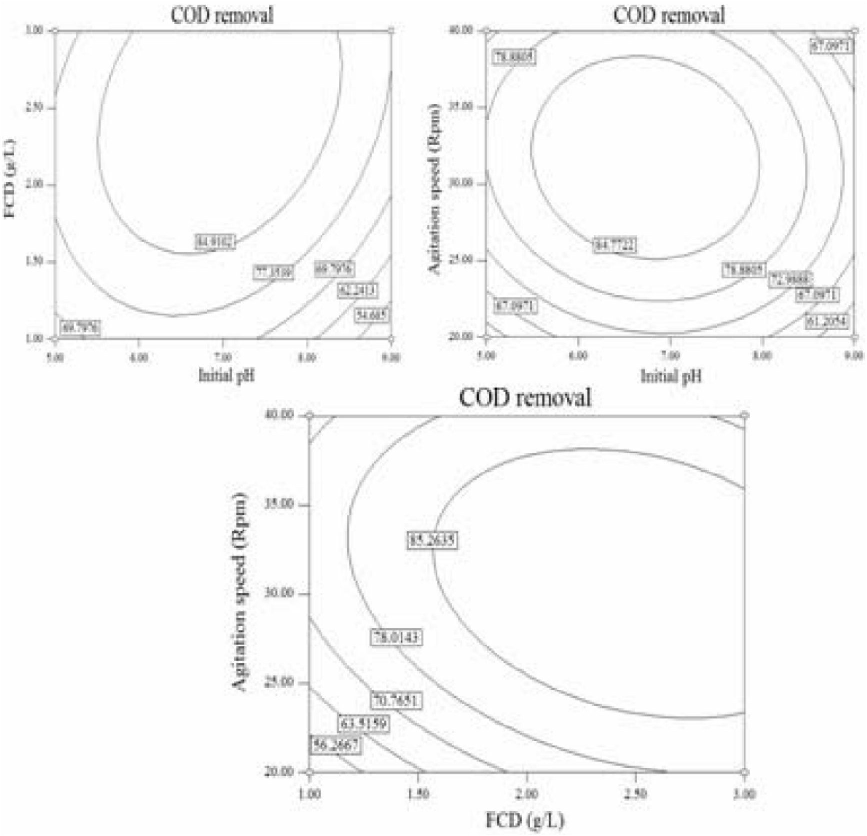

Figure 3 : Response surface contour plots for COD removal

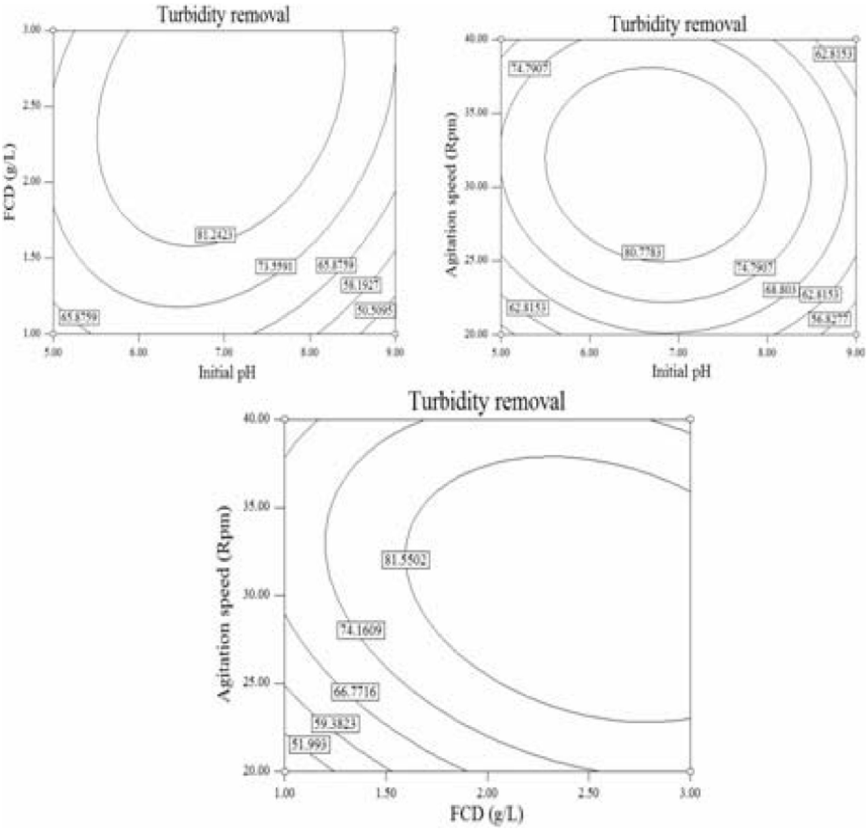

Figure 4 : Response surface contour plots for turbidity removal

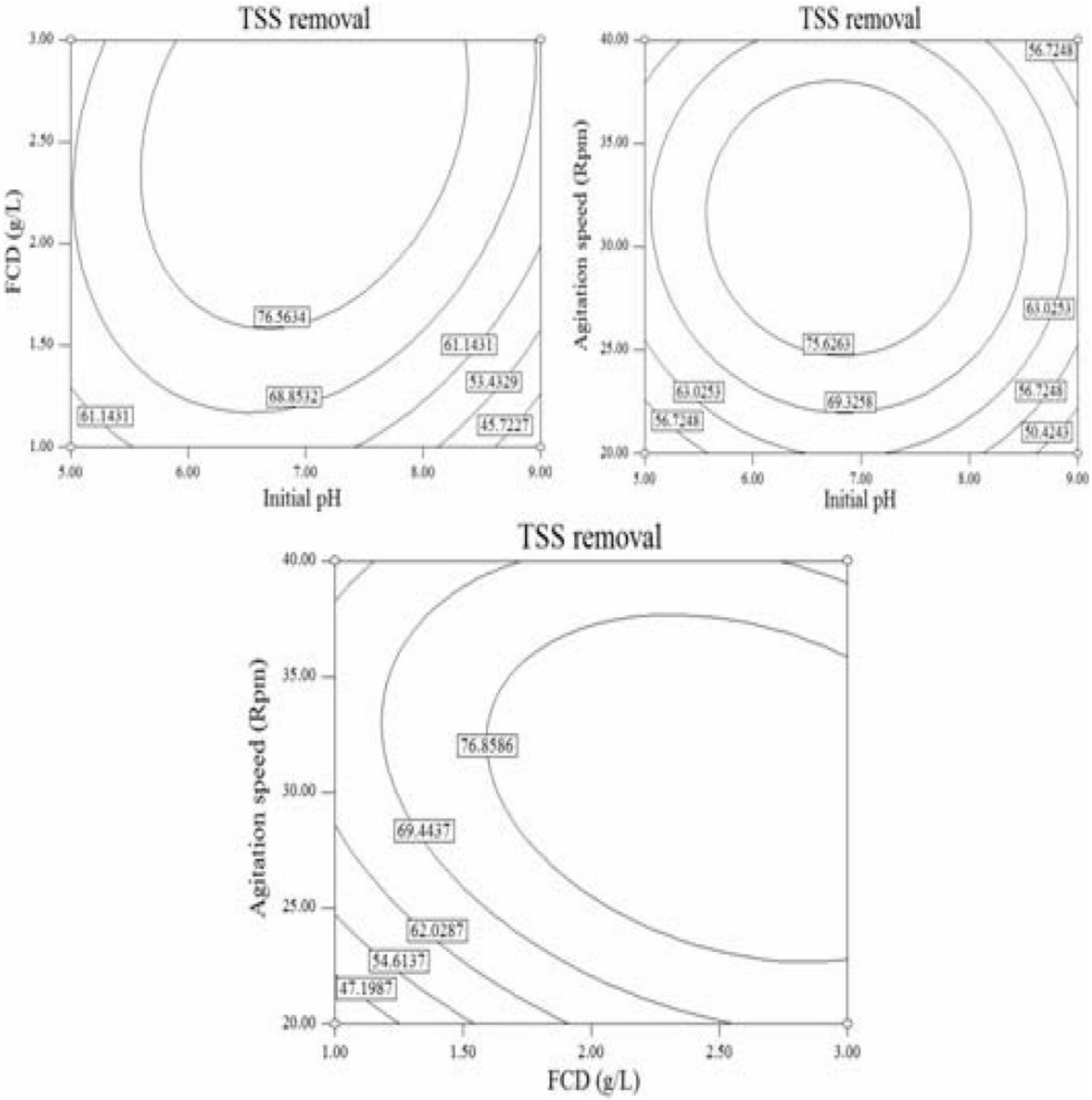

Figure 5 : Response surface contour plots for TSS removal

creased due to the formation of soluble $Fe(OH)_4^-$ which is not effective for removal of toxic pollutants[27].

Effect of ferric chloride dose

Ferric chloride dose is also an important parameter for controlling the reaction rate in chemical coagulation process. The experiments were carried out by varying the ferric chloride dose from 1 to 3 g/l and the results are shown in Figure 3-5. From the figure, it is found that the percentage of COD, turbidity and TSS removal increased with increasing ferric chloride because the production of $Fe(OH)_{3(s)}$ and hence an improvement in the removal efficiency. However it is noticed that increasing ferric chloride dose beyond 2.5 g/l did not show any significant effect on the percentage of COD, turbidity and TSS removal. This can explained the fact that, there is a formation of equilibrium between ferric chloride dose and toxic pollutants present in the wastewater[28].

Effect of agitation speed

The agitation speed is one of the most important parameter that affects the chemical coagulation process. In order to find out the optimum agitation speed for the higher removal efficiency of COD, turbidity and TSS; experiments were carried out in various agitation speed in the range of 20-40 rpm and results are depicted graphically in Figures 3-5. From the results, it was found that the removal efficiency of COD, turbidity and TSS were increased with increasing agitation speed upto 28 rpm. Thereafter, there is a drastic decrease in removal efficiencies of toxic pollutants. This is due to the fact that higher agiataion speed breaks the bond between the $Fe(OH)_{3(s)}$ and organic matters present in the rice processing industry wastewater[29].

Optimization

Simultaneous optimization of the multiple responses was carried out using Derringer's desired function methodology. This numerical optimization technique evaluates a point that maximizes the desirability function[30]. According to BBD results, optimal conditions to obtain the maximum removal of COD, turbidity and TSS were determined by Derringer's desired function methodology as follows pH 7, ferric chloride dose of 2.5 g/l, and agitation speed of 30 rpm. Under these conditions, the removal efficiency of COD, turbidity and TSS were found to be 90.80%, 87.35%, and 82.75% respectively with a desirability value of 0.996.

CONCLUSIONS

In this study, BBD was employed to study and optimize the process variables such as pH, ferric chloride dose and agitation speed on the removal COD, turbidity and TSS from rice processing industry wastewater using ferric chloride. From the results, it was observed that, the process variables have significant effects on the chemical coagulation process. Quadratic models were developed for predicting the responses. Optimum set of the independent variables was obtained by derringer's desired function methodology in order to remove the maximum levels of COD, turbidity and TSS. The optimal conditions were found to be: pH of 7, ferric chloride dose of 2.5 g/l, and agitation speed of 30 rpm. Under these conditions, the removal efficiency of COD, turbidity and TSS were found to be 91%, 87%, and 83% respectively. These results indicated that chemical coagulation process can be used as a primary treatment for the rice mill wastewater and an post treatment technique is required for the reuse of it.

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