



## Performance evaluation and optimization of electrocoagulation process to treat grey wastewater

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### ABSTRACT

In this present study, Box–Behnken response design was employed to optimize and investigate the effect of process variables such as electrolyte dose, stirring speed, and treatment time on total solids (TS), and chemical oxygen demand (COD) removal in electrocoagulation process (EC) to treat grey wastewater using iron electrodes. Regression analysis showed good fit of the experimental data to the second-order polynomial model with coefficient of determination ( $R^2$ ) value greater than 0.95 for both TS and COD removal. Three-dimensional response surface plots were generated from mathematical models in order to study the interactive effect of process variables on responses. Under the optimum operating conditions such as electrolyte dose of 1.4 g/l, stirring speed of 300 rpm, and treatment time of 8 min show the predicted removal efficiencies (96.56 and 93.60% for TS and COD removal, respectively) which were close to the experimental values.

*Keywords:* Electrocoagulation; Iron electrode; RSM; Box–Behnken design; Optimization

### 1. Introduction

Recent decades have witnessed a growing inconsistency between readily available sources of clean water and the growing demand associated with population growth. Therefore, nowadays an increasing attention has been given to the treatment of grey water [1,2]. Grey wastewater is one of the wastewater which includes water from baths, showers, hand basins, washing machines, dishwashers, and kitchen sinks and constitutes 50–80% of the total household wastewater, but excludes streams from toilets [3]. The discharges of untreated grey wastewater into the ecosystem have substantial impacts on the environment

and human health. Therefore, there is a critical need to develop an effective treatment process to treat grey wastewater in terms of removal efficiency of pollutants [4]. Electrocoagulation (EC) method for the treatment of wastewaters has gained considerable interest in nowadays, due to its simple operation, feasible for small scale requirements, cost-effective, and the task for maintenance is usually low [5]. Advantages of the EC over conventional technologies include high removal efficiency, compact treatment facility, and possibility of complete automation [6]. In the EC process for wastewater treatment, electrodes (Al or Fe) are dissolved by electrolysis, forming a range of coagulant species and metal hydroxides, which destabilize and aggregate the suspended particles or precipitates and adsorb dissolved contaminants [7]. Meanwhile

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this technology has been applied successfully to treat various wastewaters such as paper and mill wastewater, paint industry wastewater, textile wastewater, and poultry wastewater [8]. But, an extensive literature survey shows that there is no availability of research works for the treatment of grey wastewater using EC method and its mechanism is still not clearly understood. Therefore, further research and development is required to make the EC treatment method commercially competitive with other treatment processes to treat grey wastewater. Moreover, optimization of process variables in EC process such as pH, electrolyte dose, stirring speed, current density, and treatment time will give the in-depth knowledge regarding EC treatment method.

In recent years, multivariate statistical techniques have been preferred to optimize and investigate the wastewater treatment process, which are not possible to identify using the univariate method [9]. In addition, response surface methodology (RSM) is one of the multivariate statistical technique which is a very useful tool to reduce the time and cost of studies. This experimental design involves estimation of the coefficients in a mathematical model, predicting the response, and checking the adequacy of the model. [10]. Box–Behnken response surface design (BBD) is one of the RSM tools in which the response function largely depends on the nature of the relationship between the response and the independent variables, which has been used for several wastewater treatment process to optimize and investigate the independent variables [11]. An extensive literature survey shows that, there was no report available on treatment of grey wastewater using EC method via RSM. Hence, the main objective of the present study was to investigate and optimize the crucial EC operating parameters such as electrolyte dose, stirring speed, and treatment time on the removal of total solids (TS) and chemical oxygen demand (COD) to treat grey wastewater via RSM. The research outcome will be very useful to grey wastewater treatment plants to enhance its treatment efficiency and reduce the impact of grey wastewater on sustainable water resources.

## 2. Experimental method

### 2.1. Materials

Wastewater investigated in this study is collected from Erode, Tamilnadu, India. Sample collection, preservation, and characterization (pH, TS, COD) were done in accordance with the Standard Methods for the Examination of Wastewater. The characteristics of wastewater such as pH of 5.45, TS of 256 mg/l, and

COD of 638 mg/l show the harmful nature of wastewater. All the chemicals (HCl and NaOH) used in the study were analytically pure and purchased from local suppliers, Erode, Tamilnadu.

### 2.2. Experimental setup

Fig. 1 shows the schematic representation of the experimental apparatus. Experiments were conducted in electrochemical reactor of 31 capacity and in each run 1.6 l of the wastewater was filled in the reactor. DC power supply (Dolphin; 0–6 A and 0–30 V) connection was given to the electrodes in mono-polar mode and the experiments were carried out under constant current conditions of  $30 \text{ mA cm}^{-2}$  and actual pH. Electrodes were placed (5 cm gap) vertically and parallel to each other with electrodes of active area  $108 \text{ cm}^2$ . After the EC process, the DC power was switched off and the electrodes were dismantled. The treated wastewater collected and used for determination of TS and COD. All experiments were done in triplicates and the average values were recorded. Before each run, impurities on the iron electrode surfaces were removed by dipping for 5 min in acetone solution. The removal efficiency ( $R$ ) was calculated [12] using the following Eq. (1):

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

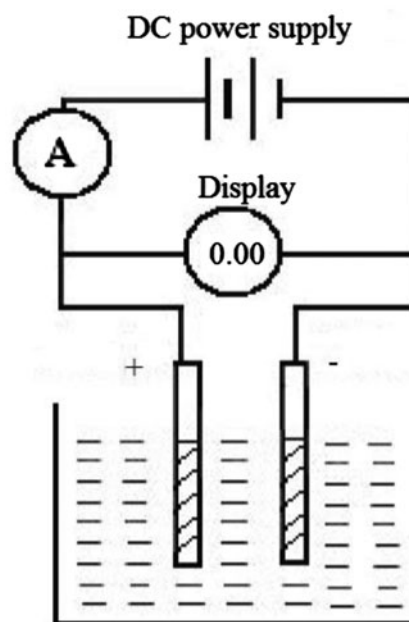


Fig. 1. Schematic diagram of EC process unit.

where  $Y_0$  (initial) and  $Y$  (final) represent the value of TS and COD, respectively.

### 2.3. Analytical method

pH was determined by using pH meter (Elico, LI120). TS (Buckner funnel) and COD (open refluxer) was determined according to the methods described in the American public health association standard methods.

### 2.4. Experimental design

In this present study, the RSM was used to determine the relationship between removal efficiencies (TS and COD) and crucial operating parameters such as electrolyte dose ( $X_1$ ), stirring speed ( $X_2$ ), and treatment time ( $X_3$ ) in EC method to treat grey wastewater. Electrolyte dose, stirring speed, and treatment time are only the effective parameters that affect the EC process (removal of TS and COD) significantly. So that, these parameters are investigated in this present study. The operating parameters ( $X_1$ – $X_3$ ) in uncoded form are converted [13] to coded form:  $x_1$ ,  $x_2$ , and  $x_3$  using the following Eq. (2)

$$x = \frac{X - ((X_{\max} + X_{\min})/2)}{(X_{\max} - X_{\min})/2} \quad (2)$$

Box–Behnken response surface experimental design (BBD) with 15 experiments with three center points was used to optimize and investigate the influence of crucial operating parameters on EC process and the total number of experiments [14] were calculated from the following Eq. (3):

$$N = 2K(K - 1) + C_0 \quad (3)$$

Then BBD experimental data were analyzed by multiple regression analysis to evaluate the adequacy of various models by using Stat ease Design Expert 8.0.7.1 statistical software package (Stat-Ease Inc., Minneapolis, USA). After the selection of suitable model, adequacy of model was verified using actual vs. predicted graphs, normal probability plots, and analysis of variance (ANOVA). Meanwhile, these models were used for the construction of 3-D contour response surface plots to predict the relationships between independent and dependent variables. After analyzing the polynomial equation on the responses, Derringer's

desired function methodology [15] was carried out in order to optimize the operating parameters to get higher removal efficiencies of TS and COD and it was validated by conducting additional experiments.

## 3. Results and discussion

### 3.1. Model development

The experimental data obtained from BBD are shown in Table 1. In order to find the effective mathematical model to describe the EC, multi regression analysis namely model summary statistics (Table 2) were done on the experimental data. From the Table 2, it was found that second-order polynomial is the best model to describe the EC process due to its higher  $R^2$  of model compared to the other models such as linear, interactive, and cubic. So, the second-order polynomial model was selected for further analysis and it was used to correlate the relationship between independent variables and responses [16,17]. The generalized mathematical form of second-order polynomial equation is given below:

$$y = \beta_0 + \beta_1x_1 + \beta_2x_2 + \beta_3x_3 + \beta_{11}x_1^2 + \beta_{22}x_2^2 + \beta_{33}x_3^2 + \beta_{12}x_1x_2 + \beta_{13}x_1x_3 + \beta_{23}x_2x_3 \quad (4)$$

where  $y$  is the response in coded units,  $\beta_0$  is a constant,  $\beta_1$ ,  $\beta_2$ , and  $\beta_3$  are the regression coefficients for linear effects,  $\beta_{11}$ ,  $\beta_{22}$  and  $\beta_{33}$  are the quadratic coefficients and  $\beta_{12}$ ,  $\beta_{13}$ , and  $\beta_{23}$  are the interaction coefficients. The final model obtained in terms of coded factors, after fitting of BBD experimental data are given in Eqs. (5) and (6):

$$Y_1 = 95.86 - 4.42X_1 - 2.38X_2 - 4.15X_3 - 2.04X_1X_2 - 1.14X_2X_3 + 1.39X_2X_3 - 6.92X_1^2 - 6.34X_2^2 - 9.28X_3^2 \quad (5)$$

$$Y_2 = 92.58 - 4.45X_1 - 2.38X_2 - 4.34X_3 - 2.43X_1X_2 - 1.15X_2X_3 + 1.13X_2X_3 - 6.40X_1^2 - 5.25X_2^2 - 8.48X_3^2 \quad (6)$$

where  $Y_1$  and  $Y_2$  are percentage removal of TS and COD, respectively;  $X_1$ ,  $X_2$ , and  $X_3$  are electrolyte dose, stirring speed, and treatment time, respectively. The adequacy of developed mathematical models were evaluated by constructing diagnostic plots such as predicted vs. actual for the experimental data obtained from this study and it is shown in Fig. 2(a) and (b).

Table 1  
Box–Behnken experimental design (BBD) with experimental data

Run order	Electrolyte dose ( $X_1$ )	Stirring speed ( $X_2$ )	Treatment time ( $X_3$ )	Removal	
				TS ( $Y_1$ )	COD ( $Y_2$ )
1	1	250	10	87.56	85.72
2	1.5	350	10	95.86	92.58
3	1	350	15	81.57	79.36
4	2	350	15	70.68	67.85
5	1.5	450	15	74.56	73.58
6	2	450	10	73.58	71.29
7	2	250	10	82.56	82.01
8	1.5	250	5	88.69	86.39
9	1.5	250	15	76.39	74.98
10	1.5	350	10	95.86	92.58
11	1	350	5	86.35	85.27
12	2	350	5	80.04	78.34
13	1.5	350	10	95.86	92.58
14	1	450	10	86.72	84.72
15	1.5	450	5	81.32	80.46

Table 2  
Model summary statistics for responses

Source	Std. Dev.	$R^2$	Adjusted $R^2$	Predicted $R^2$	Press	Remarks
<i>Model summary statistics for TS removal</i>						
Linear	7.3481	0.3634	0.1898	0.0983	841.29	
2FI	8.3998	0.3950	-0.0587	-0.2810	1195.28	
Quadratic	0.7980	0.9966	0.9904	0.9454	50.95	Suggested
Cubic	0.0000	1.0000	1.0000		+	Aliased
<i>Model summary statistics for COD removal</i>						
Linear	6.6775	0.4192	0.2608	0.1628	707.02	
2FI	7.5538	0.4595	0.0541	-0.1691	987.33	
Quadratic	0.7832	0.9964	0.9898	0.9419	49.07	Suggested
Cubic	0.0000	1.0000	1.0000		+	Aliased

From Fig. 2, it is observed that, the data points on this plot lie very close to the diagonal line, because residuals for the prediction of each response is minimum and it indicates a good agreement between experimental data and the data predicted by the developed models [18]. Moreover, residuals are thought as elements of variation, unexplained by the fitted model and then it is expected that they occur according to a normal distribution. Normal probability plots are a suitable graphical method for judging residuals normality. The observed residuals are plotted against the expected values, as they lie reasonably close on a straight line and show no deviation of the variance (Fig. 3(a) and (b)). These results can confirm the normal distribution of the data and confirm the adequacy of the models [19].

### 3.2. Significance of the developed model

The ANOVA was used to analyze the significance of the developed mathematical model equations based on their corresponding  $F$  values and  $p$ -values and it is shown in Table 3. The higher model  $F$  values (162 for TS and 152 for COD) and lower  $p$ -values ( $p < 0.0001$ ) demonstrated that, the developed model was highly significant. The effectiveness of fit of the model was analyzed by the determination co-efficient ( $R^2$ ), adjusted determination co-efficient (adj- $R^2$ ), predicted determination co-efficient (pre- $R^2$ ), and co-efficient of variance (CV), and adequate precision (AP). The high  $R^2$  values of the both models exposed that, the models are statistically significant. The value of pre- $R^2$  of models are in reasonable agreement with the value of

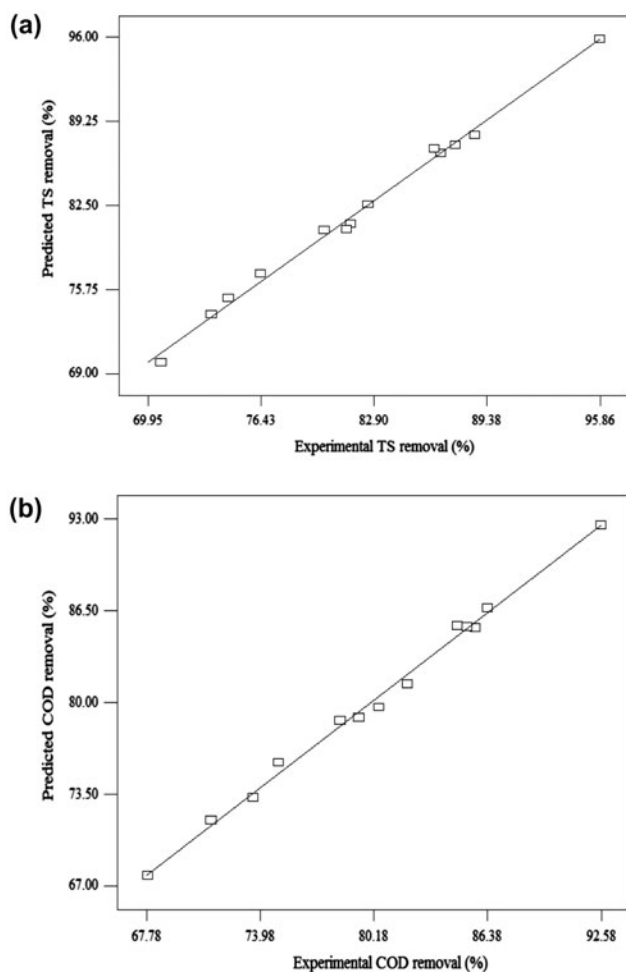


Fig. 2. Actual vs. predicted plots for the removal of TS (a) and COD (b).

adj- $R^2$ . Lower CV values clearly stated that, the deviations between experimental and predicted values are low and also showed a high degree of precision and reliability of the conducted experiments [20]. Finally, AP value > 35 of both the developed mathematical models indicates the best fitness of the models.

### 3.3. Effect of process variables on EC process

Three-dimensional response surface plots were plotted from the developed models in order to study the individual and interactive effect of the process variables on the responses and also used to determine the optimal condition of each factor for maximum removal of TS and COD from grey wastewater using EC process. The obtained results were shown in Figs. 4 and 5. Electrolyte dose is one of the primary parameters which influences the performance of EC

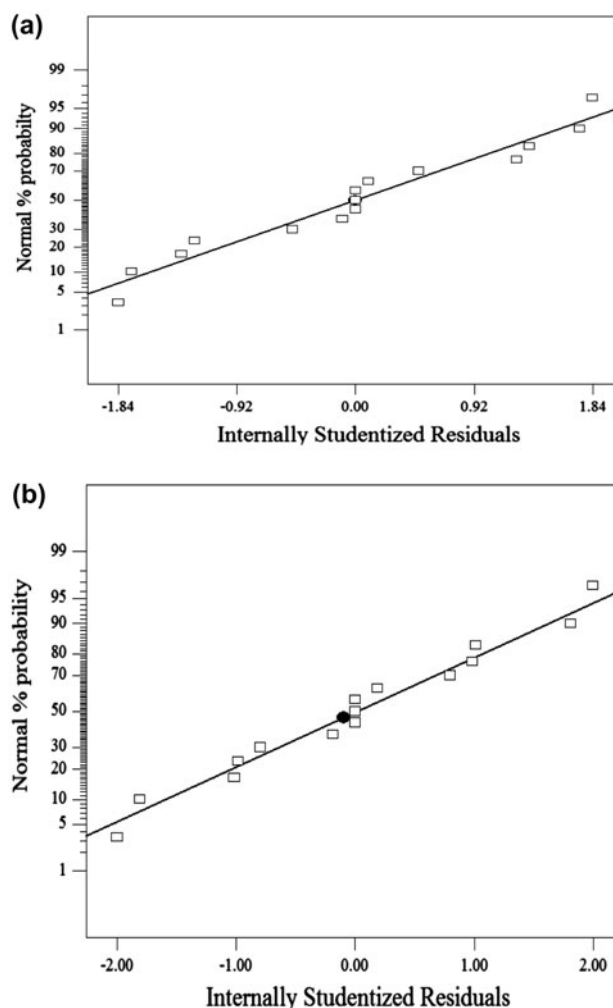


Fig. 3. Normal probability plots for TS and COD removal.

process. In order to evaluate its effect various electrolyte doses were varied and examined. From the results, it was observed that, the removal of TS and COD were increased linearly with increasing electrolyte dose from 1 to 1.5 g/l (Figs. 4 and 5). This is mainly due to the formation of hypochlorite ions, which oxidizes the pollutants present in the wastewater effectively, thus the removal of TS and COD increased during EC process. The following reactions (Eqs. (7–9)) take place in the wastewater, when NaCl is added [21].

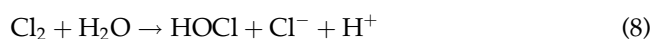


Table 3  
ANOVA results for TS and COD removal data

Source	TS removal				COD removal			
	Sum of squares	Mean square	F-value	p-value	Sum of squares	Mean square	F-value	p-value
Model	929.86	103.31	162.23	<0.0001	841.43	93.49	152.42	<0.0001
X <sub>1</sub>	156.11	156.11	245.13	<0.0001	158.24	158.22	257.99	<0.0001
X <sub>2</sub>	45.22	45.22	71.00	0.0004	45.362	45.38	73.95	0.0004
X <sub>3</sub>	137.78	137.78	216.34	<0.0001	150.42	150.42	245.24	<0.0001
X <sub>12</sub>	16.56	16.56	26.01	0.0038	23.61	23.61	38.50	0.0016
X <sub>13</sub>	5.24	5.24	8.23	0.0350	5.24	5.21	8.54	0.0329
X <sub>23</sub>	7.67	7.67	12.04	0.0178	5.13	5.13	8.36	0.0341
X <sub>1</sub> <sup>2</sup>	176.68	176.68	277.42	<0.0001	151.06	151.06	246.28	<0.0001
X <sub>2</sub> <sup>2</sup>	148.29	148.29	232.85	<0.0001	101.72	101.72	165.84	<0.0001
X <sub>3</sub> <sup>2</sup>	318.14	318.14	499.55	<0.0001	265.43	265.43	432.76	<0.0001
CV%	0.95				0.94			
AP	39.76				38.78			

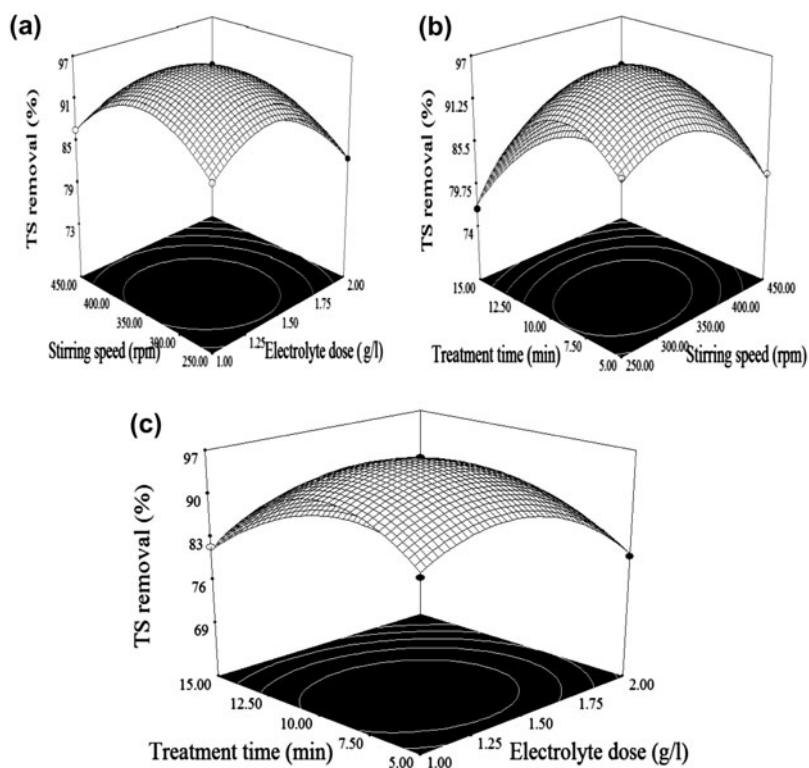


Fig. 4. Response surface contour plots for the effects of variables on the TS removal.

As shown in above reactions, when sodium chlorides are added into the solutions the products discharged from anode are  $\text{Cl}_2$  and  $\text{OCl}^-$ . The  $\text{OCl}^-$  itself is a strong oxidant, which is capable of oxidizing organic molecules present in wastewater. However, electrolyte dose beyond 1.5 g/l resulted in lower removal of TS

and COD. We confirm the optimal dose of NaCl reported in reference [22]. It also explained by the fact that the increase in the removal efficiency may be attributed to a change in the ionic strength. The higher ionic strength will generally cause an increase in current density at constant cell voltage.

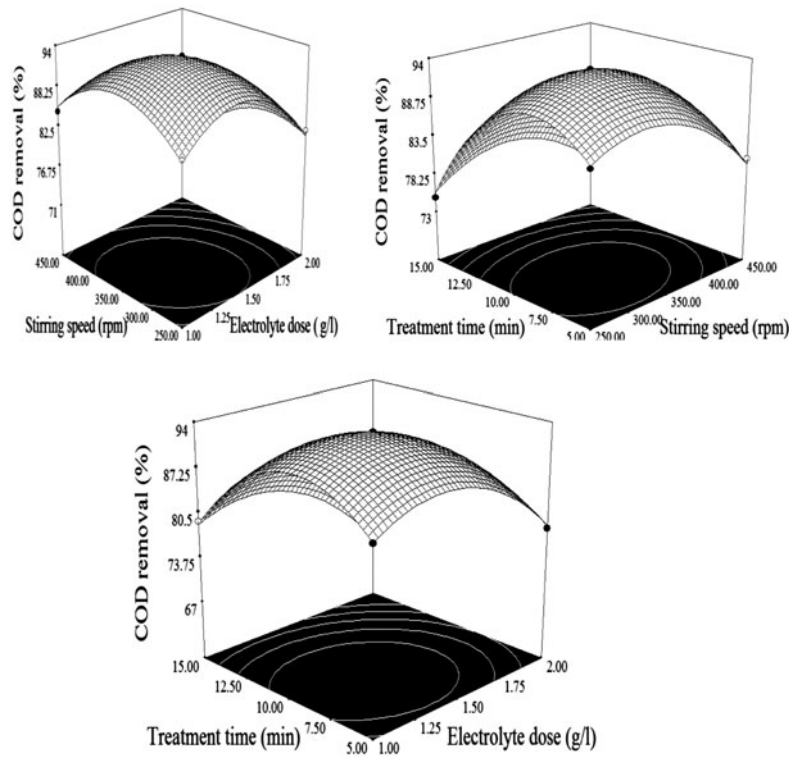


Fig. 5. Response surface contour plots for the effects of variables on the COD removal.

Moreover, stirring speed is also one of the important factors which affects the EC process considerably. It can be seen from (Figs. 4 and 5) that the increase in stirring speed significantly increases the removal of TS and COD up to 350 rpm. This confirms the fact that the removal efficiency is diffusion controlled, the increase in rotational speed leads to increase in the intensity of turbulence and reduces the diffusion layer thickness at the electrode surface and improves the mixing conditions in the electrolyte bulk [23] as well as the formed  $\text{Fe}(\text{OH})_3$  flocs were attached together easily with pollutants and removes the TS and COD via adsorption mechanism [24,25]. Beyond stirring speed of 350 rpm, the formed  $\text{Fe}(\text{OH})_3$  are degraded and adsorbed pollutants gets desorbed.

Additionally, the treatment time plays an important role in EC process. The treatment time not only determines the coagulant dosage rate but also the bubble production rate, size, and the flocs growth, and they have strong influence on treatment efficiency of the EC process. From the results, it was observed that the removal efficiency of TS and COD was increased with the increasing treatment time up to 8 min (Figs. 4 and 5). This is mainly due to direct and indirect oxidation. When sufficient voltage is developed across the electrodes, direct oxidation takes place near the anode, due to the release of electrons by the organic

compounds in order to maintain the flow of current, whereas indirect oxidation occurs due to the strong oxidants that form during the reaction. Also it can be described by the fact that amount of formation  $\text{Fe}(\text{OH})_3$  flocs increased with respect to increase in treatment time due to dissolution of electrode, thus removal efficiency of TS and COD increased during EC process. The amount of iron electrode dissolved ( $D$ ) was calculated [26] as follows:

$$D = \frac{ItA}{nFVC_i \frac{Y_t}{100}} \quad (10)$$

where  $D$  is unit electrode material demand (kg/kg);  $n$ —number of electrons involved in oxidation/reduction;  $I$ —current density (A);  $t$ —time (S);  $F$ —Faraday constant (C/mol);  $A$ —Atomic mass of electrode material (g/mol);  $Y_t$ —removal efficiency at time  $t$  (%);  $V$ —Volume of treated solution ( $\text{m}^3$ ).

### 3.4. Optimization

Finally, in this present study multi response optimization was carried out using Derringer's desirability function methodology. This numerical optimization technique evaluates a point that maximizes the

desirability function. According to BBD results, optimal operating conditions to obtain the maximum removal of TS and COD from grey wastewater is found to be: electrolyte dose of 1.4 g/l, stirring speed of 300 rpm, and treatment time of 8 min. Under these conditions, the predicted removal efficiency of TS and COD was found to be 96.56 and 93.60%, respectively, with a desirability value of 0.926.

### 3.5. Validation

The suitability of the optimized conditions for predicting the optimum response values was tested using the selected optimal conditions. Additional experiments using the predicted optimum conditions were carried out and the mean values were obtained from the experimental (96% TS and 94% COD removal) were in agreement with the predicted values obtained from Derringer's desirability function method [27]. The good correlation between these observed results and predicted values indicate the reliability of BBD incorporate desirability function method and it could be effectively used to optimize the operational conditions of EC for TS and COD removal.

### 4. Conclusion

In this present study, the performance of EC process of grey wastewater was studied focusing on the influence of operating parameters such as electrolyte dose, stirring speed, and treatment time by using RSM coupled with BBD. The results obtained from the present study revealed that RSM was a suitable method to optimize the operating conditions of EC for TS and COD removal. The response surface models developed in this study for predicting TS and COD removal efficiency were considered to be adequately applicable. ANOVA showed a high coefficient of determination value ( $R^2 > 0.93$ ) for responses, ensuring a satisfactory adjustment of the second-order regression model with the experimental data. The optimum conditions when the 96.56 and 93.60% for TS and COD removal was achieved were electrolyte dose of 1.4 g/l, stirring speed of 300 rpm, and treatment time of 8 min. The results of this study indicate that EC is applicable under reasonable operating conditions for higher removal of TS and COD from grey wastewater.

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