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Real Textile Dye Wastewater Treatment by Electrochemical Oxidation: Application of Response Surface Methodology (Rsm)

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Abstract: The influence of different variables on the electrochemical oxidation process for the treatment of textile processing industry waste water was investigated in a batch reactor. Box-Behnken statistical experiment design and response surface methodology were applied to study the effects of operating conditions such as reactor volume, electrolysis time and current density on chemical oxygen demand (COD) removal efficiency. The positive and negative effects of variables and interaction between them on COD removal were determined. This approach enabled quadratic models which were adequate to foretell responses and to carry out optimization under the conditions studied. The optimal conditions were found to be reactor volume 300 mL, electrolysis time 6 h and current density 4.0 A.dm^{-2} with a COD removal efficiency o f 97.17%. The model developed was in good agreement with experimental results.

Keywords: Electrochemical oxidation, Textile industry waste water, Batch reactor, COD removal, Box-Behnken statistical design, Response surface methodology.

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1. Introduction

Textile industry generates huge quantities of waste water as dye-bath waste water and wash waste water due to dyeing, finishing, and other intermediate processes. The dye-bath waste water which constitutes 15% (by volume) of the total waste water has a high COD, intense colour, large amount of suspended solids, low biodegradability and high salt content [1] and is difficult to treat [2]. The physical, biological and chemical treatment methods or their combinations may not be sufficiently effective in the treatment of dye bath waste water from textile industries for removing the pollutants present in them [3-11]. The electrochemical treatment can be more effective in reducing the concentration of organic contaminants and colour and increasing the biodegradability index BI [12-16]. However, several factors affect the electrochemical treatment performance and to achieve high performance, optimal conditions must be determined [17, 18].

The response surface methodology (RSM) has become an important tool in the design and optimization of processes as well as in improving existing designs [19-22]. The main types of RSM designs include three level factorial design, central composite design (CCD), Box-Behnken design and D-optimal design [23]. Among them the Box-Behnken design is stated to be more efficient [24] and requires fewer experiments [25–28]. In this study, Box-Behnken design coupled with RSM is used to verify the COD reduction efficiency as response factor for different variables in the electrochemical oxidation of textile industry waste water.

2. Materials and methods

2.1. Effluent and electrodes

The textile waste water to be treated was obtained from a textile processing dyeing industry situated in Tiruppur, Tamil Nadu, India. American Public Health Association (APHA) procedures [29] were followed to analyze the effluent samples and their characteristics are given in Table 1. In all the experiments RuOx – TiOx [28] coated titanium substrate insoluble anode (TSIA) with a stainless steel cathode were used. The reagents used were of analytical grade.

Table 1 Characteristics of textile waste water

Characteristics	Dye bath effluent
Color	Turquoise blue
Initial COD (mgL^{-1})	5400
Initial BOD ($mg L^{-1}$)	180
BI (=BOD/COD)	0.033
Chloride content (mgL^{-1})	41595
Total dissolved solids(mgL^{-1})	58000
Suspended solids(mgL^{-1})	1800
pH	8

2.2. Batch Reactor

The schematic diagram of the batch reactor setup is shown in Figure 1. The reactor is a cylindrical glass vessel closed with a lid designed to facilitate the connection of reference electrode through the salt bridge and to enable sample collection. The lid is fitted with electrodes of 7.7cm x 6.5 cm size with constant gap between them. A regulated power supply was employed for supplying the required electrical energy and a magnetic stirrer was used for stirring the effluent. Experiments were carried out with the dye bath effluent of initial COD 5400 mg L^{-1} and BOD 180 mg L^{-1} under galvanostatic conditions at room temperature. The experiments were conducted under various operating conditions of reactor hold up volumes (300 mL, 400 mL and 500 mL) and current density (2.0, 3.0 and 4.0 $A.dm^{-2}$) for 6 hours and the cell voltage and anode and cathode potentials were recorded periodically. Samples, collected every hour and kept at acidic conditions were analysed for COD. The chlorides, hypochlorites and chlorates concentrations were determined after electrolysis and energy consumption, rate constant and percentage COD reduction were experimentally computed.

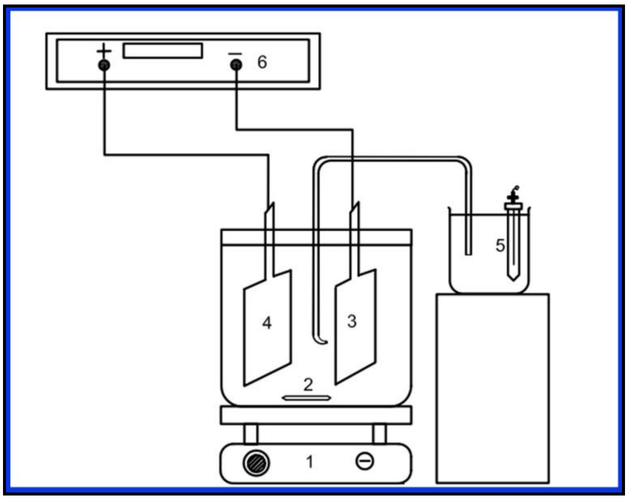

Fig. 1 A schematic view of batch experimental setup (1. Magnetic stirrer 2. Stirrer 3. Cathode 4. Noble oxide coated catalytic anode 5. Reference electrode 6. Regulated power supply)

2.3. Analysis

COD was determined by the dichromate closed reflux method. The test sample was mixed with the commercially available solution containing potassium dichromate, sulfuric acid and mercuric sulfate and the mixture was incubated at 150°C, for 2 hours in a thermo reactor (Model TR 620- Merck). Absorbance of untreated and treated samples of dye bath effluent was measured at appropriate wavelengths using UV-Visible

Spectrophotometer (Spectroquant NOVA 60, Merck), BOD was estimated using Winkler's method, following the American Public Health Association (APHA) procedures [29]. The effluent temperature was maintained constant at 35°C during electrolysis. The hypochlorite and chlorate concentrations were found to be 5 mg L^{-1} and 15.25 mg L^{-1} respectively. Experiments were repeated till the error found was less than 3%.

2.4. Design and performance of batch reactor

During electrolysis, if a constant operating current density is applied, the rate of degradation of organic contaminant will remain constant under a given set of experimental conditions, but it will vary if the applied current density is changed. The electrochemical reaction rate for the removal of COD in a batch reactor can be expressed as [15]:

$$-\frac{dC}{dt} = \frac{i}{zF} = k_h aC \qquad (1)$$

$$\frac{C}{C_0} = \exp(-k_h aC) \qquad (2)$$

where C represents COD of the effluent in the batch reactor, i is the current passed during electrolysis time t, a is specific electrode area (A_e/V_R), A_e electrode area, V_R reactor volume, k_h is pseudo first order heterogeneous rate constant, F is the Faraday constant (96485.3 coulomb mol^{-1}) and z is the number of electrons transferred per mole of reaction. The unconverted species (1-X) or (C/C_o) [by defining the extent of degradation, X= (C_o - C)/C_o], decreases exponentially with time, according to equation (2).

The rate of degradation is controlled by the operating current density and it is assessed in terms of the heterogeneous rate constant k_h (m s^{-1}) by monitoring the extent of COD removal. In electrochemical conversion, the high molecular weight aromatic compounds and aliphatic chains are broken to intermediate products for further processing. In electrochemical oxidation, the organics are oxidized to CO_2 and H_2O. The instantaneous current efficiency of the electrolysis can be calculated using the following expression [15]:

$$\text{Instantaneous Current Efficiency (ICE)} = \frac{\Delta COD \times V_R}{(16It/2F)} = \frac{C_o \times X \times V_R}{(16It/2F)} \qquad (3)$$

Process performance is defined in two ways, one with respect to the extent of reaction completion and the other with respect to the energy consumption, E (kW h kg^{-1}). Computation of the instantaneous current efficiency (ICE) indicates the path and course of the targeted reaction by monitoring the extent of COD removal (Equation 2). The utilization of energy in processing a unit quantity of the targeted reaction is found by Equation (4).

$$\text{Specific Energy Consumption, E(t)} = \frac{VIt \times 10^3}{\Delta COD \times V_R} = \frac{VIt \times 10^3}{C_o \times X \times V_R} \qquad (4)$$

2.5. Experimental design and statistical model

The effects of the three independent variables of reactor volume (X_1), electrolysis time (X_2) and current density (X_3) on the response function of COD reduction were investigated employing Box-Behnken statistical experimental design and the response surface methodology. The low, centre and high levels of each variable are designated as –1, 0 and +1 respectively as shown in Table 2. These experimental levels were decided based on previous experiments and literature values [21, 22].

Table 2 Factor levels for the experiments

Factors	Code	Coded values	Factor values
Reactor volume (ml)	x_1	–1, 0, +1	300, 400, 500
Electrolysis time (h)	x_2	–1, 0, +1	4, 5, 6
Current density (A/dm^2)	x_3	–1, 0, +1	2, 3, 4

The objective function or dependent variable was COD removal efficiency (Y). The variables in uncoded forms were converted to coded forms: x_1 , x_2 and x_3 using the following equation [17]:

$$x = \frac{X - \left(\frac{X_{max} + X_{min}}{2}\right)}{\left(\frac{X_{max} - X_{min}}{2}\right)} \qquad (5)$$

Box-Behnken design requires the number of experiments according to $N = K^2 + K + C_p$, where (K) is the factor number and (C_p) is the replicate number of the central point [16 – 20]. The number of experiments in this study was 15 based on 3 levels and a 3 factor experimental design, with three replicates at the centre of the design for estimation of a pure error and sum of squares. Minitab 17 (PA, USA) software could be used to analyze experimental data from the Box-Behnken design and fitted to a second-order polynomial model using [17],

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j \qquad (6)$$

where Y is predicted response for COD removal, β_0 is the intercept parameter and β_i ,β_{ii} and β_{ij} are parameters for linear, quadratic and interaction factor effects, respectively.

3. Results and discussion

3.1. Main effects and interaction effects plot for COD removal efficiency

The main effects plot (Figure 2a) and the interaction plot for COD removal efficiencies (Figure 2b) were developed based on Table 3 in which the uncoded values of the test variables and the COD removal efficiencies obtained experimentally are shown.

Table 3 Design matrix in uncoded units and the experimental responses (Electrode area = 50 cm^2)

Run No.	Reactor volume (ml)	Electrolysis time (h)	Current density (A/dm^2)	COD removal efficiency (%)	Cell Voltage (V)	Energy Consumption (kWh/kgCOD)	Instantaneous Current Efficiency (ICE)	$k_h \times 10^6$ (m/s)
1.	400	6	2	64	3.26	14.15	0.7719	3.78
2.	300	5	4	95	3.84	24.95	0.5156	9.99
3.	300	6	3	89	3.58	22.35	0.5367	6.13
4.	400	4	2	54	3.26	11.18	0.9769	4.31
5.	500	6	3	78	3.69	15.77	0.7839	7.01
6.	400	5	3	76	3.53	16.13	0.7333	6.34
7.	400	4	4	80	3.87	17.92	0.7236	8.94
8.	500	5	2	52	3.21	11.43	0.9407	4.08
9.	400	6	4	89	3.87	24.16	0.5367	8.18
10.	300	5	2	70	3.33	14.68	0.7598	4.01
11.	400	5	3	76	3.53	16.13	0.7333	6.34
12.	300	4	3	78	3.58	17	0.7055	6.31
13.	500	4	3	62	3.69	13.23	0.9347	6.72
14.	400	5	3	76	3.53	16.13	0.7333	6.34
15.	500	5	4	82	3.83	17.3	0.7417	9.53

Figure 2a shows the effect of the three independent experimental variables on the COD removal efficiency. It is seen that the current density and electrolysis time affect the COD removal efficiency positively with greater efficiency at high level (+1) than at low level (–1).

Table 4 Estimated regression coefficients and corresponding ‘t’ and ‘p’ values for COD

Factor	Coefficient of the model in uncoded units	‘t’ value	‘p’ value	Significance level (%)
β_0	46.5	1.38	0.227	77.3
β_1	-0.3225	-4.09	< 0.009	99.1
β_2	12.75	1.42	0.214	78.6
β_3	28.25	4.06	< 0.010	99
β_{11}	0.0002	2.33	0.068	93.2
β_{22}	-1.125	-1.40	0.222	77.8
β_{33}	-3.125	-3.88	< 0.012	98.8
β_{12}	0.01250	1.61	0.168	83.2
β_{13}	0.01250	1.61	0.168	83.2
β_{23}	-0.250	-0.32	0.760	24

Table 5 ANOVA results for the percentage COD removal

Source	Degree of freedom	Sum of squares	Mean squares	F-value	Prob > F
Model	9	2158.93	239.881	99.95	< 0 Significant
A – reactor volume	1	40.07	40.067	16.69	< 0.009
B – electrolysis time	1	4.86	4.859	2.02	0.214
C – current density	1	39.66	39.655	16.52	< 0.010
AB	1	6.25	6.250	2.60	0.168
AC	1	6.25	6.250	2.60	0.168
BC	1	0.25	0.250	0.10	0.760
A^2	1	12.98	12.981	5.41	< 0.068
B^2	1	4.67	4.673	1.95	0.222
C^2	1	36.06	36.058	15.02	< 0.012
Residual error	5	12	2.4		
Lack-of-fit	3	12	4	*	*
Pure error	2	0	0		
Total	14	2170.93			
			R-Squared		0.9945
			Adj.R-Squared		0.9845
			Pred R-Squared		0.9116

Contrarily, the reactor volume affects the COD removal negatively with higher removal at low level (–1); however, it is seen that all the variables significantly affect the response function. This is indicated in Table 4 in which the regression coefficients and corresponding ‘t’ and ‘p’ obtained by ANOVA analysis are presented. The factors reactor volume (X_1), electrolysis time (X_2) and current density (X_3) are found to have the levels 99.1%, 78.6% and 99.1% respectively indicating X_1 and X_3 are more significant (> 95% confidence level) than X_2 (< 95% confidence level).

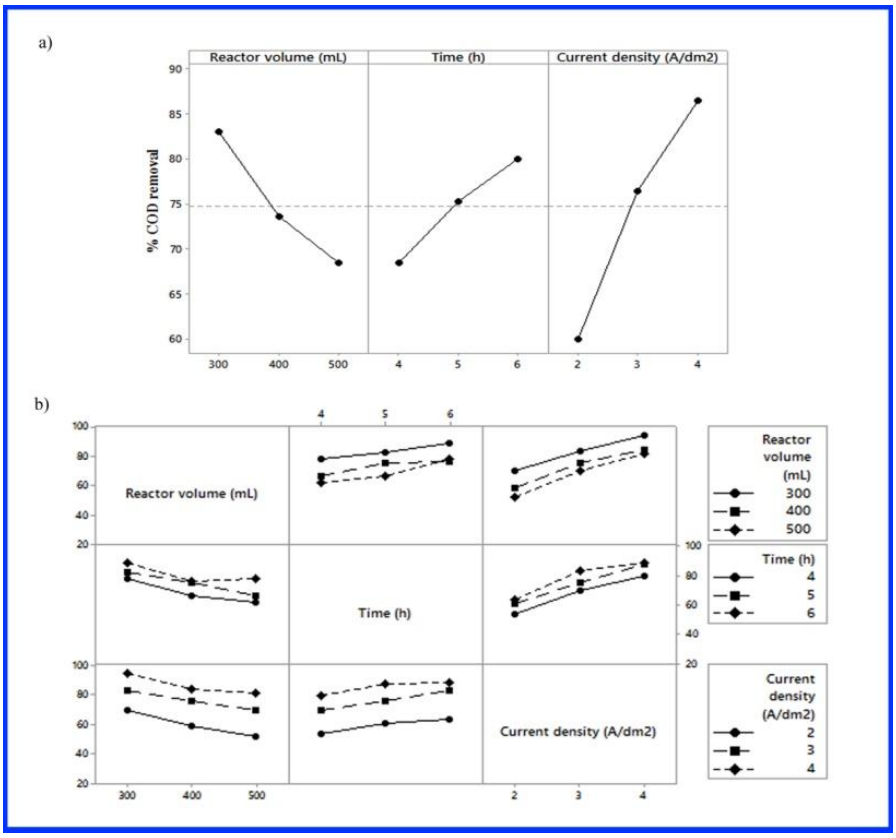

Fig. 2 a) Main Effect Plot for COD removal efficiencies b) Interaction plot for COD removal efficiencies

The degree of interaction among the factors is shown in Figure 2b. If the response variable changes differently with two design variable, an interaction may occur [24, 28] and the effects are dependent on both variables. The interaction effect between time and reactor volume is crossed while other interaction effects are almost parallel. The significance levels of interaction effects X_{12}, X_{13} and X_{23} are found to be 83.2%, 83.2% and 24% respectively (Table 4), indicating that interaction effects are insignificant as ANOVA analysis showed a < 95% confidence level. The ultimate RSM model in terms of uncoded factors was determined to calculate COD removal efficiency,

$$Y = 46.5 - 0.3225 X_1 + 12.75 X_2 + 28.25 X_3 + 0.0002 X_1^2 - 1.125 X_2^2 - 3.125 X_3^2 + 0.01250 X_{12} + 0.01250 X_{13} - 0.250 X_{23} \quad (7)$$

where Y is COD removal. Table 5 shows a higher F value of 99.95 and a lower F value for lack of fit of 0, compared to the tabulated values of 4.77 and 8.1 respectively. The coefficient of determination of the model $R^2 = 0.9945$ also confirms the statistical significance of the model. The model fails to explain only 0.55% of the variability in response. Further the experimental values are found to be close to the values calculated by the predictive equations (Figure 3) confirming further the model's significance and prediction accuracy.

3.2. Analysis of response surface plots for COD removal efficiency

The optimal conditions for achieving maximum COD removal were obtained by partially differentiating the RSM model, equating it to zero and solving the resulting equation (Monte-Carlo technique) [16]. For the maximum COD removal efficiency of 97.17%, the conditions were found to be reactor volume 300 mL, electrolysis time 6 h and current density 4 A. dm^{-2}.Three dimensional response plots developed for the optimized factor levels are shown in Figures 4a, 4b, and 4c. Figure 4a illustrates the response surface assuming the reactor volume and electrolysis time as independent factors. The COD removal increased with electrolysis time and at the initial electrolysis time of 4 h, the COD removal was observed to decrease with increase in reactor volume. It means that for treating larger volumes of effluent more time is required as depicted by Figure 4a, which predicts that when 335 mL of effluent is treated for 4 h at a current density of 4 A.dm^{-2}, 85% COD removal is possible as per RSM model.

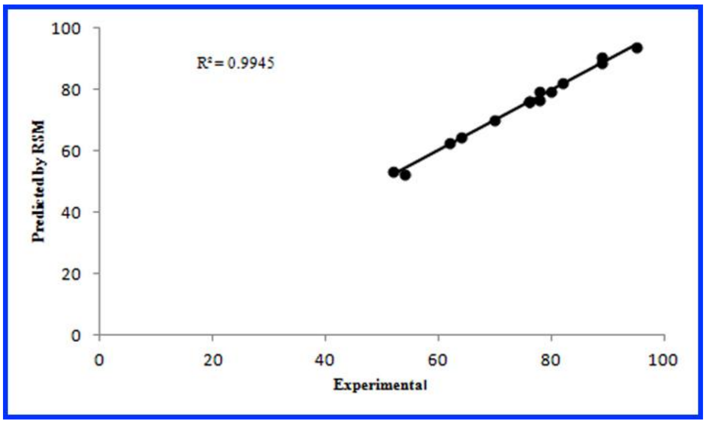

Fig. 3 Comparison of experimental COD vs. COD predicted by RSM

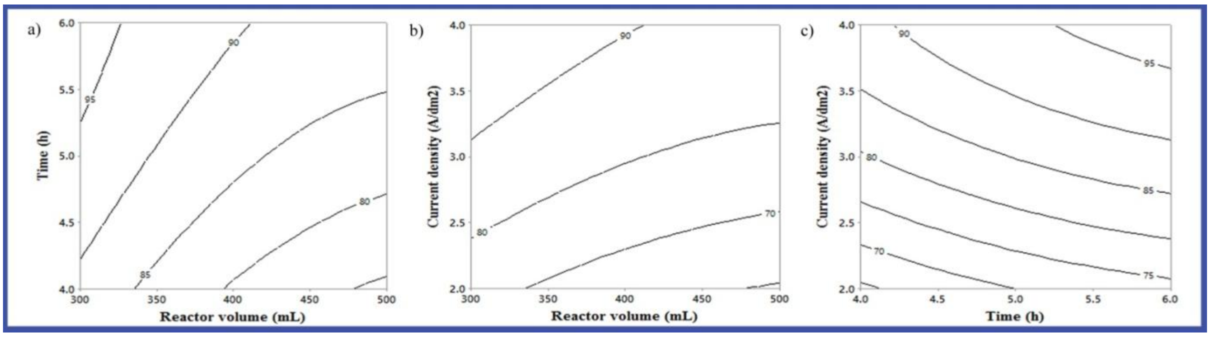

Fig. 4.Contour Plots (a) Effect of reactor volume and electrolysis time on COD removal efficiency, Current density 4 A. dm^{-2} (b) Effect of reactor volume and current density on COD removal efficiency, Electrolysis time: 6 h (C) Effect of electrolysis time and current density on COD removal efficiency, Reactor volume 300 mL

Figure 4b illustrates the response surface assuming the reactor volume and current density as independent factors. The COD removal increased with current density and at the initial current density of 2 A.dm^{-2}, the COD removal was observed to decrease with increase in reactor volume. It means that for treating larger volumes of effluents higher current density is required as depicted by Figure 4b, which predicts that when 300 mL of effluent is treated for 6 h at a current density of 3.15 A.dm^{-2}, 90% COD removal is possible as per RSM model.

Figure 4c illustrates the response surface assuming the electrolysis time and current density as independent factors. The COD removal increased with current density as well as with electrolysis time. It is seen from Figure 4c that when 300 mL of effluent is treated at 4 A.dm^{-2} and for 6 h, the COD removal efficiency was maximum at 97.17%.

It is observed (Table-3) that with increase in current density, current efficiency decreased and power consumption increased. At higher current densities, the loss of electrical energy may be more due to heat and unwanted reactions, thereby resulting in more energy loss but with higher capacity utilization.

The influences of current density and reactor hold up volume (V_R) on the heterogeneous rate constant k_h were also studied for the batch operation. k_h was found to increase with increase in current density, which may be due to the formation of more oxidizing agents. k_h was found to decrease with increase in specific electrode surface area which may be due to incomplete reactions. In general, in spite of the poorer extent of reactions, lower specific electrode surface and higher current density are preferable for better degradation of pollutants.

The absorption behaviour of treated and untreated dye bath wash water was studied at different wavelengths and is shown in Figure 5. At the wavelength of 655nm, while the untreated effluent showed a peak indicating the presence of colour, the treated effluent showed a near zero value indicating the absence of colour. The electro oxidation had obviously removed the polluting dyes from the effluent.

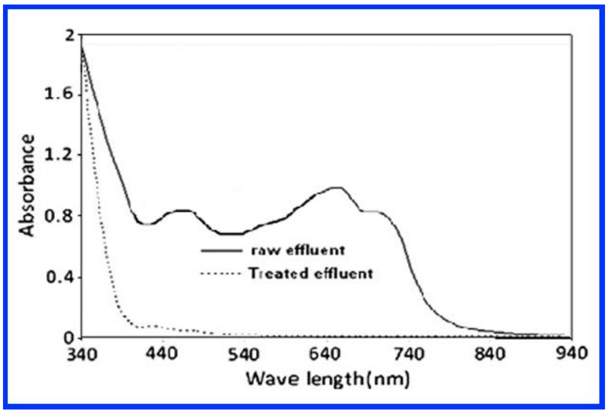

Fig. 5 Effect of electrolysis on color removal for dye-bath wastewater at the applied current density 4 A. dm^{-2}.

Table 6 Optimum conditions verification and additional experiments

Run No.	Reactor volume (ml)	Electrolysis time (h)	Current density (A/dm^2)	COD removal (%)		Error (%)	Standard deviation (%)
				Actual	Predicted		
1.	300	4	2	64.5	64.17	0.51	0.23
2.	300	7	5	98	100	-2.21	1.53
3.	400	6	3	81	80.71	0.36	0.21
4.	400	8	5	99.03	95.71	3.36	2.35
5.	500	4	2	45	44.75	0.56	0.18
6.	500	8	5	97.81	96.63	1.21	0.84

Six additional experiments were conducted to verify the actual responses with model prediction and the results are presented in Table 6. The errors and standard deviation are seen to be less, indicating that the experimental results were in good agreement with the model prediction.

5. Conclusion

Chemical oxygen demand and colour could be successfully removed/reduced from real textile industry dye bath wash water in a batch electrochemical reactor. The effects of reactor volume (X_1), electrolysis time (X_2) and current density (X_3) or COD reduction were investigated employing Box-Behnken statistical experiment design and the response surface methodology. The factors, reactor volume (X_1) and current density (X_3) were both found to have the significance level 99.1% compared to the significance level of 78.6% of electrolysis time (X_2), indicating X_1 and X_3 are more significant. The infraction effects were found to be insignificant based on ANOVA analysis. The RSM model predicted a maximum of 97.17% COD removal efficiency at the optimal conditions of reactor volume 300 mL, electrolysis time 3 h and current density 4 A.dm^{-2}. Experimental results were found to be in good agreement with the model predictions.

The investigation also indicates that in a batch electrochemical reactor more is the specific energy consumption for more COD removal. The electro oxidation process is less energy efficient and may not be a viable economic proposal if employed independently because of the low mineralization at high energy consumption. However, it can be effective in combination with a conventional technique such as biological degradation for complete mineralization of effluent. Efforts can be now made in devising a strategy in this direction.

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