Treatment Of Synthetic Wastewater Containing Rhodamine B (Rhb) Via The Electrochemical Fenton (Ecf) Process: Modeling And Optimization Using Box-Behnken Design (Bbd)

Nareshkmar R Vaghela[1*], Bhoomika Domadia[2], Ashish Modi[3]

[1,2,3]Department of Chemical Engineering, Government Engineering College, Valsad 396001, Gujarat Technological University, Gujarat, India
Email: nareshkumar.vaghela@gmail.com[1]
*Corresponding Author

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ABSTRACT
Investigations were conducted using iron plates as electrodes for the batch mode electrochemical Fenton (ECF) to treat synthetic Rhodamine B (RhB) effluent. The Box-Behnken Design (BBD) approach within the response surface methodology (RSM) was employed to develop a mathematical model and optimize process parameters for colour removal efficiency (CRE) (%) of the RhB model dye. The influence of process variables was investigated and optimised using four factors with three levels of BBD. The optimal circumstances for colour removal were pH 3.0, a current density of 97.76 A/m^2, an RhB concentration of 52.33 ppm, and an electrolysis time of 9.83 minutes. A maximum CRE of 99.57% and a desirability of 0.99 were attained in these conditions. Three replicate experiments were conducted to validate these optimum conditions, resulting in an average CRE of 98.77%, which closely matched the predictions of the regression model.

Keywords: Electrochemical Fenton, Optimization, Response surface methodology, Rhodamine B, Wastewater

INTRODUCTION

Environmental contamination, particularly wastewater, is receiving attention in developed and developing nations (Jasni et al. 2020). To fulfill discharge standards, there has been an increasing interest in developing novel technologies and approaches (Varank et al. 2018).

Many techniques, such as activated carbon adsorption, ozonation, chemical coagulation, membrane filtration, photodegradation, Fenton processes, and electrochemical oxidation processes, are used in the advanced treatment of industrial wastewater (Panizza et al. 2007; Alhamedi et al. 2009; Şengil and Özacar 2009; Puttappa and Venkatarangaiah 2011; Kabdaşlı et al. 2012; Bhatia et al. 2017). However, these methods can be expensive, energy-intensive, or may result in generating of secondary pollutants, posing challenges for their widespread implementation. Consequently, there is a growing need for sustainable and lucrative treatment methods that can effectually eliminate recalcitrant pollutants like Rhodamine B from industrial effluents.

Among these, using electrochemical techniques can be a worthwhile choice because it has several benefits attached to it (Andreozzi et al. 1999; Hsing et al. 2007; Muff 2010). Electrochemical technologies have attracted a lot of attention in the last 20 years since they promise to be widely distributed and require minimum amounts and numbers of chemicals (Guvenc et al. 2017).

Furthermore, electrochemical methods are particularly appealing due to their inherent advantages. They offer easy applicability, often operating as straightforward setups with electrode systems. Additionally, they minimize chemical consumption, relying primarily on electricity to drive reactions and utilizing electrons as the primary reagent for pollutant degradation. This enhances their environmental sustainability and is potentially more cost-effective compared to conventional methods (Darvishmotevalli et al. 2019).

The proper disposal of rinse water is a noteworthy obstacle to its efficient recovery from large commercial dyeing processes (Vaghela and Nath 2020). Because of its carcinogenic and teratogenic properties, RhB-contaminated wastewater must be restored in order to reduce its negative impacts on the ecosystem (Vaghela 2024). The electrochemical Fenton (ECF) procedure is the utmost effective alternative option. The electrochemical method may be intriguing since it runs easily and economically

without the use of chemical reagents (Vaghela and Nath 2019) and their strong efficaciousness in eliminating pollutants (Guvenc et al. 2017).
The ECF process's guiding principles were established at the beginning of 2000 by the Oturan and Brillas groups (Oturan 2000). The ECF method uses hydrogen peroxide (H_2O_2) and Fe^{2+} to trigger Fenton's reaction, resulting in OH (hydroxyl radicals) (Neyens and Baeyens 2003; Nidheesh and Gandhimathi 2012; Barhoumi et al. 2017; Özcan and Özcan 2018). Equation 1 depicts the anodic oxidation process, and Equation 2 shows the cathode reaction.

Anode:	$Fe_{(s)} \rightarrow Fe^{2+}{}_{(aq)} + 2e^{-}$	1
Cathode:	$Fe^{3+}{}_{(aq)} + e^{-} \rightarrow Fe^{2+}{}_{(aq)}$	2
In aqueous solution:	$Fe^{2+}{}_{(aq)} + H_2O_{2(l)} \rightarrow Fe^{3+}{}_{(aq)} + {}^{\bullet}OH + OH^{-}{}_{(aq)}$	3
	$Fe^{3+}{}_{(aq)} + H_2O_{2(l)} \rightarrow Fe^{2+}{}_{(aq)} + H^{+}{}_{(aq)} + H^{\circ}_{2(g)}$	4

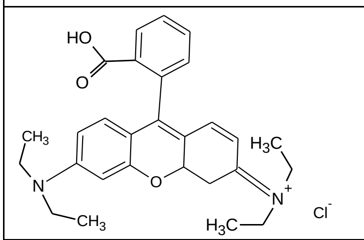
In the typical Fenton reaction, H_2O_2 is combined with a soluble ferrous iron salt, known as "Fenton's reagent," to produce hydroxyl radicals (OH). Depending on the configuration, the H_2O_2 and Fe^{2+} ions can be generated electrically either within or outside the system. H.J.H. Fenton reported in the late 19th century that the presence of ferrous iron ions enhances the oxidative strength of H_2O_2.(Fenton 1894) Equation 3 increases the Fenton reaction efficiency by electro-regenerating ferrous ions from ferric ions. Equation 4 illustrates the ECF process's overall response mechanism.
The traditional optimization method of altering one variable at a time is both time-consuming and costly when investigating the influence of different factors on a response. Response Surface Methodology (RSM) offers a more efficient approach by employing a set of empirical and mathematical techniques to assess the relationships between multiple parameters and optimize processes. This method considerably decreases both the total cost and the number of experimental trials needed. The Box-Behnken Design (BBD) is a popular design within RSM, and is particularly effective and versatile, providing comprehensive information on the effects of variables and the overall experimental error with a minimal number of experiments (Thirugnanasambandham and Sivakumar 2015).
In the context of dye wastewater treatment by the ECF method, the current study aims to develop a mathematical model to optimise operating conditions on colour and evaluate the impacts and interactions of parameters, including starting pH, current density (CD), RhB content, and time. By examining the interaction effects of several process factors on colour removal and creating a mathematical model using BBD, the RSM technique was utilized to improve the ECF process (Virkutyte et al. 2010; Cruz-González et al. 2012).

MATERIALS AND METHODS

Chemicals

Rhodamine B (RhB) was supplied by the Kohinoor Group of Industries, Ankleshwar, India. The primary characteristics of both dyes are detailed in Table 1. Commercially available iron plates, measuring 150×50×2 mm, were utilized as electrodes.

Table 1. Dye Characteristics used for the research.

Chemical structure	**Colour index name:** Basic Rhodamine B
	$\lambda_{max:}$ (nm): 554
	Molecular formula: $C_{28}H_{31}ClN_2O_3$
	Molecular weight (g/mol): 479.017

For synthesis and testing, deionized water (DI) with a conductivity of 1 µS/cm was used in all the investigations. We bought H_2O_2, NaCl, and HCl of analytical grade from M/s Merck, India.

Experimental setup and procedure

Using about 250 mL of RhB dye solution, each batch experiment was carried out in a 250 mL cylindrical glass beaker (Figure 1). Uniform mixing was accomplished by a magnetic stirrer operating continuously at 200 rpm. An effective electrode area of 25 cm^2 was achieved by using two iron plates, each sized 150×50×2 mm, as the anode and cathode in a monopolar configuration with a 20 mm interelectrode gap. The synthetic wastewater's pH was raised or lowered using 0.1 M NaOH or 0.1 M HCl. The solution was mixed with a preset quantity of NaCl before direct current (DC) from a DC power source (SIGMA, 0-30 V, 0-5 A) was applied. The target current density (A/m^2) was then attained by adjusting the voltage of the DC power source. Samples were taken at various intervals, and the CRE (%) was calculated after the Whatman filter paper filtration of the samples to remove particulate matter. After each run, the electrodes were cleaned with a 0.1 M HCl solution to remove any residual impurities and the oxide layer, then dried and reused.

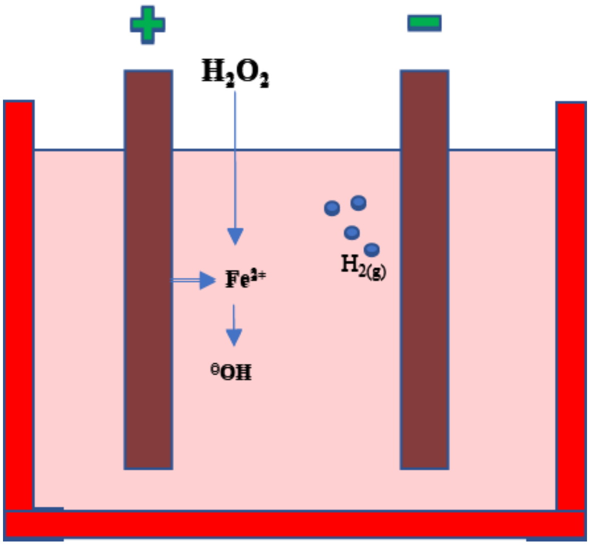


Figure 1. Electrochemical Fenton process. (Vaghela 2021)

Colour Removal Efficiency CRE (%)

The concentration of dye was determined using a UV-Vis spectrophotometer. (Systronics, model CL 335). The RhB dye concentration in the unidentified solution was ascertained using the calibration curve. Equation 5 (Vaghela and Nath 2022) was utilized to ascertain the sample's colour removal efficiency (CRE) (%) throughout various periods:

$$\text{CRE }(\%) = \left(1 - \frac{A}{A_0}\right) \times 100 \quad (5)$$

where A_0 and A represent the light absorbance of a sample taken before and following the experiment, respectively, as ascertained by a UV/VIS spectrophotometer.

Experimental design

RSM is an effective approach for process optimization when the outcome is influenced by various independent factors and their interactions. RSM enables the simultaneous assessment of multiple variables with a limited number of experiments because of its distinctive design of experiments (DOE) (Cruz-González et al. 2012).

The BBD, an experimental approach within RSM, was applied to reclaim synthetic RhB dye wastewater using an ECF method. The study was carried out using Minitab 17, which provided features such as optimization, graphical and statistical analysis, and regression. The four process variables were initial pH (X_1), current density (X_2), RhB concentration (X_3), and electrolysis time (X_4). Each variable was tested at three levels, as depicted in Table 2. A total of 27 experiments were executed as per the DOE, and the CRE (%) (Y) was calculated.

Table 2. According to BBD, the experiment's range and independent variable levels.

Variables		**Levels**	
	-1	**0**	**+1**
Initial pH: X_1	3.0	7.0	11.0
CD (A/m^2): X_2	25	75	125
RhB Conc. (ppm): X_3	50	100	150
Time (min): X_4	5	15	25

The transformation of variable levels from coded (X) to uncoded might be obtained as follows: $X_1 = 4X + 7.0$, $X_2 = 50X + 75$, $X_3 = 50X + 100$, and $X_4 = 10X + 15$.

A second-order polynomial model was employed to fit the experimental data, and regression coefficients were calculated. Equation 6 generated an expanded second-order polynomial model, which was employed for the RSM (Bashir et al. 2015).

$$Y = b_0 + \sum_{j=1}^{k} b_j X_j + \sum_{j=1}^{k} b_{jj} X_{jj}^2 \sum_{i} \sum_{<j=2}^{k} b_{ij} X_i X_j + e \qquad 6$$

Here, Y represents the chosen response of the process. At the same time, X_i and X_j are the independent process variables ranging from 1 to k (with k being the total number of independent variables). The regression coefficients are denoted as b_0, b_j, b_{jj}, and b_{ij}, where b_0 is the intercept, b_j represents the main effects, and b_{jj} and b_{ij} indicate the quadratic effects and interactions, respectively. The term e accounts for the experimental error.

RESULT AND DISCUSSION

Statistical Analysis and Modeling of the BBD

Table 3 summarizes the proposed experimental runs designed using the built experimental model. After completing the studies, each trial's CRE (%) was calculated. Table 4 presents the significance of the quadratic model. ANOVA data were used to assess the significant impact of process parameters on the CRE (%).

The f-value and P-value are depicted in Table 4 with their significant model terms for CRE (%). The P-value is computed to assess each coefficient's statistical significance and elucidate the interaction pattern among the variables under investigation. A P-value < 0.05 indicates that the corresponding coefficient is statistically significant, indicating that less than 5% of the time such a result would occur by random chance alone (Garg and Prasad 2016).

The quadratic regression model exhibited strong significance (Table 4), as indicated by a Fisher's "F" value of 266.54 and an associated P-value (<0.0001) is extremely low indicating the model is statistically significant. This result indicates that merely 0.01% of the model's variance is due to random noise (Virkutyte et al. 2010; Poonia and Gaur 2024).

The model's statistical significance was supported by the coefficient of determination (R^2). The model accurately predicted with an R^2 value of 98.06%, leaving less than 1% of the variation unexplained. The regression coefficients confirmed the model that was created (Tezcan et al. 2014). The adjusted R^2 value of 99.19% for the proposed regression model corresponds to the correlation coefficient (R^2) value of 99.56%. It demonstrates how closely the experimental and statistically anticipated values correspond (Darvishmotevalli et al. 2019; Samarghandi et al. 2020).

Table 3. BBD Matrix and CRE (%) for the Process.

Experimental Run	**Parameters (Coded Value)**				**CRE (%): Y**	
	pH (X_1)	**CD (A/m^2) (X_2)**	**RhB Conc. (ppm) (X_3)**	**Time (min) (X_4)**	**Expt.**	**RSM model**
1	-1	0	0	-1	70.12	71.89
2	-1	+1	0	0	98.1	99.45
3	0	0	+1	+1	61.89	62.56
4	0	-1	0	-1	21.34	20.48
5	-1	0	0	+1	89.99	88.73
6	-1	0	+1	0	75.09	73.44
7	0	+1	+1	0	73.08	74.15
8	0	0	0	0	56.55	56.95
9	0	+1	0	+1	87.12	85.60
10	0	0	-1	+1	74.15	75.22
11	+1	0	0	-1	9.88	11.40
12	0	-1	+1	0	23.43	25.61
13	0	+1	-1	0	85.33	82.43
14	0	0	-1	-1	54.5	54.25
15	+1	-1	0	0	13.05	11.14
16	0	0	0	0	57.03	56.95

17	+1	0	-1	0	40.00	41.95
18	-1	0	-1	0	97.7	99.62
19	0	+1	0	-1	60.06	58.53
20	0	-1	-1	0	56.65	54.86
21	0	0	+1	-1	30.02	29.37
22	0	-1	0	+1	45.00	47.55
23	+1	0	+1	0	32.21	30.59
24	-1	-1	0	0	71.12	68.99
25	+1	0	0	+1	50.22	48.71
26	0	0	0	0	55.31	56.95
27	+1	+1	0	0	55.22	56.79

The term "lack of fit" refers to the discrepancy between observed and predicted values, as well as the recurrence of errors in the observed values. Even when a model is not well-fitted, lack of fit can still hold significance. In other words, the model's performance can be reliably assessed by examining the significance of the lack of fit (Hasani et al. 2020). The developed regression model's lack of fit value is not statistically significant, indicating that the model well fits the experimental data (Singh et al. 2010).
For a suitable proposed model, the coefficient of variation (CV), a parameter indicating the range of probable data variances, should ideally not exceed 10%. However, Table 4 reveals that the study's CV value is 3.88%, indicating a commendable level of accuracy.

Table 4. ANOVA outcomes for the quadratic model of CRE (%) employing the ECF process.

Source	Df	F- Value	P- Value	Status	Statistical Indicators
Model	12	266.54	0.000	**Significant**	R^2= 99.56% Adj R^2= 99.19% Pred R^2= 98.06% CV = 3.88%
Linear	4	770.87	0.000		
X_1	1	1539.41	0.000		
X_2	1	882.58	0.000		
X_3	1	214.69	0.000		
X_4	1	446.79	0.000		
Square	3	13.63	0.000		
X_1X_1	1	5.59	0.033		
X_3X_3	1	6.5	0.023		
X_4X_4	1	18.64	0.001		
2-Way Interaction	5	14.82	0.000		
X_1X_2	1	11.72	0.004		
X_1X_3	1	11.16	0.005		
X_1X_4	1	21.28	0.000		
X_2X_3	1	22.33	0.000		
X_3X_4	1	7.58	0.016		
Error	14				
Lack-of-Fit	12	7.12	0.13	**Not Significant**	
Pure Error	2				
Total	26				

Equation 7 shows how CRE (%) relates mathematically to operating variables including initial pH (X_1), current density (X_2), the concentration of RhB (X_3), and electrolysis time (X_4). A synergistic effect on the decolourization of RhB dye is shown by the positive sign of the coefficients in the equation. In contrast, an antagonistic effect is indicated by the negative sign (Basturk et al. 2021).

$$\text{Colour removal efficiency (\%)} = 139.11 - 13.35\ X_1 + 0.0379\ X_2 - 0.7511\ X_3 + 1.020\ X_4 + 0.1338\ X_1X_1 + 0.000924\ X_3X_3 - 0.03910\ X_4X_4 + 0.01899\ X_1X_2 + 0.01853\ X_1X_3 + 0.1279\ X_1X_4 + 0.002097\ X_2X_3 + 0.00611\ X_3X_4 \quad (7)$$

In a regression equation, a parameter's coefficient indicates how important it is. (Hasani et al. 2020) The form equation shows that the most important factor influencing the CRE (%) is pH.
It is essential to ascertain whether the selected model sufficiently approximates the real system. The adequacy of the model can be evaluated using the parity plot and the normal probability plot. Figure 2 also shows a parity plot of the experimental and predicted CRE (%) values. The model's statistical significance for the selected dye removal is validated by its high R^2 value of 99.93%.
Furthermore, normal % probability plots were used to assess model suitability and determine the relationship between predicted and experimental values (Thirugnanasambandham et al. 2014). There is a strong fit between the experimental and predicted data, as seen by the data points' proximity to the diagonal line (Figure 3 (a)). Thus, the ECF procedure for RhB dye decolourization can be represented by a quadratic model.
The model's reliability is assessed through the residual versus fitted value plot (Singh et al. 2017). Figure 3 (b) illustrates the residuals plotted against the expected responses, indicating that most residuals fall within the ±3% range. Any observations outside this range suggest an operational error in the experimental data or a potential error in the model (Nayak and Vyas 2019).

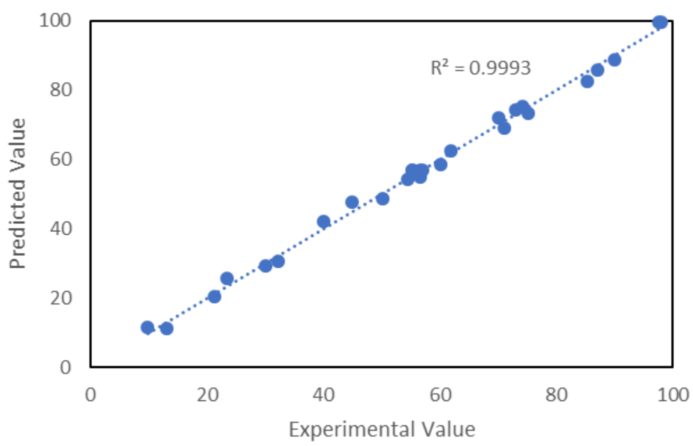


Figure 2. Predicted versus Experimental value for ECF process.

The histogram in Figure 3 (c) shows the frequency of residuals against their values, indicating that most residuals are close to zero, signifying minimal deviation from the experimental data. Figure 3 (d) presents the residuals concerning their corresponding data points, demonstrating that the residuals fluctuate unpredictably around the centerline.

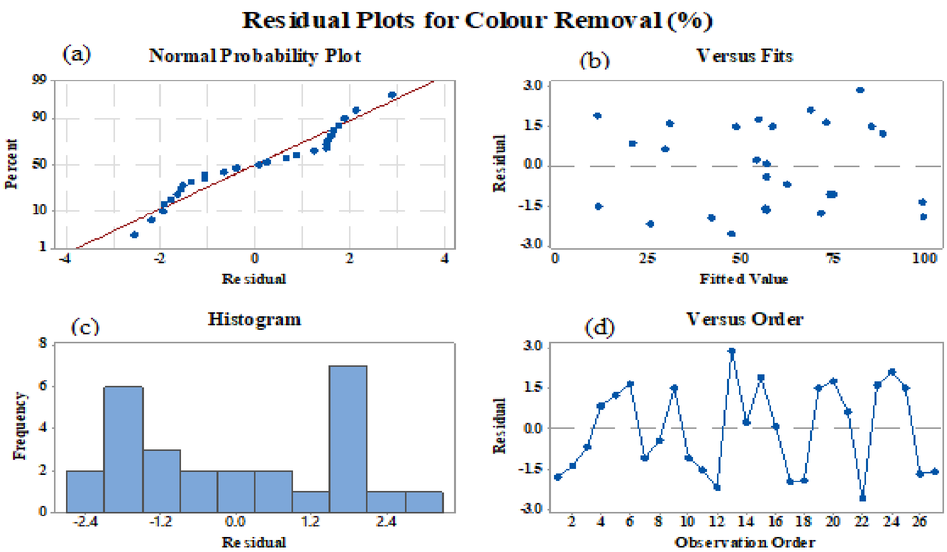


Figure 3. Residual plots for CRE (%) of ECF process (a) Normal probability plot (b) Residual versus fitted values (c) Histogram plot (d) Residual versus observation order.

Effect of Operational Parameters

Based on the established mathematical models, three-dimensional response surface plots and contour plots were created to investigate the interactive effects of the independent variables on the outcomes presented in Figures 4 and 5 (Thirugnanasambandham et al. 2014). The surface and contour plots were created with two targeted variables, while the coded values of the other two variables were held constant at zero (Nayak and Vyas 2019).

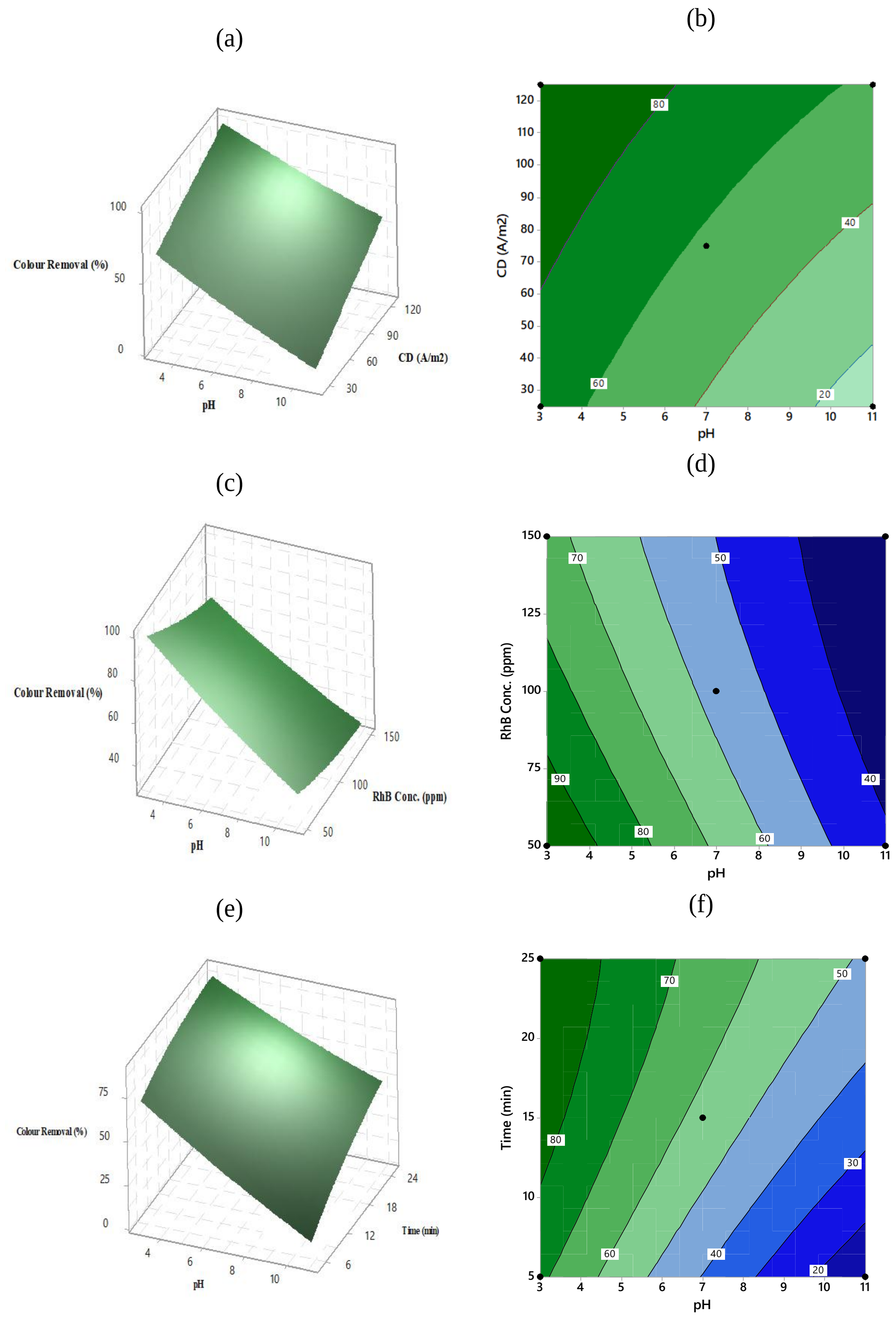


Figure 4. Surface plot and contour plot of the interaction of pH and CD (a,b), RhB Concentration (c,d), time (e,f)

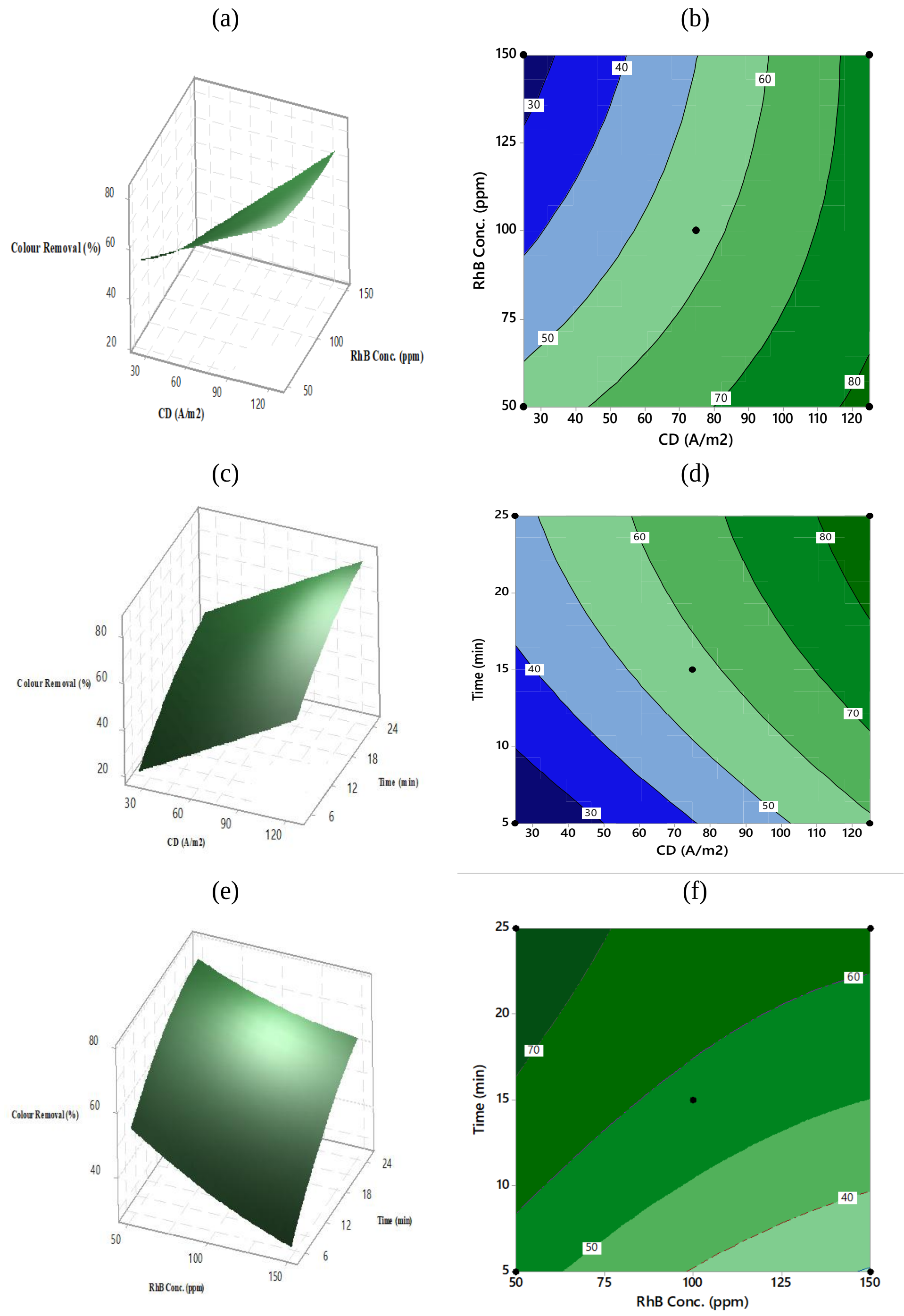


Figure 5. Surface plot and contour plot of the interaction of CD and RhB Concentration (a,b), time (c,d), the interaction of RhB Concentration and time (e,f)

To examine the impact of initial pH on the current ECF process, experiments were conducted at different initial pH levels (3-11). The results are presented in Figure 4. Figure 4 illustrates the surface and contour plots depicting the interactions between pH (X_1), and RhB concentration (X_3), time (X_4), CD (X_2). These plots demonstrate that the initial pH of the RhB dye is an important process parameter.
Figures 4(a) and (b) display surface and contour plots to examine the effects of pH and CD on colour removal effectiveness (%) while maintaining the same values for the other two parameters at a concentration of 100 ppm RhB and an electrolysis period of 15 minutes.
It is observed that the highest colour removal was obtained in acidic conditions. The soluble percentage of ferrous ions is larger, leading to the breakdown of H_2O_2 and the formation of $\bullet OH$ in acidic conditions

(Hasani et al. 2020). The anodic reaction generates more ferrous ions, increasing the concentration of •OH radicals and improving CRE (%) with CD at pH 3.0 (Cruz-González et al. 2012).

Figures 4(c) and (d) show that as the pH of the solution increased, the colour removal effectiveness (%) decreased, and an acidic pH resulted in a larger degree of dye decolourization. More than 90% declourization efficiency was accomplished at pH 3.0, low RhB concentrations (less than 75 ppm), 75 A/m^2 CD, and a 15 minute electrolysis time. Higher RhB concentrations at pH 3.0 require more •OH at constant CD for the same length of electrolysis time, resulting in a decrease in colour removal effectiveness (%).

Figure 4 (e) and (f) show how pH and electrolysis time interact. One may argue that pollutant removal increases with processing time, as Hasani et al. (Hasani et al. 2020) noted. More ferrous ions are created during longer electrolysis times, and finally, more •OH is produced as a result of the interaction between hydrogen peroxide and ferrous ions, increasing the removal efficiency.

The interaction between Rhodamine B (RhB) dye concentration and current density (CD) on dye decolourization is illustrated through the response surface plot depicted in Figure 5(a) and the corresponding contour plot represented in Figure 5(b), focusing on conditions of pH 3.0 and an electrolysis duration of 15 minutes. At lower concentrations of Rhodamine B (RhB) dye, there is a modest increase in CRE (%), whereas at the highest concentration, the efficiency improves significantly and more rapidly (Cruz-González et al. 2012).

At a pH of 3.0 and a 15 minute electrolysis time, the highest removal efficiency was achieved with a CD of 120 A/m^2 and a RhB dye concentration of 50 ppm. Figures 5(c) and (d) demonstrate that simultaneous increases in both CD and electrolysis duration lead to enhanced CRE (%). Figures 5 (e) and (f) illustrate the correlation between Rhodamine B (RhB) dye concentration and electrolysis duration. In overextended electrolysis periods, increasing the dye concentration did not significantly impact decolurization, likely due to the greater amount of •OH available in the solution.

Optimization of the ECF process

The present investigation focused on optimizing the operational parameters, specifically initial pH (X_1), current density (X_2), Rhodamine B (RhB) concentration (X_3), and electrolysis duration (X_4), to ascertain the optimal CRE (%). Hence, the numerical optimization of the operational parameters was executed using the Box-Behnken Design (BBD) model methodology. Under such ideal circumstances, the maximum percentage of colour removal (99.57 %) with 0.99 desirabilities was attained: RhB concentration of 52.33 ppm, pH 3.0, 97.76 A/m^2 CD, and 9.83 min electrolysis time (Guvenc et al. 2017). Following the identification of the ideal conditions, three runs of the actual tests were conducted. The results demonstrated an actual CRE of 98.77%, which is closely aligned with the predictions of the regression model.

CONCLUSION

In this study, BBD was used to investigate and optimise the effect of process variables such as pH, Current Density (A/m^2), Rhodamine B dye concentration (ppm), and electrolysis time (min) on CRE (%) from synthetic Rhodamine B (RhB) wastewater using an electrochemical Fenton (ECF) treatment process with iron electrodes. The experimental design employed the BBD, a statistical approach that helps in understanding the interactions between multiple variables and optimizing the response. Through numerical optimization using the BBD model, the study found that the maximum CRE of 99.57% was achievable with a desirability factor of 0.99 under the following conditions: pH 3.0, current density 97.76 A/m^2, RhB concentration 52.33 ppm, and electrolysis time 9.83 minutes. In order to verify these results, triplicate experiments were carried out. under the identified optimal conditions. The test results demonstrated strong agreement with the regression model. This high level of agreement confirms the robustness and accuracy of the BBD model in optimizing the ECF process for dye wastewater treatment.

CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest.

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