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# Optimization of Brilliant Green Dye Removal Efficiency by Electrocoagulation Using Response Surface Methodology

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**Abstract** The response surface methodology has been used to determine the optimum conditions for the Brilliant Green dye removal efficiency from aqueous solution by electrocoagulation. The experimental parameters which have been investigated were initial dye concentration: 100–500 mg/L; voltage: 4-12V; NaCl Concentration:0.5-1.5g/l and reaction time: 10–30min. These parameters were changed at three levels according to the Box Behnken Design to evaluate their effects on decolorization through analysis of variance. High $R^2$ value of 96.16% shows a high correlation between the experimental and predicted values and expresses that the second-order regression model is acceptable for Brilliant Green dye removal efficiency. Optimum dye removal efficiency of 99.0% was observed experimentally at NaCl concentration of 0.5008g/l, initial dye concentration of 500 mg/L, applied voltage of 4.0065V and reaction time of 12.22 min, which is close to model predicted (98.9997%) result.

***Keywords:*** *brilliant green dye, decolorization, electrocoagulation, response surface methodology, box behnken design*

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## 1. Introduction

Wastewater from industries contains several harmful chemicals that are toxic to biological life. Textile industry is one of the major contributors of Wastewater. Thus wastewater from textile industry has to be treated for carcinogenic, mutagenic, teratogenic, coloring agents prior to disposal [1]. Several techniques are available for treatment of wastewater such as adsorption, chemical coagulation, electrocoagulation, membrane separation, biological methods such as fungal decolourization and microbial decolourization. But some of these techniques are not techno-economic. Adsorption is a competitive technique because higher removal efficiency is obtained but problem is associated with the cost of adsorbent and its regeneration. In chemical coagulation, though the dyes are almost completely removed but it's quite expensive. The disposal of concentrated sludge is a problem and use of chemicals lead to secondary pollution problem. In membrane separation cost is a big issue that's why it is not practiced at industrial level. The only problem associated with biological methods is the requirement of large area but it has advantage of low cost over other techniques [2,3,4].

Brilliant green is a very well-known cationic dye used for various purposes like biological stain, dermatological agent, veterinary medicine, an additive to poultry feed to inhibit propagation of mold, intestinal parasites and fungus. It is also used extensively in textile dying and paper printing [5]. The main reason Brilliant Green dye is used because its widely used in industries for dying of wool and silk fibres. [6] The chemical structure of the selected dye is illustrated in Figure 1.

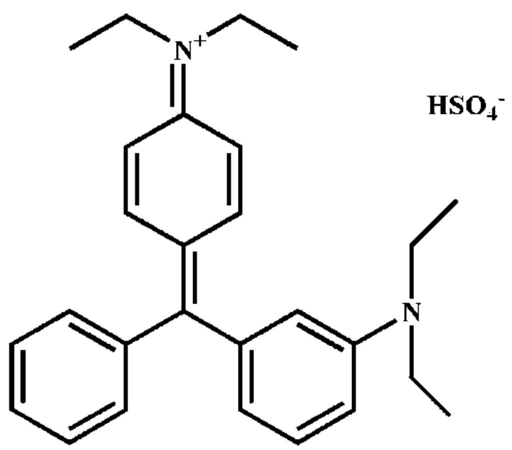


**Figure 1.** Structure of Brilliant Green Dye

Electrocoagulation (EC) is a promising technique for removal of dye because it employs the low cost, simple and easily available equipment. Also the operating cost of a process can be justifiably low. Furthermore the amount of sludge produced by this technique is less and it can be dewatered easily by the use of commonly available methods [7,8]. The dye molecule after coagulation can be effectively removed by the emerging membrane technology to achieve the goal of process intensification.

In this process, generation of coagulants (iron or aluminium ions) by electro dissolution of the sacrificial anode(s) leads to formation of particles that entrap the pollutants. The main reactions that followed using iron as electrodes are:

At Anodes:

$$Fe(s) \rightarrow Fe^{+3} + 3e^{-} \quad (1)$$

At the cathode:

$$3H2O + 3e^{-} \rightarrow 3/2H_2 + 3OH^{-} \quad (2)$$

In the solution:

$$Fe(s) + 3H_2O \rightarrow Fe(OH)_3 + 3H^{-} \quad (3)$$

Response surface methodology (RSM) is a collection of mathematical and statistical techniques for modelling and analysis of problems in which a response of interest is influenced by a set of independent variables [9,10]. Main advantages of optimization by RSM over conventional methods are reduction of experimental trials in providing sufficient information for statistically valid results and evaluation of the relative significance of parameters and their interactions [10,11]. In recent years, the area of optimization dye removal efficiency by electrocoagulation has received enormous attentions. However, according to our knowledge, application of RSM design in decolorization by EC is rarely presented in scientific papers. On the other hand, as of now there is no research available on decolorization on brilliant green dye using response surface methodology except by biological procedures.

The aim of the present study is to optimize Brilliant Green dye removal from aqueous solution by electrocoagulation process using RSM. For this purpose, Box behnken design was used to develop a mathematical correlation between Brilliant Green dye removal efficiency and four selected independent parameters including NaCl concentration, initial dye concentration, applied voltage and reaction time.

## 2. Materials and Methods

Synthetic Dye Wastewater was prepared by dissolving Brilliant Green Dye which was provided by Merck Company. All the runs were performed at room temperature. In each run, 500ml of the dye solution was taken into the electrolytic cell sodium chloride (NaCl) was used to increase the conductivity of the solutions and samples were collected during the run at an interval of five minutes for analysis. The total effective electrode area was 72.8cm$^2$ and the spacing between the electrodes was 10mm. The voltage was adjusted to a desired value and the coagulation was started. At the end of electrocoagulation, the solution was filtered and then analyzed. Before each run, electrodes were washed with acetone to remove grease from the surface and the impurities on the iron electrode surfaces were removed by dipping the electrodes for two minutes in a solution freshly prepared by mixing 100ml HCl solution (35%) and 200ml of hexamethylenetetramine aqueous solution (3%).The samples were filtered and analyzed in UV-IV Spectrophotometer.

For optimization of Brilliant Green dye removal efficiency using Box behnken Design, 27 experiments consisting of 4 continuous factors and 3 replicates at the centre point were designed. Levels of selected parameters are shown in Table 1. As presented in Table 1, each independent variable was coded in 3 levels (-1, 0, and 1) as $x_i$ as according to equation:

$$x_i = (X_i - X_o)/\Delta X \quad (4)$$

where $X_o$ is value of the $X_i$ (selected parameters) at the centre point and ΔX presents the step change Brilliant Green removal efficiency was taken as the response of the experiments according Equation:

$$Y_i = b_o + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} b_{ii} x_i^2 + \sum_{i=1}^{n} \sum_{j=i+1}^{n} b_{ij} x_i x_j \quad (5)$$

Where

$Y_i$ is the percentage of dye removal efficiency

$b_o$=the constant coefficient

$b_i$= regression coefficients for linear effects

$b_{ii}$=the quadratic coefficients

$b_{ij}$=the interaction coefficients

and $x_i$ , $x_j$ are the coded values of parameters.

The statistical software “Minitab”, version 17.0 was used for the regression and graphical analyses of the experimental data obtained. The accuracy of the fitted model was justified through analysis of variance (ANOVA) and the coefficient of R$^2$.

**Table 1. Experimental range and levels of independent parameters**

| Parameters | X | Levels | | |
|---|---|---|---|---|
| | | -1 | 0 | 1 |
| Voltage | $X_1$ | 4V | 8V | 12V |
| Time | $X_2$ | 10min | 20min | 30 min |
| NaCl conc. | $X_3$ | 0.5g/l | 1.0g/l | 1.5g/l |
| Dye conc. | $X_4$ | 100mg/l | 300mg/l | 500mg/l |

## 3. Observations

The design matrix with experimental and predicted Brilliant Green removal efficiencies are listed in Table 2.

The final model is expressed by:

$$\begin{aligned} &\text{Removal Efficiency} \\ &= 88.54 + 1.804\text{Voltage} + 0.094\text{Time} \\ &- 5.97\text{NaCl} + 0.02465\text{Dye} \\ &0.0321\text{Voltage*Voltage} - 0.00403\text{Time*Time} \\ &+ 2.995\text{NaCl*NaCl} - 0.000020\text{Dye*Dye} \\ &- 0.04112\text{Voltage*Time} + 0.407\text{Voltage*NaCl} \\ &- 0.002653\text{Voltage*Dye} - 0.0332\text{Time*NaCl} \\ &+ 0.001223\text{Time*Dye} - 0.01049\text{NaCl*Dye}. \end{aligned} \quad (6)$$

**Table 2. Experimental and Predicted values using Box Behnken Design**

| RUN | Applied Voltage($X_1$) | Time($X_2$) | NaCl conc. ($X_3$) | Initial Dye conc. ($X_4$) | Dye Removal Efficiency | |
|---|---|---|---|---|---|---|
| | | | | | Experimental | Predicted |
| 1 | 0 | 0 | -1 | -1 | 95.3% | 95.7311% |
| 2 | 0 | 0 | 0 | 0 | 97.00% | 97.633% |
| 3 | 0 | -1 | 0 | 1 | 95.8% | 95.6779% |
| 4 | -1 | 0 | 1 | 0 | 96.087% | 96.4658% |
| 5 | 1 | 0 | 0 | -1 | 98.05% | 97.6898% |
| 6 | 0 | 1 | 1 | 0 | 97.236% | 96.9174% |
| 7 | 0 | -1 | -1 | 0 | 99% | 98.7089% |
| 8 | -1 | 0 | 0 | -1 | 92.70% | 92.8069% |
| 9 | 0 | 1 | 0 | 1 | 98.8820% | 99.3176% |
| 10 | -1 | 0 | 0 | 1 | 99.441% | 99.1914% |
| 11 | 0 | 0 | 1 | -1 | 97.485% | 97.2896% |
| 12 | 0 | 0 | 0 | 0 | 97.95% | 97.633% |
| 13 | 1 | 0 | -1 | 0 | 97.8567% | 97.6434% |
| 14 | 1 | 0 | 0 | 1 | 96.3% | 95.5833% |
| 15 | 0 | 1 | -1 | 0 | 98.2% | 97.7895% |
| 16 | 0 | 0 | -1 | 1 | 99.3292% | 99.9688% |
| 17 | -1 | 0 | -1 | 0 | 98.79% | 98.6341% |
| 18 | 0 | -1 | 1 | 0 | 98.7% | 98.5007% |
| 19 | 0 | 0 | 1 | 1 | 97.3168% | 97.3299% |
| 20 | 0 | 1 | 0 | -1 | 92% | 92.2876% |
| 21 | 1 | 1 | 0 | 0 | 94.5% | 94.7652% |
| 22 | 0 | -1 | 0 | -1 | 98.7% | 98.4299% |
| 23 | 0 | 0 | 0 | 0 | 97.950% | 97.633% |
| 24 | 1 | 0 | 1 | 0 | 98.41% | 98.7314% |
| 25 | 1 | -1 | 0 | 0 | 98.6030% | 99.3065% |
| 26 | -1 | -1 | 0 | 0 | 95.2% | 95.3791% |
| 27 | -1 | 1 | 0 | 0 | 97.677% | 97.4177% |

Estimated P values of the parameters for Brilliant Green Dye removal efficiency (%) are illustrated in the Table 3:

**Table 3. Estimated P-Values for the various parameters**

| TERMS | P-VALUE |
|---|---|
| CONSTANT | 0.000 |
| $X_1$ | 0.050 |
| $X_2$ | 0.002 |
| $X_3$ | 0.117 |
| $X_4$ | 0.000 |
| $X_1*X_1$ | 0.054 |
| $X_2*X_2$ | 0.119 |
| $X_3*X_3$ | 0.009 |
| $X_4*X_4$ | 0.006 |
| $X_1*X_2$ | 0.000 |
| $X_1*X_3$ | 0.012 |
| $X_1*X_4$ | 0.000 |
| $X_2*X_3$ | 0.560 |
| $X_2*X_4$ | 0.000 |
| $X_3*X_4$ | 0.003 |

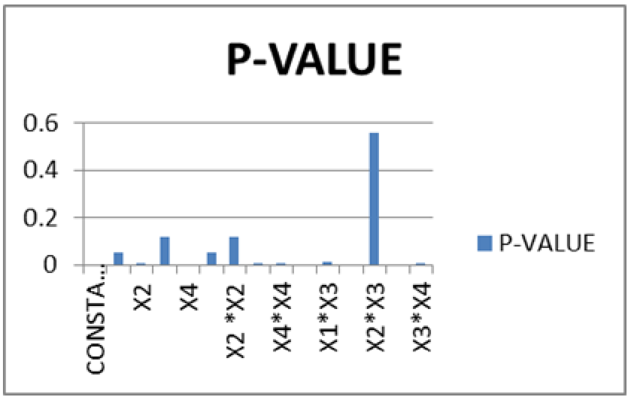


**Figure 2.** P-Values of various parameters

As depicted in the above Table 3 and Figure 2, the amounts of P ($P<0.05$) for all independent parameters confirms that the three out of four selected factors are significant i.e. NaCl concentration is insignificant whereas initial dye concentration is most significant. However, it was found that all square and interaction terms ($P< 0.05$) except $x_1*x_1$, $x_2*x_2$ and $x_2*x_3$ ($P>0.05$) were significant to the response. The analysis of variance (ANOVA) for the Brilliant Green dye removal efficiency is given in Table 4. According to this table, the P value of 0 ($P = 0.05$)

justifies the reliability of the fitted polynomial model through ANOVA with 95% confidence level. Furthermore, parity plot for the experimental and predicted value of Brilliant Green dye removal efficiency (%) is demonstrated in Figure 3. High $R^2$ value of 96.16% validates the statistical significance of the model for the selected dye removal.

**Table 4. Analysis of variance (ANOVA) for Brilliant Green dye removal efficiency (%)**

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Model | 14 | 92.2385 | 6.5885 | 21.44 | 0.00 |
| Linear | 4 | 20.5179 | 5.1295 | 16.69 | 0.00 |
| Square | 4 | 11.7847 | 2.9462 | 9.59 | 0.001 |
| 2-Way Interaction | 6 | 59.9359 | 9.9893 | 32.50 | 0.00 |
| Error | 12 | 3.6879 | 0.3073 | | |
| Lack-of-fit | 10 | 3.0862 | 0.3086 | 1.03 | 0.590 |
| Pure Error | 2 | 0.6017 | 0.3008 | | |
| Total | 26 | 95.9264 | | | |

**Table 5.**

| Term | Coefficient | T-Value | P-Value | Significance |
|---|---|---|---|---|
| Constant | 88.54 | 305.04 | 0.000 | - |
| $X_1$ | 1.804 | 1.99 | 0.050 | Significant |
| $X_2$ | 0.094 | -3.91 | 0.002 | Significant |
| $X_3$ | -5.97 | -1.69 | 0.117 | - |
| $X_4$ | 0.02465 | 6.68 | 0.000 | Significant |
| $X_1*X_1$ | -0.0321 | -2.14 | 0.054 | - |
| $X_2*X_2$ | -0.00403 | -1.68 | 0.119 | - |
| $X_3*X_3$ | 2.995 | 3.12 | 0.009 | Significant |
| $X_4*X_4$ | -0.000020 | -3.34 | 0.006 | Significant |
| $X_1*X_2$ | -0.04112 | -5.93 | 0.000 | Significant |
| $X_1*X_3$ | 0.407 | 2.94 | 0.012 | Significant |
| $X_1*X_4$ | -0.002653 | -7.66 | 0.000 | Significant |
| $X_2*X_3$ | -0.0332 | -0.60 | 0.560 | - |
| $X_2*X_4$ | 0.001223 | 8.82 | 0.000 | Significant |
| $X_3*X_4$ | -0.01049 | -3.79 | 0.003 | Significant |

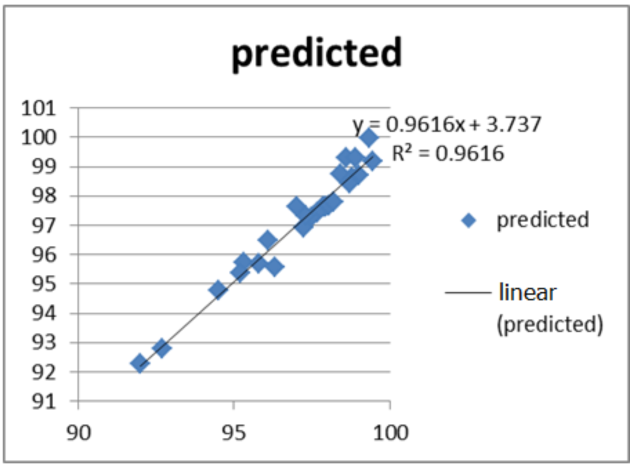


**Figure 3.** Parity Plot for the experimental v/s predicted value of Brilliant Green Removal Efficiency with $R^2$=96.16%

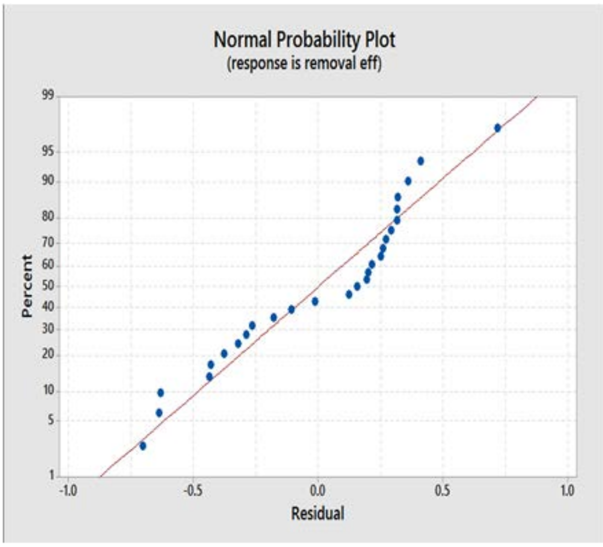


**Figure 4.** Normal Probability Plot

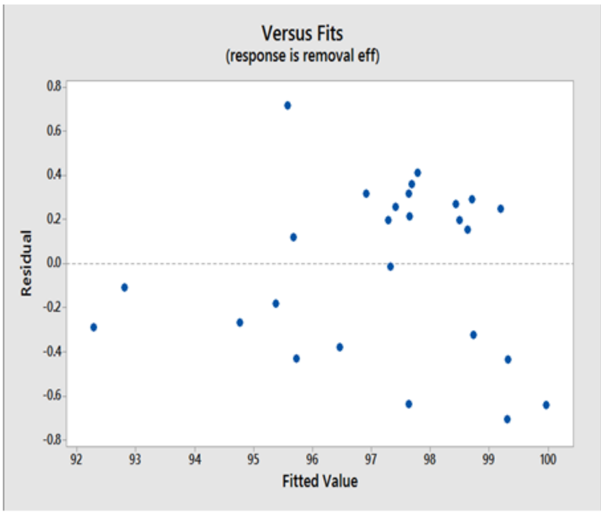


**Figure 5.** Residual v/s fitted value plot

## 4. Effect of Operating Parameters

The main effect of each parameter on the Brilliant Green dye removal efficiency have been clearly illustrated in Figure 6 given below and for more clarity 3D graphs have also been provided in Figure 7. The effect of the parameters can be explained as follows:

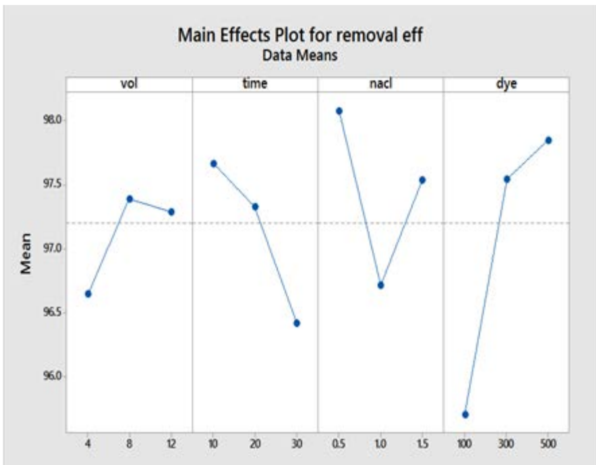


**Figure 6.** Main effects plot for Removal Efficiency of Brilliant Green Dye

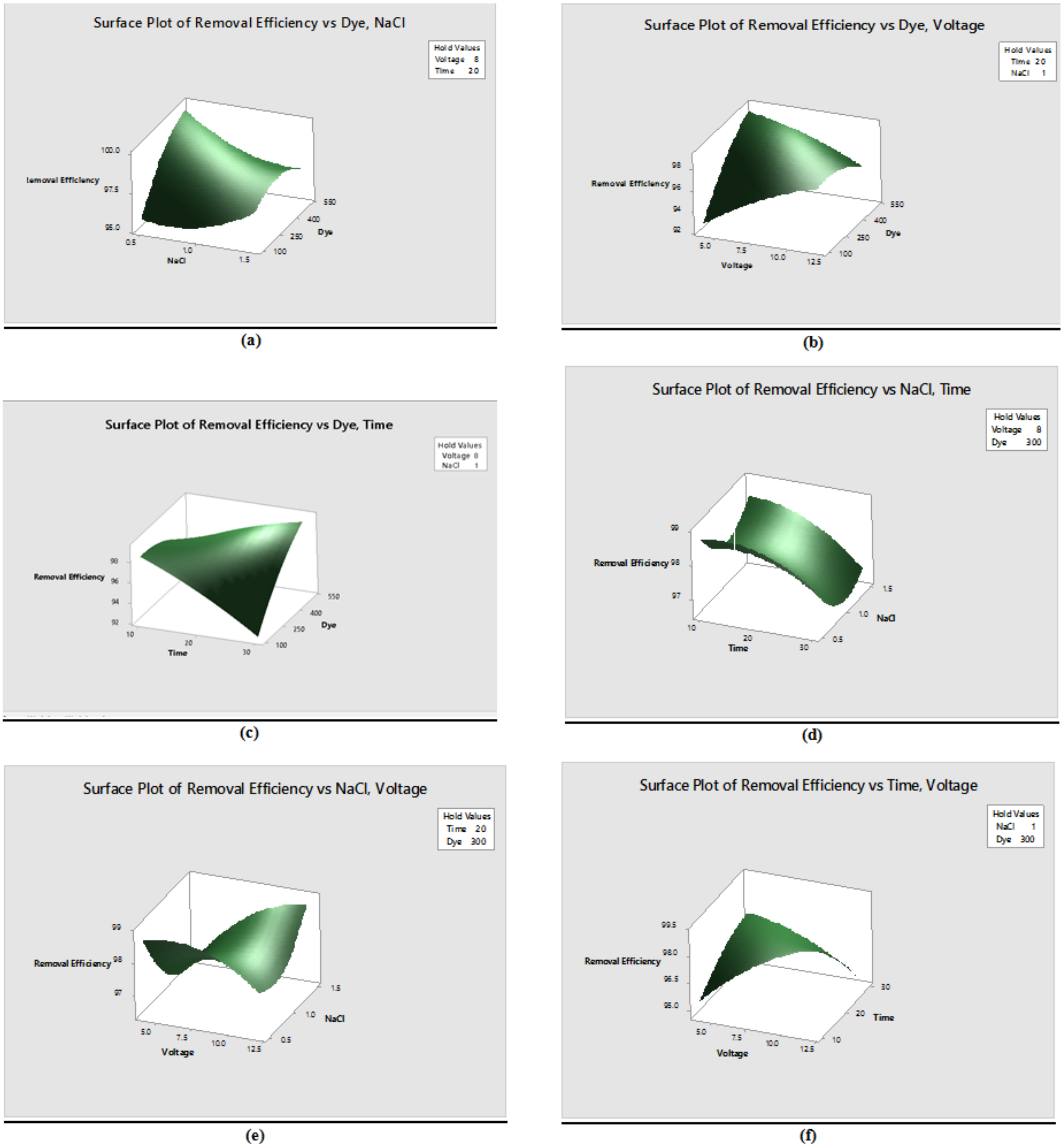


**Figure 7.** 3-D plots for Removal Efficiency of Brilliant Green Dye as a function of (a) initial dye concentration and NaCl conc. (b)initial dye concentration and voltage (c) initial dye concentration and time(d)NaCl conc. and time (e) NaCl conc. and voltage(f)time and voltage

## 4.1. Voltage

Voltage is one of the most important operating parameters in electrocoagulation process as it forms the basis of cost analysis factor. As the voltage is increased, current density increases resulting in higher removal efficiency. It can be seen from the graph below that as the voltage increases, the removal efficiency of dye initially increase approximately from96.5% to 97.5% and then when the voltage is increased from 8V to 12V the removal efficiency decreases from 97.5% to 97.3%.

## 4.2. Time

The percentage removal efficiency depends directly on the concentration of ions produced by the electrodes. The graph obtained indicates that with an increase in the electrolysis time there is a decrease in the brilliant green dye removal efficiency

## 4.3. NaCl Concentration

At certain conditions of the process, the ability of pollutant removal will be dependent on the rate of coagulant generation which is related to the conductivity of the media. Table salt (NaCl) is usually employed to increase the conductivity of the wastewater to be treated by electrocoagulation. Increase in salt concentration, increases the ion concentration in the solution and hence reduces the resistance between the electrodes. As a result, cell voltage decreases at constant current density and reduces the power consumption in electrolytic cells. Also, at higher anode potential, other reactions may occur at the anode such as direct oxidation of organic compounds or $H_2O$ [12]. To study the effect of wastewater conductivity on dye removal and specific electrical energy consumption, various experiments were performed using NaCl as the electrolyte in the range of 0.5–1.5g/L. The graph below summarizes the variations of applied voltage and percentage dye removal at constant voltage for different salt concentration. From the graph below it can be observed that on raising the conductivity of dye solutions did not have a considerable effect on dye removal efficiency but had great effect in decreasing power consumption.

### 4.4. Initial Dye Concentration

To observe the effect of initial dye concentration on the dye removal efficiency by electrocoagulation, experiments were carried out for three different dye concentrations (100, 300, and 500mg/L) for 30minutes. The graph below shows the percentage removal of dyes for different initial dye concentration. As the results indicate, the dye removal efficiency increases with an increase in initial dye concentration.

## 5. Optimization of Removal Efficiency

**Table 6. Optimum value for decolorization of Brilliant Green dye**

| S.No. | Voltage | Time | NaCl conc. | Dye Conc. | Removal Efficiency | |
|---|---|---|---|---|---|---|
| | | | | | Experimental | Predicted |
| 1 | 4.0065V | 12.22mins | 0.5008gm/l | 500ppm | 99.0% | 98.9997% |

One of the main objectives in designing the experiments using response surface methodology was in order to optimize the parameters of the electrocoagulation for maximum removal efficiency of Brilliant Green dye. In the present work, Minitab 17.0 software was utilized to determine the optimum values of the parameters from the model developed by Box Behnken Design. The optimum values of the factors along with the experimental and predicted removal efficiencies for Brilliant Green dye are given in Table 6. The experiments were carried out with the predicted optimum values in order to verify the optimized values. The results thus obtained revealed that optimum removal efficiency (99%) was achieved using the optimum values of each parameter which was in good conformity with the predicted value (98.9997%) .

## 6. Result and Discussion

The important parameters that effects the removal efficiency of Brilliant Green dye by electrocoagulation includes applied voltage, time and initial dye concentration out of which initial dye concentration is the most significant one whereas NaCl concentration is the most insignificant. The effects of each parameter are shown in the Figure 6 and Figure 7 for Brilliant Green dye. From the results obtained, it was observed that optimum applied voltage; time of electrolysis; NaCl concentration and initial dye concentration are 4.0065V, 12.22 mins, 0.5008gm/l, and 500ppm respectively.

Box Behnken Design was employed to determine the simple and combined effect of operating parameters on dye removal efficiency of Brilliant Green. The validity of the model was determined by the help of Normality plot, Residual versus fitted value plot, and Lack-of-fit test as shown in Table 5.

**Normality Plot**: Normality plot have been illustrated in the Figure 4, and normality assumption is clearly satisfied as the points in the plot form a relatively straight line [13,14].

**Residual versus Fitted value Plot:** The reliability of the model is determined by the help of residual versus fitted value plot as depicted in the Figure 5, the model is correct and assumptions made are satisfied as the residuals are structureless; in particular they are unrelated to any other variable including the predicted response i.e., the residuals scatter randomly on the graph and they do not reveal any obvious pattern which clearly indicates model adequacy[15].

**Lack-of-fit Test**: Lack of fit is determined by observing the P-value as it is shown in Table 5. As the P-value of lack-of-fit is larger than significance level (i.e. 0.05) hence it can be concluded that there is no lack of fit in the model and it's adequate [9].

As a result, Figure 4 and Figure 5 and Table 5 clearly signifies that the model is adequate to describe the Removal Efficiency of Brilliant Green dye by response surface methodology. Various other studies have been previously made on Electrocoagulation which has been shown in Table 7. Nandi et al. [12] have studied the removal efficiency of Brilliant Green from aqueous solution by electrocoagulation using aluminum electrodes (height=15cm and width=6cm) and ascertained the effect of operational parameters such as initial dye concentration, electrolysis time, current density, inter electrode distance, initial pH, and salt (NaCl) concentration. They have reported the maximum dye removal efficiency of 99.9% which was observed for current density of 416.7 $A/m^2$ after 30 minutes of operation from the dye solutions of 100 mg/L. We have obtained the similar results but compared to our study we have used iron electrodes which are superior to aluminum as sacrificial electrode material which have been studied by Kobya et al. [16] and moreover we have used response surface methodology for examining the optimum conditions and validity of our model. Sudamalla et al.[17] have also examined the optimization of Brilliant Green adsorption by adsorbent prepared from Citrus limetta peel using response surface methdology and have examined the influence of parameters like pH, temperature, initial concentration, and adsorbent dosage.They have reported maximum dye removal of 95% which is close to result obtained in the present study.

According to the best of our knowledge, up to now there is no research available on optimization of Brilliant Green Dye decolorization by Electrocoagulation using response surface methodology.

**Table 7. Studies on Dye Removal**

| S.No. | Dye Used | Method Applied | Inference | | Reference |
|---|---|---|---|---|---|
| | | | Removal Efficiency | Time | |
| 1 | Drimarene K2LR CDG Blue | Electrocoagulation | 90.7% | 105min | [18] |
| 2 | Red Dye(2 Naptholic acid + 2 Naphthol) | Electrocoagulation | 95% | 14min | [19] |
| 3 | Indigo Caramine | Electrocoagulation | 88% | 12min | [20] |
| 4 | Basic Blue | Enzyme, Fungus, Polymer | 90% | 7 Days | [21] |
| 5 | Remazol Brilliant Orange 3RID | Fenton's Oxidation | 97% | 93mins | [22] |
| 6 | Red 3BS | Liquid Membrane | 70% | - | [23] |
| 7 | Acid Black 172 | Electrocoagulation | 90.4% | 9.16min | [11] |
| 8 | Bismark Brown | Electrocoagulation | 97% | 30mins. | [24] |
| 9 | Brilliant Green | Adsorption By Citrus Limetta Peel | 95% | 4 hours | [17] |
| 10 | Brilliant Green | Electrocoagulation | 99.9% | 30mins | [12] |

# 7. Conclusion

According to the results of this investigation, RSM is a powerful statistical optimization tool for Brilliant Green dye removal using electrocoagulation process. The RSM results revealed that four selected parameters as well as some of their squares and interactions influenced the electrocoagulation performance. High $R^2$ value of 96.16% through ANOVA, verified that the accuracy of the Minitab proposed polynomial model is acceptable. The optimum Brilliant Green dye removal efficiency were found at NaCl concentration of 0.5008g/l, initial dye concentration of 500 mg/l, applied voltage of 4.0065V and reaction time of 12.22 min. An experiment was performed in optimum conditions which confirmed that the model and experimental results are in close agreement (99% compared to 98.9997% for the model).

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