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Retraction

Retracted: Optimization of Remazol Black B Removal Using Biochar Produced from *Caulerpa scalpelliformis* Using Response Surface Methodology

Advances in Materials Science and Engineering

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This article has been retracted by Hindawi, as publisher, following an investigation undertaken by the publisher [1]. This investigation has uncovered evidence of systematic manipulation of the publication and peer-review process. We cannot, therefore, vouch for the reliability or integrity of this article.

Please note that this notice is intended solely to alert readers that the peer-review process of this article has been compromised.

Wiley and Hindawi regret that the usual quality checks did not identify these issues before publication and have since put additional measures in place to safeguard research integrity.

We wish to credit our Research Integrity and Research Publishing teams and anonymous and named external researchers and research integrity experts for contributing to this investigation.

The corresponding author, as the representative of all authors, has been given the opportunity to register their agreement or disagreement to this retraction. We have kept a record of any response received.

References

[1] R. Gokulan, S. Balaji, and P. Sivaprakasam, "Optimization of Remazol Black B Removal Using Biochar Produced from *Caulerpa scalpelliformis* Using Response Surface Methodology," *Advances in Materials Science and Engineering*, vol. 2021, Article ID 1535823, 8 pages, 2021.

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Research Article

Optimization of Remazol Black B Removal Using Biochar Produced from *Caulerpa scalpelliformis* Using Response Surface Methodology

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Optimization of process conditions for the removal of Remazol Black B was investigated using response surface methodology (Box–Behnken design). The biodecolorization of dye was studied using biochar produced from waste biomass of *Caulerpa scalpelliformis* (marine seaweeds). The reactions were optimized by varying sorbent dosage, solution pH, temperature, and initial dye concentration. The results indicated that dye removal efficiency of 80.30% was attained at an operating condition of 4 g/L (sorbent dosage), 2.0 (solution pH), 35°C (temperature), and 0.25 mmol/L (initial dye concentration). The regression coefficient of the developed model was calculated to be 97% which shows good fit of the model.

1. Introduction

Wastewater generation has become one of the most important pollutants. All types of industrial wastewater and dye-bearing wastewater need effective treatment. Every year approximately 7 lakh metric tonnes of dyes are utilized. It is also predicted that 5% to 10% of these dyes are not utilized and are directly released into the water bodies [1, 2]. The use of dyes has increased in the textile and industrial sectors due to their convenience and natural coloring [3]. Dyes are commonly complex structures that are more stable, and it is difficult to remove them from the wastewater completely [4]. If these dyes without proper treatment are discharged into the nearby streams or rivers, they will affect the aquatic life [4]. Dyes will change the color of water bodies and reduce the photosynthesis process [4]. Dyes when mixed with drinking water will cause serious health issues; since dyes are toxic and poisonous in nature, they may cause many side effects to human health [5].

The removal of dyes can be achieved by treating with physical, chemical, or biological methods. These methods include coagulation with sedimentation and flocculation [6], photochemical oxidation [7], adsorption [8], ozonation [9], and electrochemical oxidation [10]. However, there are several limitations related to these methods [11]. The important disadvantage is cost associated with the treatments and generation of huge quantity of secondary pollutants to the environment [11]. In recent times, many researchers investigated biosorption of dye molecules using low cost adsorbents that are produced from naturally available waste materials. Some of the commonly used biomass materials are bagasse [12], rice husk [12], coffee bean husk [12], vine shoots [13], pecan shell [12], corn cob [12], walnut shell [12], coconut shell [14–16], and seaweeds [17, 18]. These naturally available materials are considered as wastes that are generated in huge quantity every year. Hence, utilizing waste biomass for the removal of toxic pollutants will result in the conversion of waste to energy [19]. Seaweed-based biochar is a recent eco-friendly tool that can be used as adsorbent, and it can be easily regenerated [20]. It is also reported that biochar is used for the soil enrichment [21, 22].

Biochar is produced in a limited oxygen environment at a temperature greater than 300°C. Feedstock used for the production of the biochar plays a very important role in deciding the characteristics of the biochar. Dry feedstocks (moisture content less than 30%) have more advantages than wet feedstocks (moisture content more than 30%). The main objective of the present research is to utilize green marine seaweed that is naturally available in the seashores as a sorbent for the remediation of the dye molecule. The cost associated with this adsorbent is very low. Nowadays, the statistical program developed by many researchers helped in carrying out many experiments in a shorter period of time in finding the optimum process conditions for the best outcome [23]. Response surface methodology (RSM) is a statistical tool used to study the interaction between different parameters at different levels [24–26]. The main objective of the present research is to utilize *Caulerpa scalpelliformis* for the remediation of the Remazol Black B molecule. *Caulerpa scalpelliformis* is a seaweed that is naturally overgrown in the coastal region of south Tamil Nadu. These seaweeds are well known for their antimicrobial activity, and their application in biosorption is less reported. The cost associated with this adsorbent is very low. Very limited research has been conducted using *Caulerpa scalpelliformis* for toxic pollutant removal, and production of biochar from this seaweed is not reported. So, the current research will provide a sustainable solution for the toxic pollutant removal.

2. Materials and Methods

2.1. Biochar and Dye. *Caulerpa scalpelliformis* marine seaweeds were obtained from seashores of Mandapam region (Tamil Nadu, India). The biomass was cleaned and sun-dried for 7 days. The dried biomass is used for the biochar production [27]. Different temperature ranges between 300 and 500°C are used for the pyrolysis to obtain maximum biochar yield. The maximum biochar yield of 47.5% was obtained at 350°C. Remazol Black B (RBB) used in the study was procured from Sigma-Aldrich. Remazol Black B has the following empirical formula: $C_{26}H_{21}N_5Na_4O_{19}S_6$, having color index of 20505, molecular weight of 991.82 mmol/g, and $\lambda_{\max}$ of 597. Figure 1 illustrates the structural composition of Remazol Black B.

2.2. Batch Studies. The batch studies were investigated in controlled environment using rotary shaker for 8 hours at 160 rpm. After the completion of the batch studies, the sample was centrifuged at 3000 rpm for 5 min to separate pellet. 5 ml of the clear solution was taken for the measurement of the final dye concentration using a spectrophotometer. The experiments were conducted under different conditions by varying sorbent dose (2, 4, and 6 g/L), pH (2, 3, and 4), temperature (25, 35, and 45 °C), and initial dye concentration (0.25, 0.5, and 1 mmol/L). Equations (1) and (2) are used to calculate the dye sorption and dye removal efficiency.

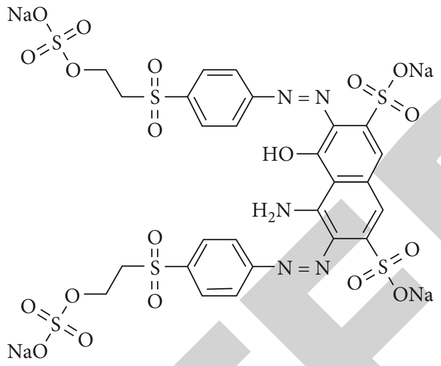

Figure 1: The structural composition of Remazol Black B (RBB).

$$Q = \frac{V\left(C_0 - C_e\right)}{W}, \tag{1}$$

$$\text{removal efficiency} = \frac{\left(C_0 - C_e\right)}{C_0} \times 100. \tag{2}$$

2.3. Experimental Design (RSM). The Box–Behnken design (BBD) was used to find the interaction of different parameters. Analysis of variance (ANOVA), residual plots, surface plot, and response optimizer were used to understand the interaction among different variables that will result in maximum removal efficiency. Table 1 summarizes the different levels of all independent variables. Equation (3) is used to analyze the BBD:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{K} \beta_{ii} x_i^2 + \sum_{i=1} 0 \sum_{j=i+1} \left(\beta_{ij} x_i x_{ij} + \varepsilon\right), \tag{3}$$

where Y denotes the response (% removal), β is the regression coefficient, x_i and x_j are independent variables, and ε represents the error.

3. Results and Discussion

3.1. Predictive Model. Equation (4) shows the model developed by the BBD using the input variables, and the response can be calculated.

$$\begin{aligned} Y &= 65.42 - 1.6A + 7.87B - 1.63C + 0.52D + 0.08A^2 \\ &\quad - 1.99B^2 - 20.66C^2 - 0.006D^2 + 3AC. \end{aligned} \tag{4}$$

The R^2 value decides the quality of the developed model. The R^2 value for the developed model is 0.9717, which is very close to unity and relatively high. This shows that 97% of the removal efficiency is based on the independent variable and only 3% of the total deviations are not correlated with the developed model. The high value of R^2 shows that the good fit of the developed model. The P value exceeding 0.05 is considered not to be significant and less than 0.05 is considered to be significant. From Table 2, the results show that equilibrium pH and initial dye concentration are significant. Table 2 summarizes the different factors of ANOVA. F value of 29.48 shows the significance of the model. The values of R^2 and $R^2{}_{\text{adj}}$ were found to be 0.9717 and 0.9388, respectively.

Table 1: BBD input variables with levels.

Levels			Variables	Variable code
1	0	−1		
2	4	6	Biochar dose (g/L)	*A*
2	3	4	Equilibrium pH	*B*
0.25	0.50	1.0	Initial dye concentration (mmol/L)	*C*
25	35	45	Temperature (°C)	*D*

These values showed that the developed model is good and the values of the independent variables are accurate with very less error. Thus, the developed model can predict the RBB removal using biochar derived from *Caulerpa scalpelliformis*. The developed model has good agreement with experimental results using four independent variables [28].

3.2. Optimization of Dye Removal. Table 3 summarizes the removal efficiency for 27 experimental trails. Optimization of the process variable is carried out using the quadratic model created with different levels of different variables. The RBB dye removal efficiency of 79.97% is achieved at a biochar dosage 4 g/L, equilibrium pH of 2, initial dye concentration of 0.25 mmol/L, and temperature of 35°C. The corresponding RBB dye removal efficiency with respect to the experiment is found to be 80.30%. The residual error of 0.333 was obtained between experimental and predicted values of the best trail. Thus, the result confirmed that the RSM is a reliable optimization tool for the RBB dye removal using biochar derived from *Caulerpa scalpelliformis.*

3.3. Residual Plots for Response Yield. From Figure 2, one can clearly understand the response of each experiment by studying the normal probability plot, fitted values, histogram, and observational orders. The normal probability plot and fitted values clearly show that all the experimental data are in accordance with the predicted values of the RSM. But only two observations deviate with a residual error of more than −1 and 2. From the histogram, frequency is grouped into five ranges. 8 frequencies are in the range of −0.25 to +0.25 residual errors, and 8 frequencies are in the range of −0.25 to −0.75. 9 frequencies are between 0.25 and 0.75, 1 frequency is in the range of −1.25 to −1.75, and 1 frequency is in the range of 1.75 to 2.25. From Figure 1, it can be clearly seen that all the values are found to be close to the predicted value of RSM, and a maximum residual error of −1.33 and 2.00 is observed in the plot for observations 13 and 14.

3.4. Influence of Process Parameters on Dye Removal. The dependent variable and the independent variable that influence the % dye removal can be studied by three-dimensional response surface plot. By fixing the other two variables at fixed levels, the dependent variable can be used to understand the mechanism. It can give a clear understanding of the main variables and interaction effects that influence the % dye removal.

3.5. Influence of Initial Dye Concentration and Biochar Dosage. Figure 3 shows the influence of initial dye concentration and biochar dosage on % dye removal for fixed pH of 3 and temperature of 35°C. Biochar dosage is a crucial parameter which decides the economy of the treatment. Figure 3 clearly shows that when the concentration is increased, % removal of dye decreases. As reported by Vijayaraghavan and Yun [29], the surge in dye concentration will reduce % removal of dye, and this is because at higher concentration, the uptake capacity becomes saturated, and further sorption will not take place and affects the % dye removal. It is also observed from Figure 3 that % removal of dye increases with the surge in the biochar dosage. % removal of dye gradually increases from a dosage of 2 g/L to 6 g/L. A similar type of work was carried out by Gokulan et al. [18] who reported that increase in dosage increases the % removal of dye. It is also reported that % removal of dye depends not only on the mass of sorbent but also on the uptake capacity of the biochar. Higher biochar dosages will have sufficient exchangeable active binding sites on the sorbent matrix [30].

3.6. Influence of Initial Dye Concentration and pH. Figure 4 illustrates the influence of initial dye concentration and pH at fixed biochar dosage of 4 g/L and temperature of 35°C. Figure 4 shows that surge in pH from 2 to 3 reduced the removal efficiency. This clearly shows that if pH is not maintained properly, it will decrease the % removal of dye. Due to the presence of lignocellulosic constituents that comprise carboxyl, sulfate, and amine groups, the removal efficiency will increase with a decrease in pH. Presence of these compounds will increase the interactions between biochar and the dyes which will increase % removal of dye.

3.7. Influence of Initial Dye Concentration and Temperature. From Figure 5, it is obvious that the rise in temperature improved % removal of dye from the solute. Gokulan et al. [18] in his work reported that increase in temperature strongly influences % dye removal and also increases the sorption capacity of the sorbent that is used for dye removal. But from the economic point of view, cost will be high if the temperature is maintained at 45°C, and the difference in % dye removal between 35°C and 45°C is only around 2 to 3%. Since 35°C is the room temperature, it may be considered as optimum for the dye removal.

3.8. RSM Optimizer. RSM optimizer is employed to visualize the percentage increase in removal efficiency of the dye by considering process conditions. From Figure 6, it is

Table 2: Analysis of variance for the removal of Remazol Black B.

Source	Degree of freedom	Seq SS	Adj MS	*F*	*P*
Regression	14	343.969	24.5692	29.48	<0.0001
Linear	4	302.918	2.4373	2.92	0.067
A	1	15.413	1.0898	1.31	0.275
B	1	199.267	5.2132	6.26	0.028
C	1	79.568	0.0177	0.02	0.886
D	1	8.670	1.9743	2.37	0.150
Square	4	32.051	8.0127	9.62	<0.001
A^2	1	8.263	0.5926	0.71	0.416
B^2	1	14.681	21.1559	25.39	<0.001
C^2	1	7.078	8.8981	10.68	0.007
D^2	1	2.028	2.0281	2.43	0.145
Interaction	6	9.000	1.5000	1.80	0.182
AC	1	9.000	9.0000	10.80	0.007
Residual error	12	10.000	0.8333	*	*
Lack of fit	10	10.000	1.0000		
Pure error	2	0.000	0.0000		
Total	26	353.969			

$R^2 = 0.9717$; $R^2_{\text{adj}} = 0.9388$.

Table 3: Experimental and predicted responses of BBD with residual error.

Run order	*A* Biochar dosage (g/L)	*B* Equilibrium pH	*C* Initial dye concentration (mmol/L)	*D* Temperature (°C)	Yield (%) Experiment	Yield (%) RSM	Residuals
1	4	2	0.25	35	80.30	79.97	0.333
2	4	2	0.75	35	74.15	74.82	−0.667
3	2	3	0.75	35	71.10	70.43	0.667
4	4	3	0.50	35	76.60	76.60	0.000
5	6	3	0.50	45	77.80	78.30	−0.500
6	4	4	0.50	45	71.10	70.77	0.333
7	4	3	0.25	25	76.75	76.42	0.333
8	4	2	0.50	45	79.25	78.92	0.333
9	2	4	0.50	35	69.90	69.73	0.167
10	4	4	0.50	25	69.40	69.07	0.333
11	4	3	0.50	35	76.60	76.60	0.000
12	2	2	0.50	35	78.05	77.88	0.167
13	2	3	0.25	35	77.25	78.58	−1.333
14	6	3	0.75	35	77.70	75.70	2.000
15	4	3	0.75	45	72.30	72.97	−0.667
16	4	3	0.25	45	78.45	78.12	0.333
17	4	3	0.50	35	76.60	76.60	0.000
18	2	3	0.50	25	74.50	74.33	0.167
19	4	4	0.75	35	66.00	66.67	−0.667
20	2	3	0.50	45	76.20	76.03	0.167
21	6	2	0.50	35	79.65	80.15	−0.500
22	4	4	0.25	35	72.15	71.82	0.333
23	6	3	0.25	35	77.85	77.85	0.000
24	4	2	0.50	25	77.55	77.22	0.333
25	6	4	0.50	35	71.50	72.00	−0.500
26	6	3	0.50	25	76.10	76.60	−0.500
27	4	3	0.75	25	70.60	71.27	−0.667

apparent that removal efficiency of 80.95% is achieved at a biochar dose of 2 g/L, equilibrium pH of 2.0, initial dye concentration of 0.25 mmol/L, and temperature of 41.96°C (≈42°C). It is also predicted that the composite desirability of 0.9973 is achieved for these process conditions, which shows that the predicted values are accurate. Batch studies were conducted with these predicted process conditions. Three batch trails were conducted, and the removal efficiency obtained was 81.10%, 81.05%, and 80.98%, respectively. So, the average removal efficiency obtained from the batch study is 81.04%. The obtained values agree with the predicted value. From the results, it is concluded

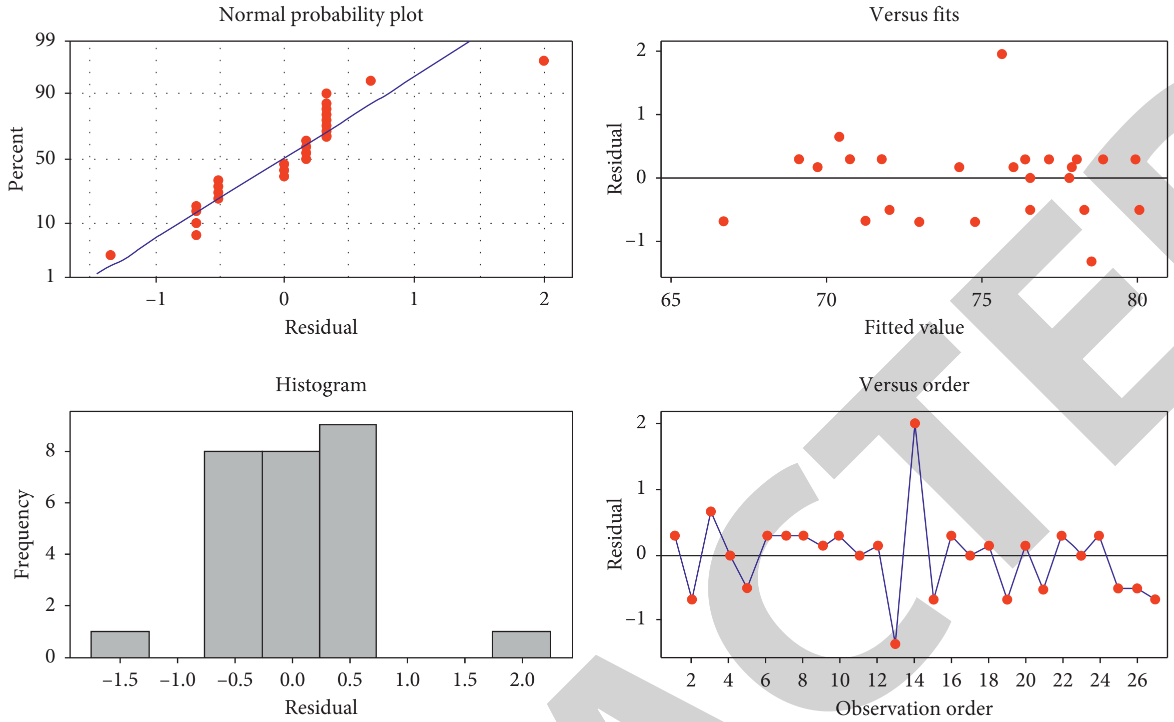

Figure 2: Residual plots for response yield.

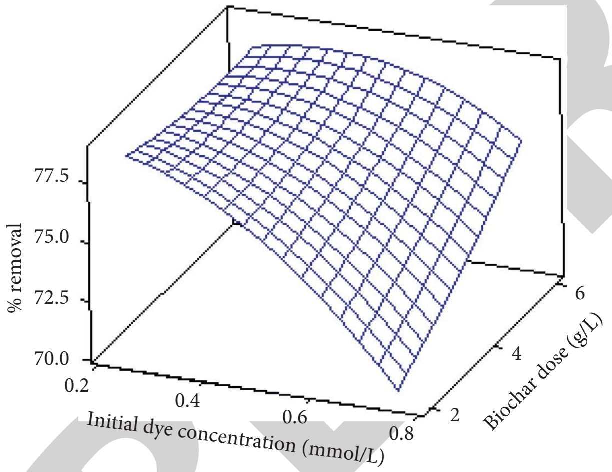

Figure 3: Effect of initial dye concentration (mmol/L) and biochar dosage (g/L).

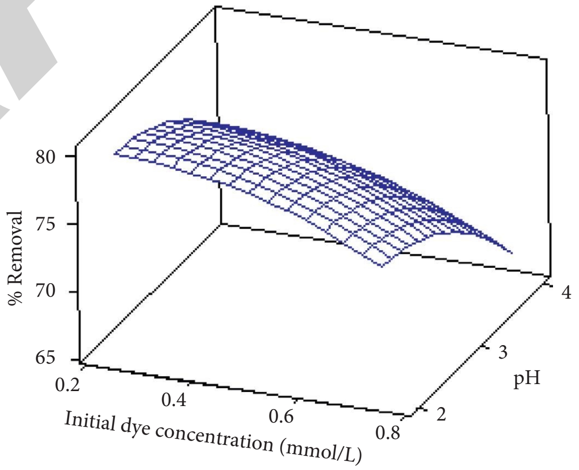

Figure 4: Effect of initial dye concentration (mmol/L) and pH.

that RSM optimizer increased the removal efficiency from 79.97% to 80.95%, i.e., increase of 0.98% (≈1%). The batch studies revealed that the removal efficiency increased from 80.30% to 81.04%, i.e., increase of 0.74%.

3.9. Sorption Isotherm and Kinetic Studies. To understand the mechanism of adsorption, batch study was carried out at different initial dye concentrations varying from 0.1 to 1 mmol/L at constant pH, temperature, and biochar dosage. Kinetic study is also carried at varying time interims from 5 to 360 minutes to determine the removal efficiency with respect to time. From isotherm studies, it is concluded that the highest uptake of 0.161 mmol/g is attained in the Toth model. Langmuir, Freundlich, Sips, and Toth models were used, and the Toth model is found to have a highest regression coefficient of 0.9999 and % error of 0.6042. From the isotherm studies, it is also concluded that the increase in initial dye concertation decreased removal efficiency. For instance, at an initial dye concentration of 0.05 mmol/L, the removal efficiency is found to be 81.2%, whereas at 1 mmol/L, the removal efficiency is found to be 32.1%. So, at low concentration, biochar is capable of acting as an effective adsorbent. The kinetic study results showed that adsorption was maximum in the first 90 minutes. At a time interval of 120 minutes, almost 90% of the dyes are adsorbed, and a

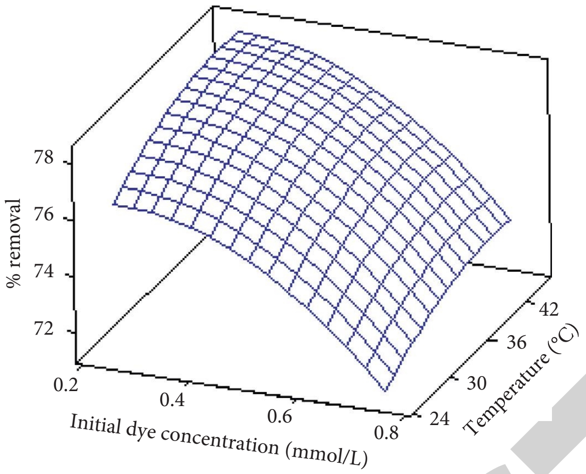

Figure 5: Effect of initial dye concentration (mmol/L) and temperature (°C).

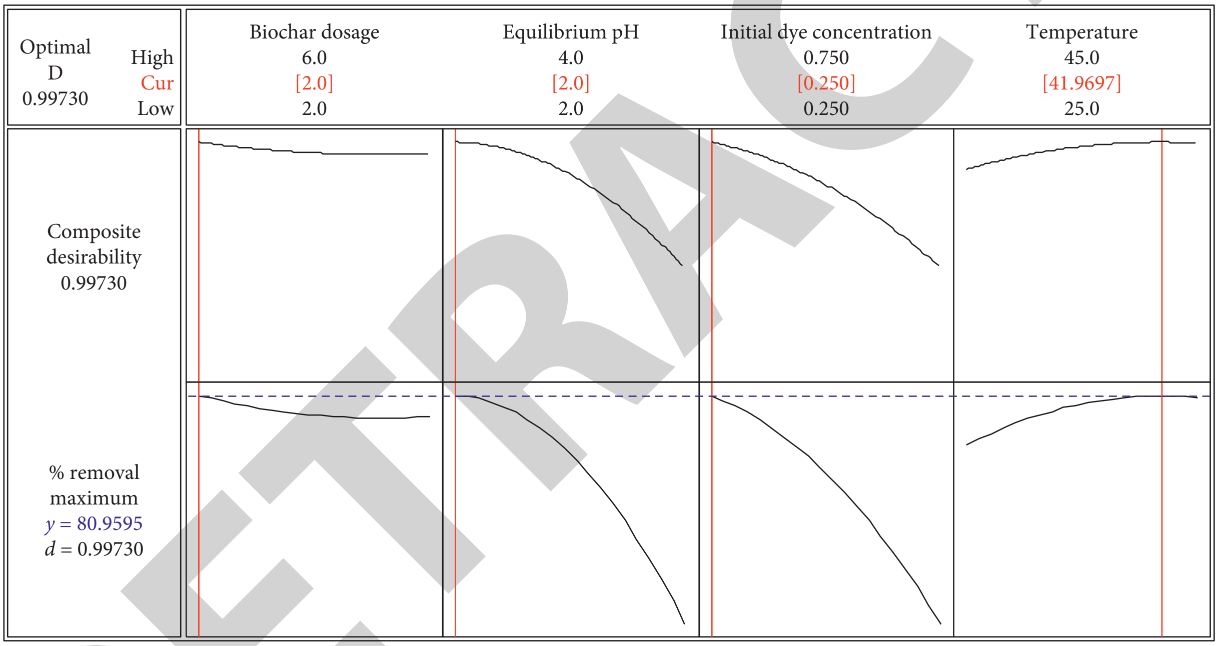

Figure 6: RSM optimizer for enhanced dye removal efficiency.

further increase in time resulted in very less adsorption. Kinetic study results show that a contact time of 120 minutes gives optimum values. Pseudo-first-order and pseudo-second-order models were used to predict the uptake capacity of the adsorbent, and the pseudo-first-order model was found to have highest regression coefficient of 0.99 under all conditions.

4. Conclusion

From the study, it is concluded that *Caulerpa scalpelliformis*-derived biochar can be effectively used for the removal of RBB. The RSM-based BBD matrix for an independent variable is developed, and the results showed that the predicted value and experimental value are close to each other. RBB dye removal efficiency of 79.97% is achieved at a biochar dosage of 4 g/L, equilibrium pH of 2, initial dye concentration of 0.25 mmol/L, and temperature of 35°C, whereas for the same conditions, the removal efficiency of 80.30% is obtained in experimental studies, which is very close to the predicted value of the RSM with a residual error of 0.333. Thus, the RSM model successfully determined the removal efficiency of RBB dye using biochar derived from *Caulerpa scalpelliformis*.

Data Availability

The data used to support findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest regarding the publication of this paper.

Acknowledgments

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