P-ISSN: 2659-1561
E-ISSN: 2635-3040

Research Article

Sorption of Bromo Cresol Purple Dye with *Gelidium amansii* Red Algae Powder Along With Statistical Optimization

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Received: May 14, 2020 **Accepted:** May 24, 2020 **Published:** May 30, 2020

Abstract: The current examination is on the expulsion of Bromo Cresol Purple dye from watery arrangements utilizing *Gelidium amansii* powder as sorbent. The aggregate impacts of working boundaries, for example, biosorbent size, dye concentration, pH of the arrangement, biosorbent dose and temperature on the dye biosorption were dissected utilizing UV Spectrophotometer. For getting the common association between the factors and enhancing these factors a Central Composite Design was used. As indicated by ANOVA results, the proposed model for CCD was in appropriate understanding with the test information. Portrayal examines involving Fourier Transform Infrared Spectroscopy (FTIR), XRD and Scanning Electron Microscope (SEM) examination was performed to confirm the examination. Tentatively the ideal arrangement of conditions for greatest rate biosorption of Bromo Cresol Purple color are discovered to be at biosorbent size= 53 µm, pH= 7, biosorption dosage (w) = 25 g/L, beginning dye Concentration (C0) = 20 mg/L and temperature=303 K and the biosorption determined at these qualities was discovered to be 85%. The Freundlich isotherm fitted well with a connection factor followed by Langmuir and Temkin. The whole biosorption measure followed pseudo second request energy. By applying the Van't Hoff condition the thermodynamic boundaries, for example, enthalpy (ΔH), entropy (ΔS) and free energy (ΔG) were assessed which depicted the biosorption interaction as unconstrained, irreversible and endothermic in nature. The advanced qualities acquired through focal composite plan and each factor in turn measure is in acceptable understanding.

Keywords: Sorption of dyes, pH, Kinetics, Isotherms, and Optimization.

Introduction

Water consumption has increased worldwide in every economic sector. In recent years, water pollution has improved by industrial development. Therefore, population increases without any planning urbanization. Several kinds of pollutants could be found in industrial wastewater in huge amounts. The aquatic ecosystem is suffering from piles of organic pollutants including dyes causing toxic and carcinogenic effects on the human beings. For example, just textile industry uses around 10,000 different types of dyes; its annual production is higher than 7 × 105 tons. It has been estimated that 10–15% of dyes are lost in dying textile water. Some of them are hard to remove from aqueous phase because of their solubility. For this reason, dyes removal is an important challenge for the environmental chemistry. Recently, the concern about finding other methods for organic compounds degradation has been substantially increased [01–09].

Materials and Methodology

The present experimentation is carried out in batch-wise, on biosorption of Bromo Cresol Purple (BCP) dye from aqueous solution onto *Geledium amansii* red algae powder.

Reagents and Chemicals

Bromo Cresol Purple (BCP) is used as source of dye stock solution.

Preparation of the bisorbents

Gelidium amansii algae was collected from Tenneti Park, Jodugullapalem beach in Visakhapatnam city, Andhra Pradesh, India. The collected algae was washed with distilled water several times until the dirt particles are removed. After through washing with distilled water, biosorbent was sun dried until it became crispy, cut into small pieces, powdered and sieved. In the present study, 53, 75, 105, 125 and 152 µm size powders was used as biosorbent without any other pretreatments.

Figure 1. *Gelidium amansii* algae

Characterization of biosorbents

Sorbent characterization was carried out using FTIR, the X-Ray Diffractograms (XRD) and Scanning Electron Microscopy.

Preparation of Dyes stock solutions

Bromo Cresol Purple (BCP), is used as the source for preparing stock solution (synthetic). All the required solution is prepared with double distilled water. 1.0 g of dye was dissolved in 1.0 L of distilled water to prepare 1000 mg/L stock solution. Samples of different concentrations of dye is prepared from this stock solution by appropriate dilutions. 10 mg/L of BCG solution is prepared by proper dilutions. 10 ml of 1000 mg/L BCG stock solution is taken in a 1000 ml volumetric flask and is made up to the mark with distilled water. Similarly solution with different concentrations such as 20 mg/L, 50 mg/L, 100 mg/L, 150 mg/L and 200 mg/L were prepared. The pH of the solution is varied by adding required amounts of 0.1 N HCl and 0.1N NaOH.

Results and Discussion

Effect of Contact Time

The rate biosorption is plotted against contact time in figure 1. The % biosorption is found to increase up to 25 min. The highest level of biosorption is achieved at 25 min of agitation and gets steady after 25 min with % biosorption of 64. The analysis revealed that a directly proportionate relationship between the increase in contact time and adsorption process, and in particular, areas where large amounts of dyes were adsorbed within the first 20 min. It was observed that within 25 min, the dye removal ratios for BCP had reached equilibrium. This result suggests two sequential steps in BCP dye adsorption on Gelidium: (i) A quick BCP dye transfer to the external surface of Gelidium; and (ii) A relatively slower step of diffusing dyes between the Gelidium bundles [10-12].

P-ISSN: 2659-1561
E-ISSN: 2635-3040

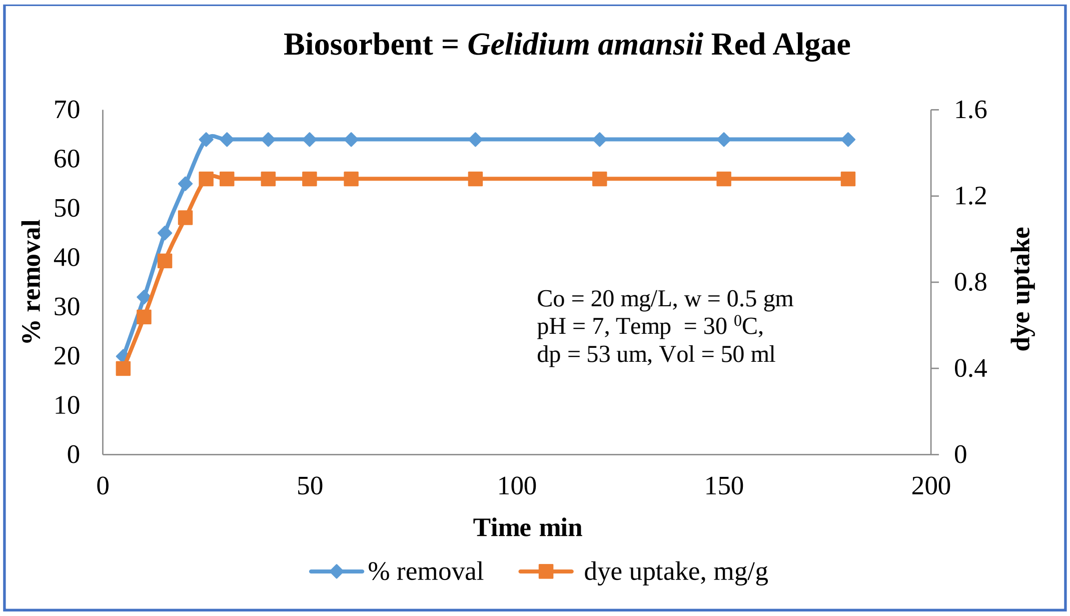

Figure 1. Effect of Contact Time on % removal of BCP dye

Effect of Bisorbent Size

Figure 2 represents the rate biosorption of Bromo Cresol Purple with the biosorbent size. The rate biosorption is expanded from 45 % to 64 % as the biosorbent size is diminished from 152 to 53 µm. As the size of the molecule diminishes, surface territory of the biosorbent upgrades and additional number of dynamic destinations on the biosorbent are accessible to the biosorbate. The rate of dye removal shows an increasing trend as the particle size decreases. The highest efficiency (64%) was obtained with gelidium with a particle size of 53 µm [13-15].

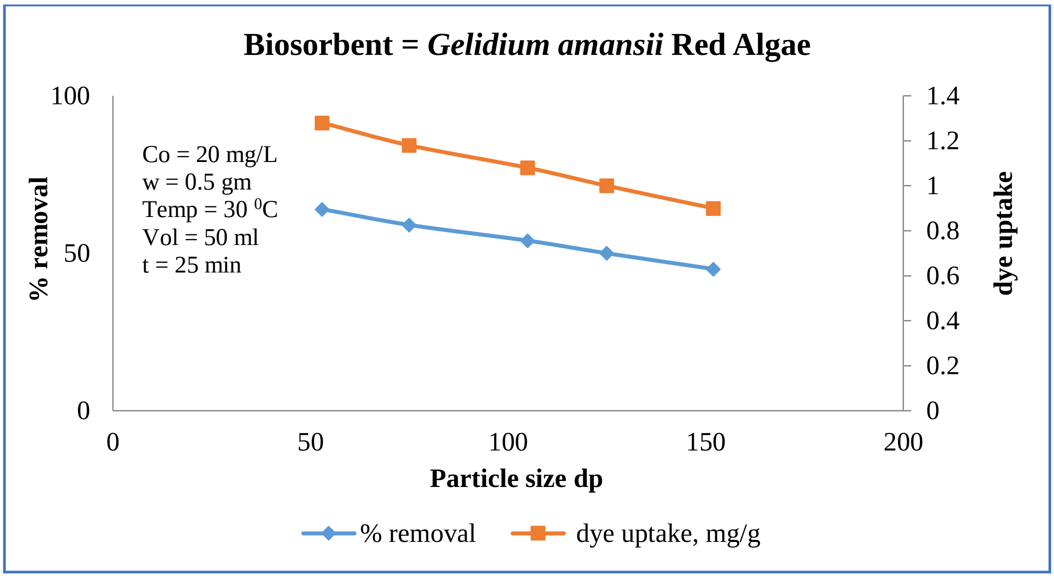

Figure 2. Effect of Bisorbent size on % removal of BCP dye

Effect of pH in aqueous solution

The impact of pH of watery arrangement on rate biosorption of Bromo Cresol Purple is presented figure 3. The rate biosorption is expanded from 40 % to 64 % as pH is expanded from 2 to 7. The rate biosorption is diminished from 64 % to 50 % as pH increments from 7 to 8. The main factor for dye biosorption is the electrostatic force of attraction between biosorbent and biosorbate. At acidic pH values, the surface of the biosorbent carries a positive charge due to the presence of H^+ in the solution. This leads to electrostatic repulsion between the cationic bromo cresol purple dye and positively charged Gelidium surface as biosorbent. On the contrary, at alkaline pH values, due to the existing of OH^- ions in excess, the surface of the biosorbent carries a negative charge. This results in electrostatic attraction between cationic bromo cresol purple dye and negatively charged Gelidium surface leading to enhanced adsorption at higher pH values [16, 17].

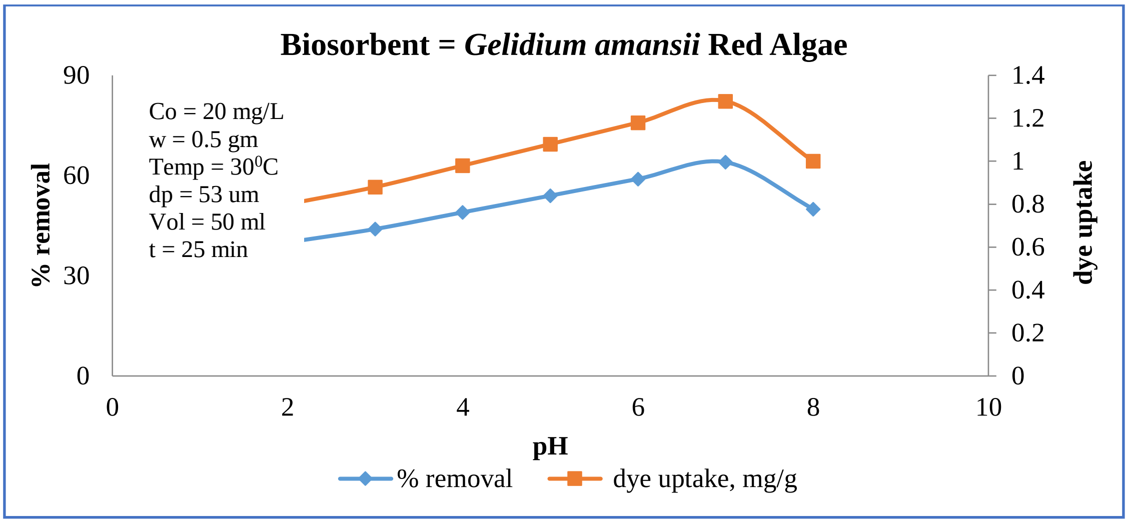

Figure 3. Effect of pH on %removal of BCP dye

Effect of Initial Concentration of BCP dye

A plot figure 4 with rate biosorption of Bromo Cresol Purple as an element of initial concentration of Bromo Cresol Purple. The rate biosorption is decreased from 64 % to 45 % as the Bromo Cresol Purple in the aqueous solution increased from 20 mg/L to 200 mg/L. A higher initial concentration provides an important driving force to overcome all mass transfer resistances of the dye between the aqueous and solid phases, thus increases the uptake. In addition, increasing initial dye concentration increases the number of collisions between dye anions and sorbent, which enhances the sorption process. When the initial dye concentration increased from 20 to 200 mg/L, the uptake capacity of Gelidium increased from 1.28 to 9.0 mg g^{-1} for BCP dye at 30 ^{0}C. However, higher adsorption yields were observed at lower concentrations of each dye [18-20].

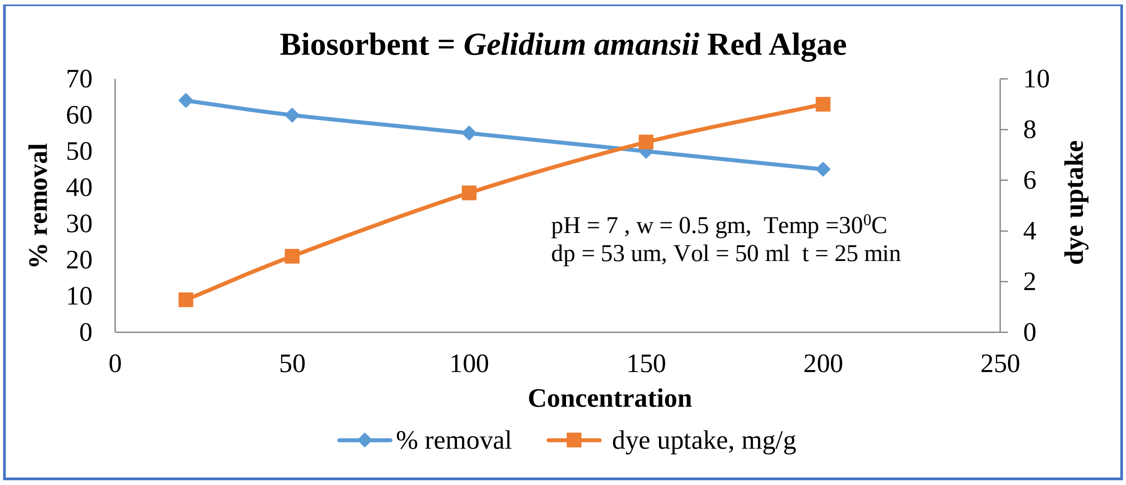

Figure 4. Effect of Initial Concentration on %removal of BCP dye

Effect of Bisorbent Dosage

The rate biosorption of Bromo Cresol Purple is drawn against biosorbent size in figure 5. The rate biosorption increments with increase in biosorbent measurements. For a biosorbent dosage of 53µm, rate biosorption increments from 64 % to 89 %, as dosage is increased from 10 to 70 g/L. Such that it is clear on the grounds that the quantity of accessible sites for dye removal would be more as the dosage of the biosorbent increased [21-22].

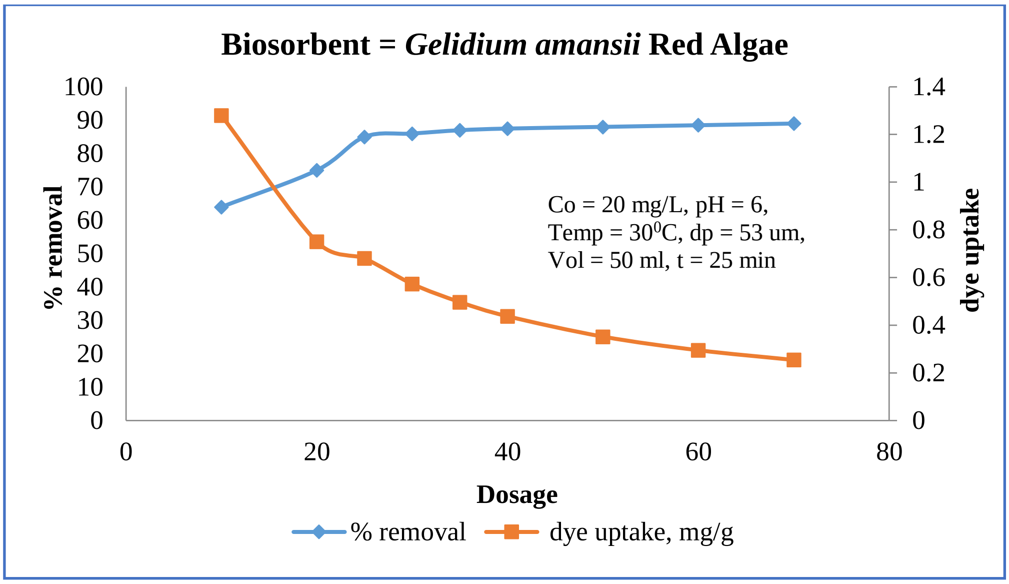

Figure 5. Effect of Bisorbent Dosage on %removal of BCP dye

Effect of Temperature

The impact of temperature on the balance color take-up was critical. The impact of changes in the temperature on the Bromo Cresol Purple take-up is appeared in figure 6. The biosorption limit of color is expanded at higher temperatures, which shows that biosorption of colors in this framework is an endothermic cycle. This might be credited to expanded entrance of responsive colors inside micropores at higher temperatures or the making of new dynamic sites [23-25].

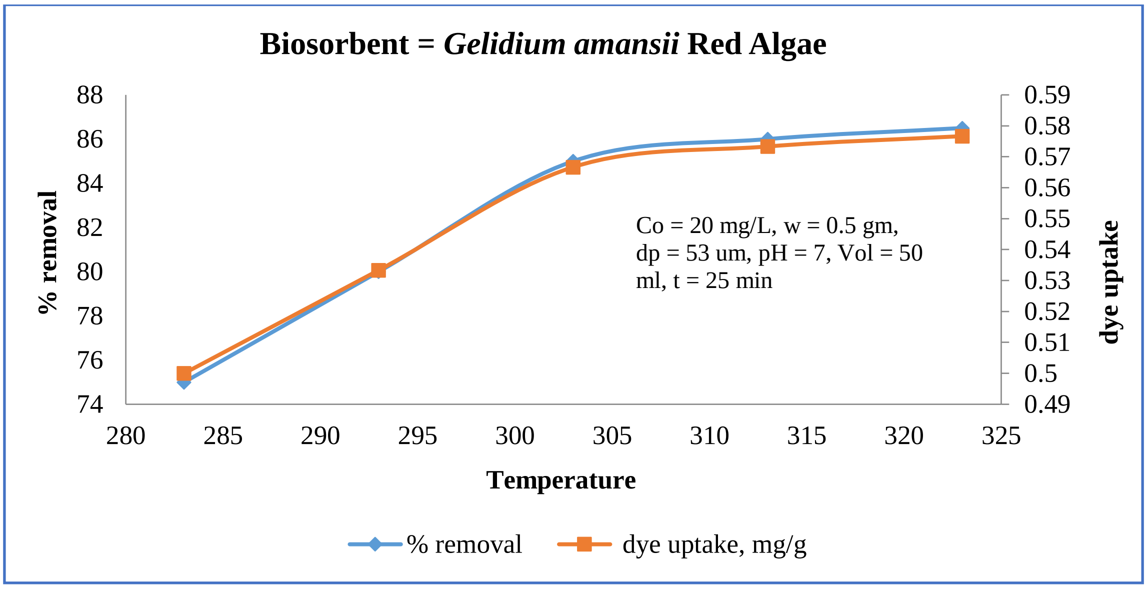

Figure 6. Effect of Temperature on %removal of BCP dye

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Isotherms

Langmuir isotherm

Langmuir isotherm is drawn between Ce/qe and Ce in figure 7 for the current information. The subsequent condition is

Ce/qt = 0.0631 Ce + 502911 - (3)

The connection coefficient estimation of 0.9989 shows solid official of Bromo Cresol Purple color on to the biosorbent [26-27].

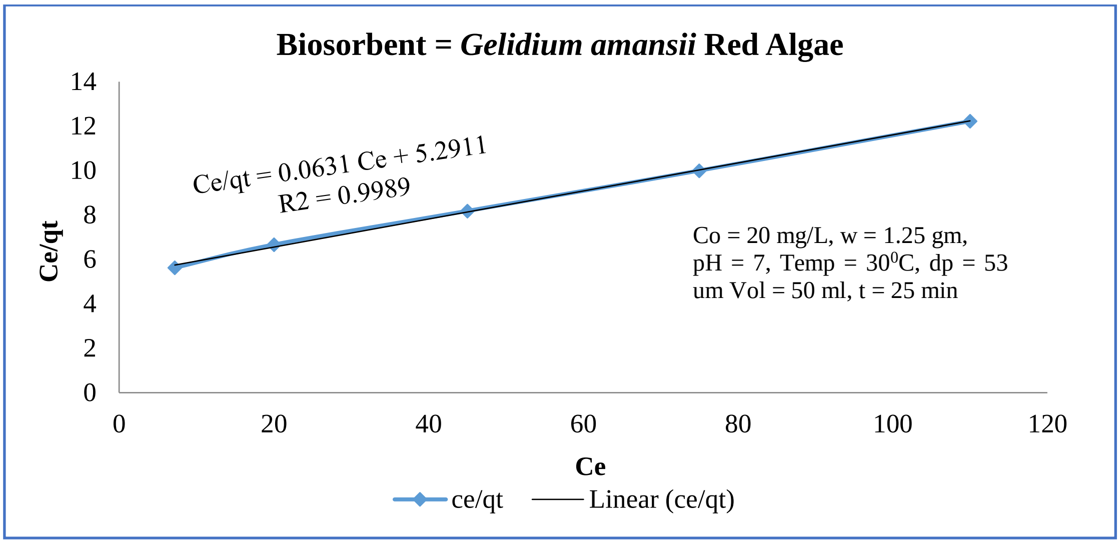

Figure 7. Langmuir isotherm for %removal of BCP dye

Freundlich isotherm

Freundlich isotherm is drawn between ln C_e and ln q_e in figure 8, resulted in the following equation

ln q_e = 0.7233 ln C_e - 1.1217 ----- (4)

The equation has a correlation coefficient of 0.9927. The 'n' value of 0.61472 indicates favorable biosorption satisfying the condition of 0<n<1.

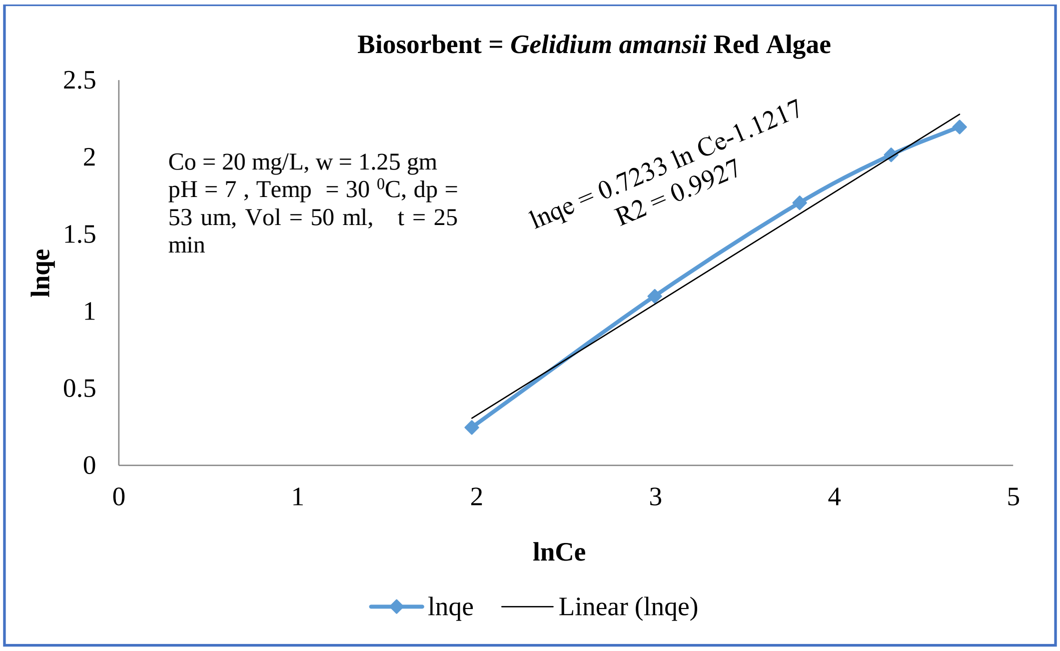

Figure 8. Freundlich isotherm for %removal of BCP dye

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Temkin isotherm

The current information are investigated by the direct structure. The direct plot of Temkin isotherm is appeared in figure 9.

The condition acquired for Bromo Cresol Purple biosorption is:

qe = 2.8536 ln Ce – 4.8997 - (4)

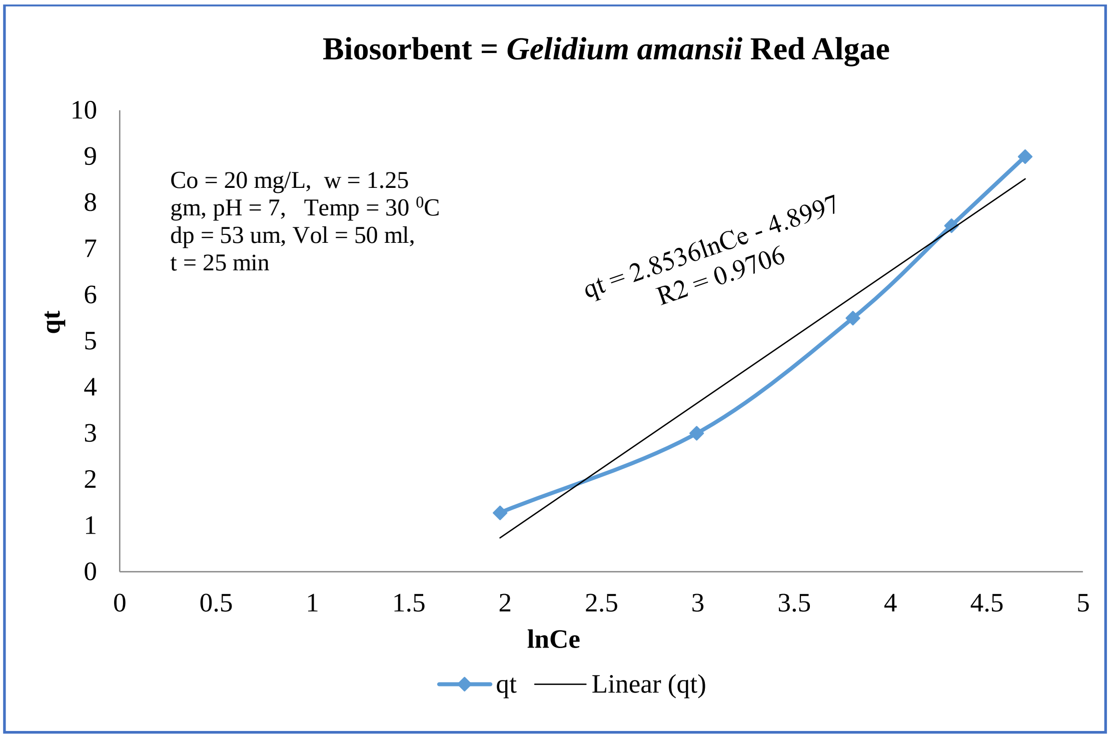

Figure 9. Temkin isotherm for %removal of BCP dye

Table 1. Isotherm constants

Langmuir isotherm	Freundlich isotherm	Temkin isotherm
q_m = 15.84786 mg/g	K_f = 0.325726 mg/g	A_T = 0.1796 L/mg
K_L = 0.011926	n = 0.61472	b_T = 882.7944
R^2 = 0.9989	R^2 = 0.9927	R^2 = 0.9706

Kinetic Studies

The test information are tried for Lagergren first request rate condition and pseudo second request rate condition. Lagergren plot of log (qe-qt) versus disturbance time (t) is appeared in figure 10(a) and pseudo second request energy plot between 't' vs 't/qt' for biosorption of Bromo Cresol Purple is attracted figure 10 (b).

Table-2 sums up rate consistent qualities for first and second request rate conditions. It is noticed that both first and second request rate conditions clarify the biosorption cooperations [28-29].

Lagergren First order Kinetics

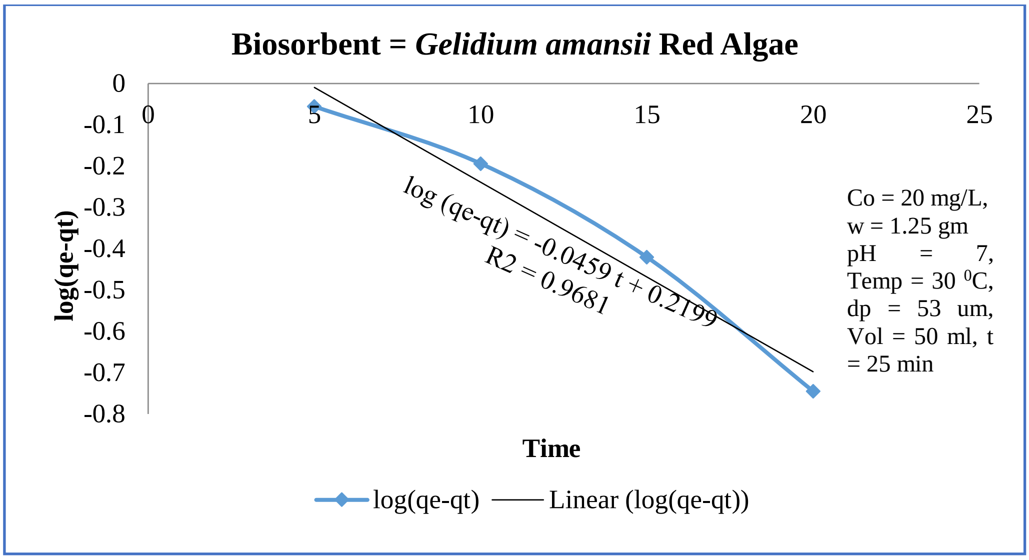

Figure 10 (a). First order kinetics for %removal of BCP dye

Pseudo Second order Kinetics

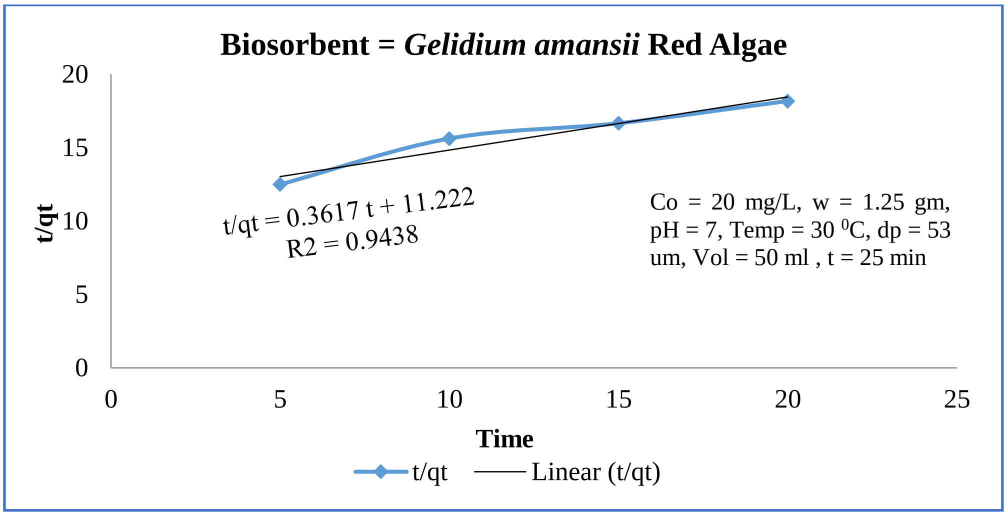

Figure 10 (b). Pseudo second order kinetics for %removal of BCP dye

The correlation coefficients for the second-order kinetic model are obtained greater than 0.9438 for all sorbents studied. The theoretical *q*eq values also agreed very well with the experimental *q*eq values in the case of pseudo-second-order kinetics. These suggest that each of the sorption process may be best described by the pseudo-second order with intraparticle diffusion as one of the rate determining steps with fairly high correlation coefficients.

Dependence of adsorption rate constants on temperature gives valuable information about the activation energy of adsorption. The findings showed that dye sorption process is endothermic and involves chemical sorption for Gelidium [31-33].

Table 2. Equations and rate constants

Order	Equation	Rate constant	R^2
Lagergren first order	$\log(q_e - q_t) = -0.0459\ t + 0.2199$	0.063563 min^{-1}	0.9681
Pseudo second order	$t/q_t = 0.3617\ t + 11.222$	0.020805g/(mg-min)	0.9438

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Thermodynamics

A progression of thermodynamic boundaries-change in Gibbs free energy (ΔG) change in enthalpy (ΔH) and change in entropy (ΔS) are resolved. ΔG estimation of -10398.3 J/mole demonstrates that biosorption of Bromo Cresol Purple by *Gelidium amansii* red algae powder could occur unexpectedly.

Higher temperatures have profited biosorption and expanded the harmony biosorption limit. Positive ΔH of 14.97307 J/mole demonstrates shows endothermic nature of biosorption while positive ΔS = 34.36721 J/mole-K demonostrates the liking of *Gelidium amansii* red algae powder to Bromo Cresol Purple [30-32].

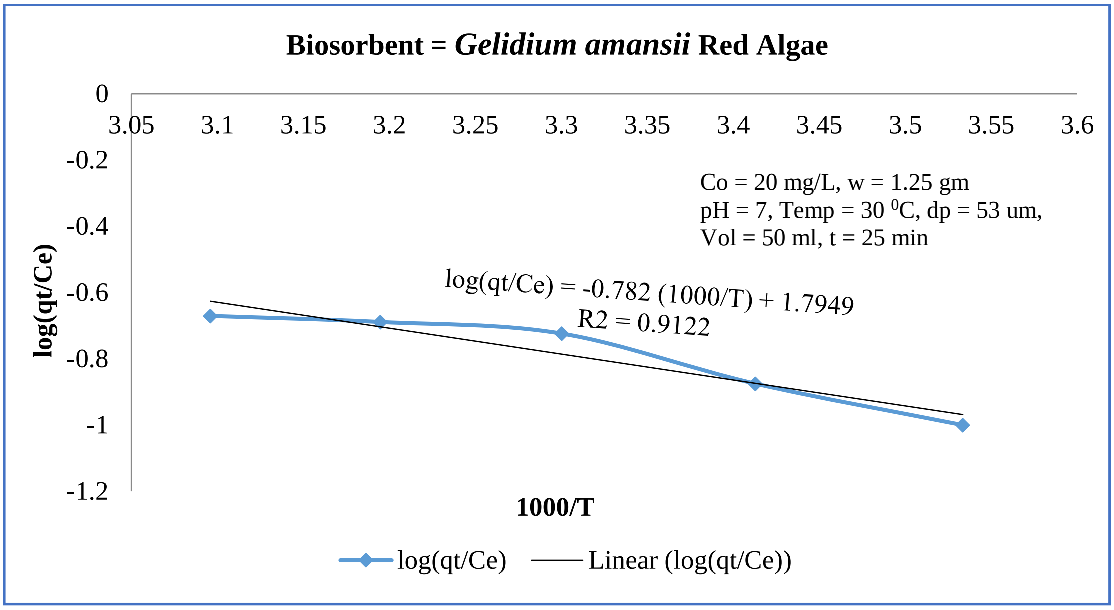

Figure 11. Vanthoff's plot for %removal of BCP dye

Optimization using Response Surface Methodology (RSM)

Optimization of biosorption conditions using CCD

In the current investigation, the degrees of four cycle input factors for % biosorption are appeared in table–3.

Table 3. Levels of different process variables in coded and un-coded form for % biosorption of Bromo Cresol Purple using *Gelidium amansii* red algae

Variable	Name	Range and levels				
		-2	-1	0	1	+2
X_1	pH of aqueous solution	3	4	5	6	7
X_2	Initial concentration, C_o, mg/L	10	15	20	25	30
X_3	Biosorbent dosage, w, g/L	20	25	30	35	40
X_4	Temperature, K	283	293	303	313	323

The boundaries that have more prominent impact over the reaction are to be distinguished to locate the ideal condition for the biosorption of BCP color. For improvement of medium constituents, the relapse condition is:

$$Y = -3154.84 + 70.75\,X_1 + 4.97\,X_2 + 4.38\,X_3 + 18.95\,X_4 - 5.00\,X_1^2 - 0.13\,X_2^2 - 0.08\,X_3^2 - 0.03\,X_4^2 + 0.00\,X_1X_2 - 0.00\,X_1X_3 - 0.00\,X_1X_4 + 0.00\,X_2X_3 - 0.00\,X_2X_4 + 0.00\,X_3X_4 \quad (10)$$

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Table 4. Results from CCD for Bromo Cresol Purple biosorption by *Gelidium amansii* red algae powder

Run No.	X_1, pH	X_2, C_o	X_3, w	X_4, T	% biosorption of Bromo Cresol Purple	
					Experimental	Predicted
1	6	15	20	293	74.28000	74.30303
2	6	15	20	313	76.52000	76.46303
3	6	15	30	293	76.98000	76.98226
4	6	15	30	313	79.18000	79.22726
5	6	25	20	293	74.52000	74.52387
6	6	25	20	313	76.68000	76.72887
7	6	25	30	293	77.32000	77.28810
8	6	25	30	313	79.62000	79.57810
9	8	15	20	293	75.18000	75.19720
10	8	15	20	313	77.28000	77.32220
11	8	15	30	293	77.88000	77.84143
12	8	15	30	313	80.08000	80.05143
13	8	25	20	293	75.52000	75.48303
14	8	25	20	313	77.68000	77.65303
15	8	25	30	293	78.18000	78.21226
16	8	25	30	313	80.48000	80.46726
17	5	20	25	303	69.82000	69.81554
18	9	20	25	303	71.58000	71.59887
19	7	10	25	303	77.78000	77.76887
20	7	30	25	303	78.38000	78.40554
21	7	20	15	303	79.52000	79.50807
22	7	20	35	303	84.98000	85.00154
23	7	20	30	283	75.28000	75.28720
24	7	20	30	323	79.78000	79.78720
25	7	20	30	303	89.98000	89.98080
26	7	20	30	303	89.98000	89.98080
27	7	20	30	303	89.98000	89.98080
28	7	20	30	303	89.98000	89.98080
29	7	20	30	303	89.98000	89.98080
30	7	20	30	303	89.98000	89.98080

Experimental conditions [Coded Values] and observed response values of central composite design with 2^4 factorial runs, 6-central points and 8-axial points. Agitation time fixed at 25 min and biosorbent size at 53 µm. % biosorption of Bromo Cresol Purple (Y) is an element of pH (X_1), Co (X_2), w (X_3), and T (X_4). The varieties in the relating coded estimations of four boundaries and reaction are introduced in table-5 relying upon exploratory runs and anticipated qualities proposed by CCD plan. The accompanying condition speaks to different relapse examination of the trial information:

The consequences of eq. 7 are introduced as ANOVA. From the Fisher's F-test and an exceptionally low likelihood esteem (Pmodel > F=0.000000), the ANOVA of the model obviously clarifies that the model is profoundly critical (Refer table 6). It shows that the treatment contrasts are critical.

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Table 5. ANOVA of Bromo Cresol Purple biosorption for entire quadratic model

Source of variation	SS	df	Mean square (MS)	F-value	*P* > F
Model	993.0965	14	70.9354	51402.4638	0.00000
Error	0.0207	15	0.00138		
Total	993.1172				
df- degree of freedom; SS- sum of squares; F-factor F; *P*- probability, R^2=0.99998; R^2 (adj):0.99996.					

It is anticipated from table 6 that the bigger the estimation of t and more modest the estimation of P, the more huge is the relating coefficient term. The ‘t’ and 'P' values are investigated from table 6 to foresee the reaction. It is discovered that X_1, X_2, X_3, X_4, X_1^2, X_2^2, X_3^2, $X_4^{2,}$ X_1X_2, X_1X_3, X_2X_3 and X_2X_4 have high noteworthiness to clarify the individual and collaboration effect of information factors on biosorption of Bromo Cresol Purple.

Table 6. Estimated regression coefficients for the Bromo Cresol Purple biosorption onto *Gelidium amansii* red algae powder

Term	Regression	Standard error	T(15)	p
Mean/Intercept	-3154.84	7.281774	-433.251	0.000000
Dosage, w, g/L (L)	70.75	0.311938	226.814	0.000000
Dosage, w, g/L (Q)	-5.00	0.008568	-584.068	0.000000
Conc, Co, mg/L (L)	4.97	0.060155	82.699	0.000000
Conc, Co, mg/L (Q)	-0.13	0.000343	-368.738	0.000000
pH (L)	4.38	0.054898	79.716	0.000000
pH (Q)	-0.08	0.000412	-205.580	0.000000
Temperature, T, K (L)	18.95	0.044358	427.284	0.000000
Temperature, T, K (Q)	-0.03	0.000072	-433.356	0.000000
1L by 2L	0.00	0.001859	1.748	0.100844
1L by 3L	-0.00	0.001859	-0.941	0.361421
1L by 4L	-0.00	0.000929	-0.941	0.361421
2L by 3L	0.00	0.000372	2.286	0.037201
2L by 4L	0.00	0.000186	1.210	0.244870
3L by 4L	0.00	0.000161	2.640	0.018562
[a]insignificant ($P \geq 0.05$)				

The model is decreased to the accompanying structure by eliminating unimportant term (X_2).
$Y = –3154.84 + 70.75 X_1 + 4.97 X_2 + 4.38 X_3 + 18.95 X_4 – 5.00 X_1^2 – 0.13 X_2^2 – 0.08 X_3^2 – 0.03 X_4^2$ (11)

The relapse coefficient estimation of 0.99996 shows that 0.004 % of the absolute varieties are not agreeably clarified by the model. The factual essentialness of the proportion of mean square because of relapse and mean square because of lingering mistake are tried. It is demonstrated from table that, the F-measurements esteem for whole model is higher. i.e., % biosorption of Bromo Cresol Purple can be sufficiently clarified by the model condition. By and large P esteems lower than 0.05 demonstrates that the model is viewed as measurably huge at 95% certainty level. The % biosorption expectation from the model is appeared in table 6. It is suggested from table 6 that all the squared terms of the factors are huge contrasted with the straight terms. Among the communication terms, all the terms ($P < 0.05$) are profoundly critical on biosorption limit [33-34].

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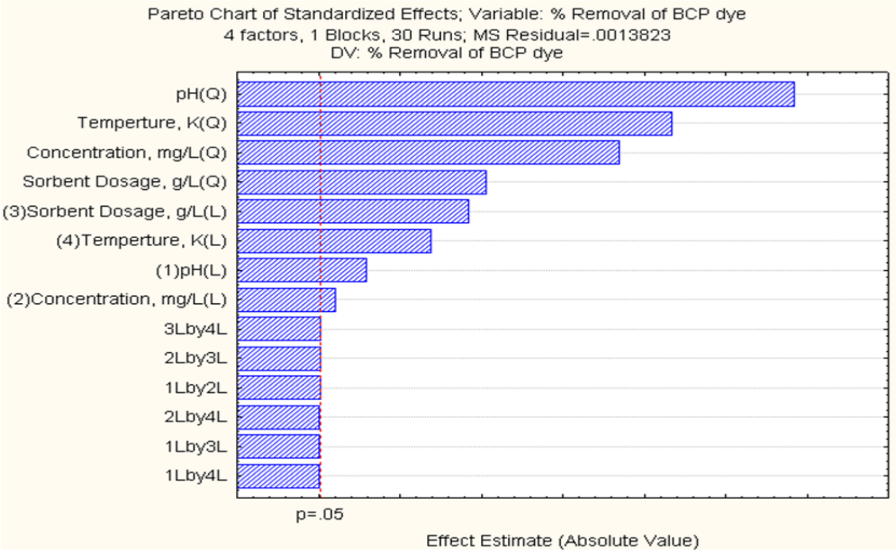

Figure 12. Pareto Chart

Interpretation of residual graphs

Figure 13 shows typical likelihood plot of lingering esteems. The test esteems are in acceptable concurrence with anticipated qualities with least mistake.

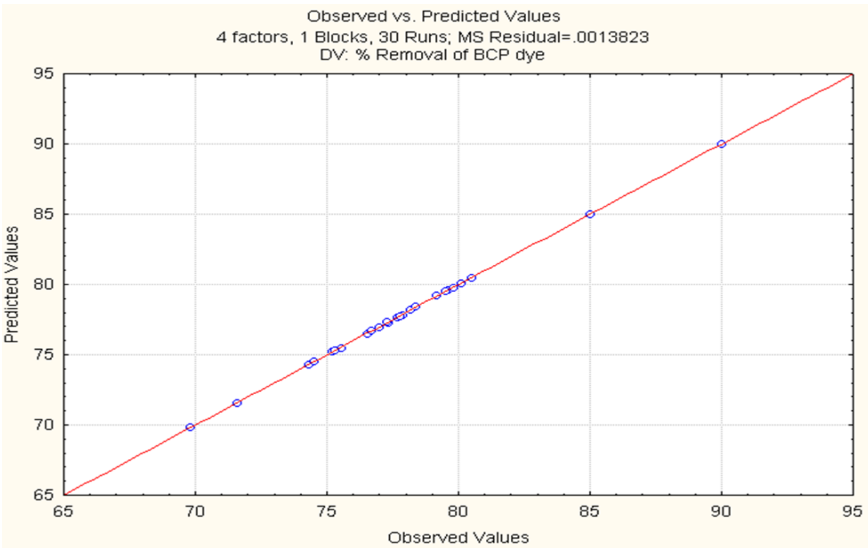

Figure 13. Normal probability plot for % biosorption of Bromo Cresol Purple

Interaction effects of biosorption variables

Figures 14(a) to (f)) portray the three-dimensional perspective on reaction surface plots. The % biosorption of biosorbent is maximal at low and significant levels of the factors however there is where expanding/diminishing pattern in % biosorption isn't noticed.

The anticipated ideal qualities for rate biosorption of Bromo Cresol Purple are:

pH of aqueous solution	= 7.0441,
Initial BCP dye concentration	= 20.1336 mg/L
Biosorbent dosage	= 26.6261 g/L,
Temperature	= 304.7850 K
% biosorption of BCP	= 91.05878

 P-ISSN: 2659-1561
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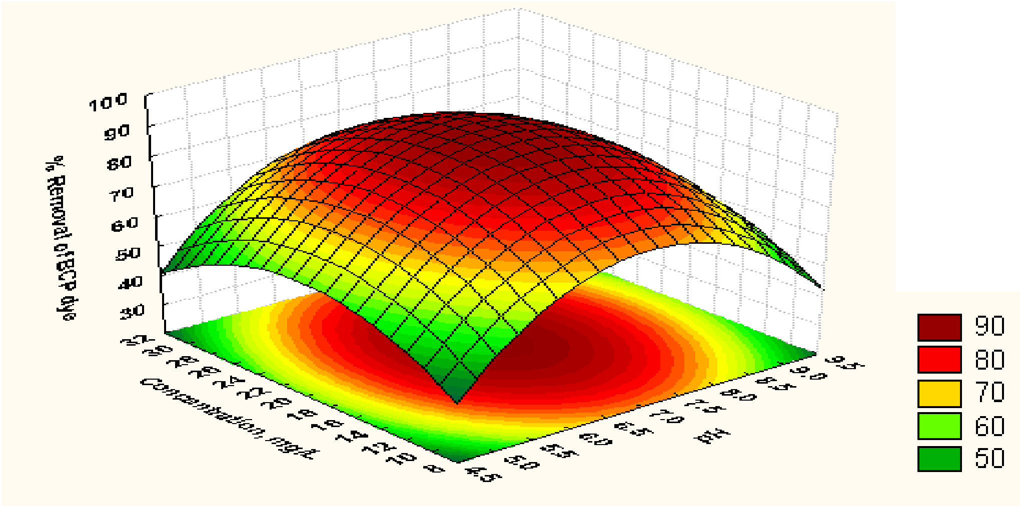

Figure 14(a). Surface contour plot for the effects of pH and initial Bromo Cresol Purple concentration on % biosorption

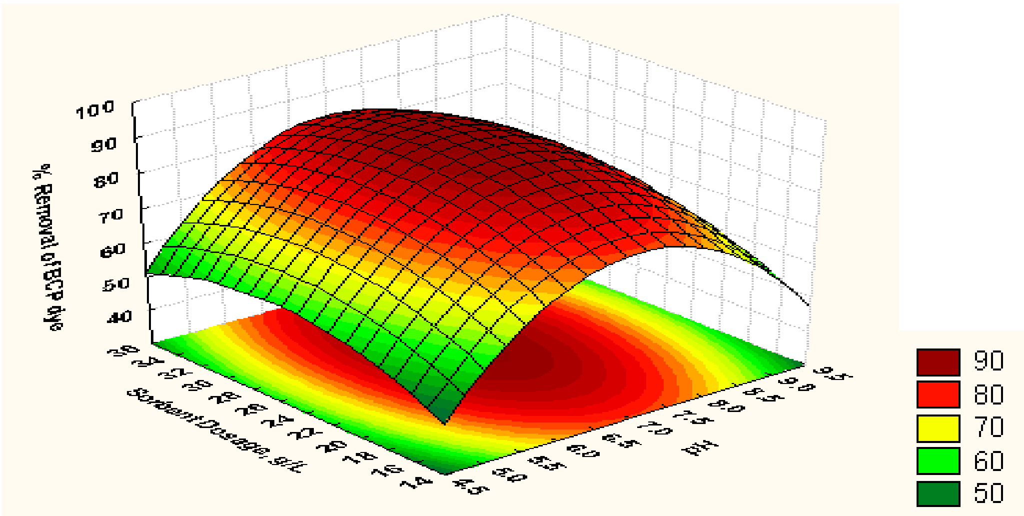

Figure 14(b). Surface contour plot for the effects of pH and dosage on % biosorption of Bromo Cresol Purple

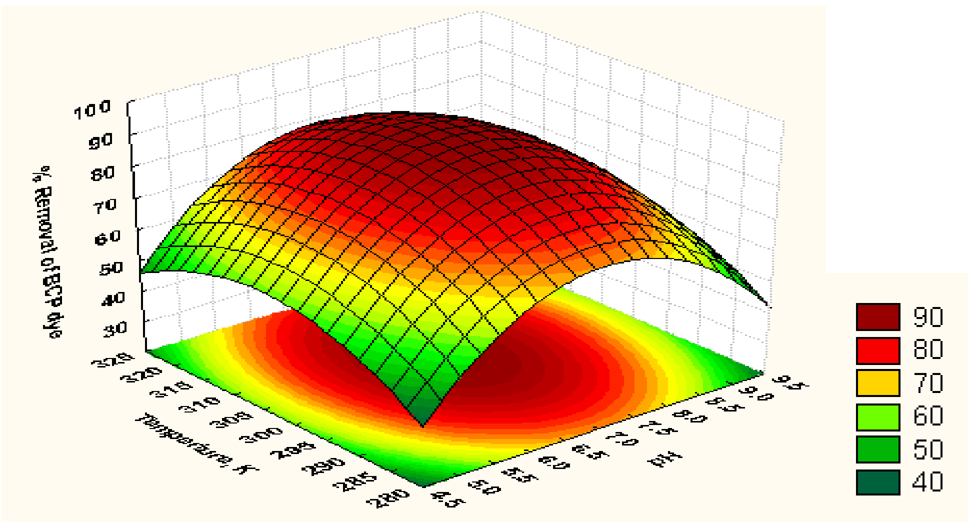

Figure 14 (c). Surface contour plot for the effects of pH and Temperature on % biosorption of Bromo Cresol Purple

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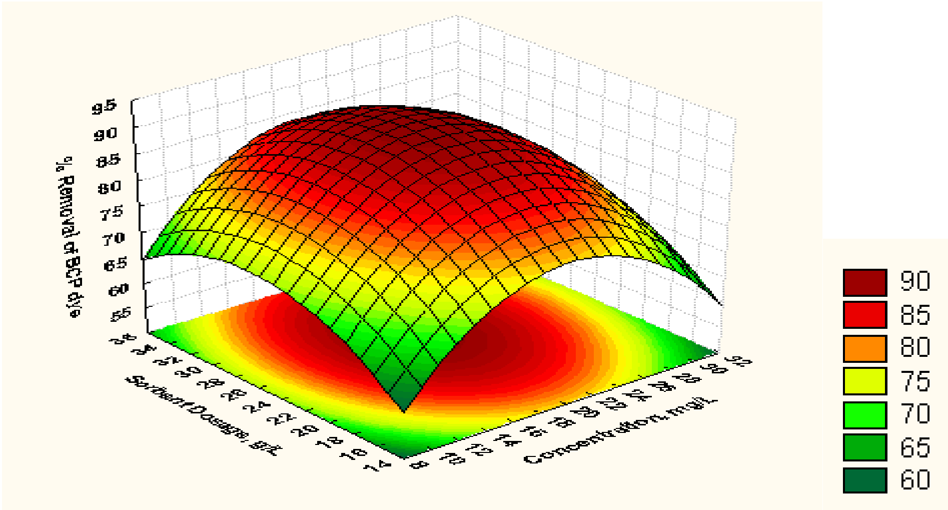

Figure 14(d). Surface contour plot for the effects of initial concentration and dosage on % biosorption of Bromo Cresol Purple

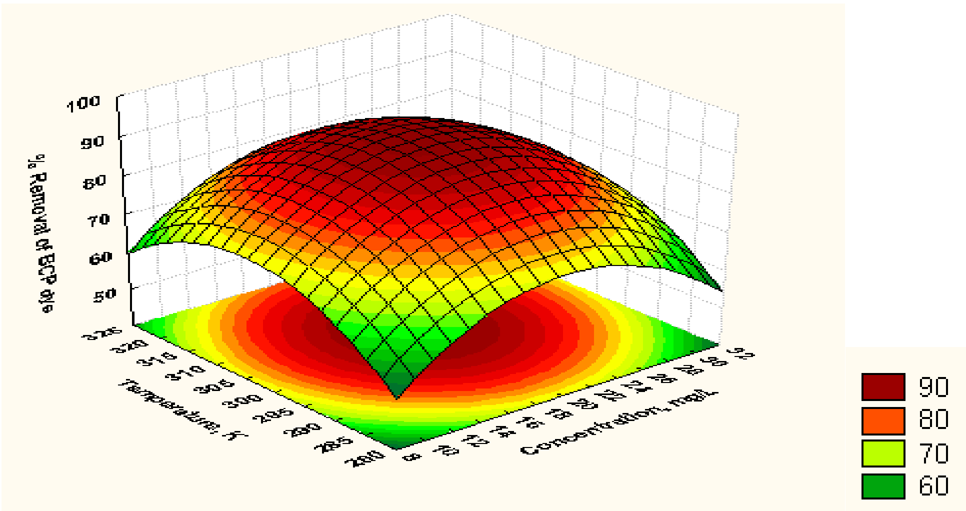

Figure 14 (e). Surface contour plot for the effects of initial concentration and Temperature on % biosorption of Bromo Cresol Purple

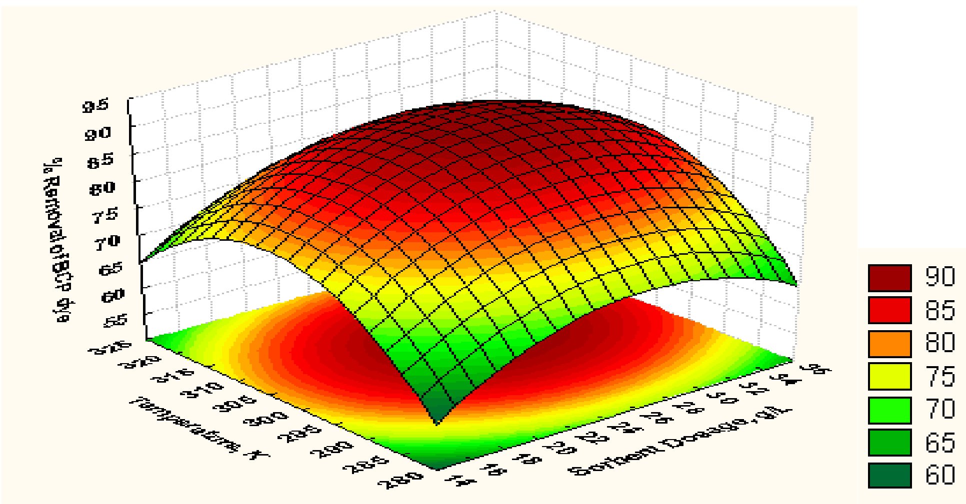

Figure 14(f). Surface contour plot for the effects of Dosage and Temperature on % biosorption of Bromo Cresol Purple

 P-ISSN: 2659-1561
E-ISSN: 2635-3040

Table 7. Comparison between optimum values from CCD and experimentation

	Experimentation	CCD
pH	7.0	7.0441
Initial Concentration (mg/L)	20.00	20.1336
Bisorbent Dosage (g/L)	25	26.6261
Temperature (K)	303	304.7850
% Biosorption		91.05878

Conclusion

The aim of this investigation is to determine the suitability of *Gelidium amansii* red algae powder as sorbent for the removal of BCP dyes from aqueous solutions. The equilibrium, kinetic and thermodynamic studies are carried out for sorption of BCP experimentally. The analysis of the experimental data result in the following conclusions:

1) The equilibrium agitation time for BCP dye sorption is 25 minutes.
2) Percentage sorption of BCP dye from the aqueous solution increases significantly with increase in pH from 2 (40%) to 7 (64%).
3) The optimum dosage for sorption is 25 g/L (1.25 mg/g).
4) The maximum uptake capacity of 15.8478 mg/g is obtained at 303 K.
5) The maximum sorption of BCP dye (91.05878 %) onto *Gelidium amansii* red algae powder is observed when the processing parameters are set as: pH = 7.0441, w = 26.6261 g/L, Co = 20.1336 mg/L and T = 304.7850 K using CCD.
6) The thermodynamic data show that % sorption of BCP dye is increased with increase in temperature. The investigation also reveals the endothermic nature of sorption as ΔH is positive (14.97307), irreversible nature of sorption as ΔS is positive (34.36721) and spontaneity of sorption as indicated by negative ΔG (ΔG = –10398.3 J/mole).
7) It can be concluded from the above results that *Gelidium amansii* red algae powder is capable of removing Bromo Cresol Purple dye.

Conflicts of interest

The authors declare no conflicts of interest.

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Citation: Yagati Vamsi Kumar and Korupolu Raghu Babu. 2020. Sorption of Bromo Cresol Purple Dye with *Gelidium amansii* Red Algae Powder Along With Statistical Optimization. International Journal of Recent Innovations in Academic Research, 4(5): 44-60.