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Process Sorption and Optimization of Bromo Cresol Purple with Gelidium Cartilagineum Powder using Central Composite Design

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Abstract
A Novel dye Bromo Cresol Purple was selected for the present study to evaluate the capacity and capability of Gelidium cartilagineum red algae powder. The characterization of biosorbent powder incorporates the studies on FTIR, XRD and SEM. The parameters studied are agitation time (1 – 180 min), pH (2–8), Initial concentration of BCP dye (20-200 mg/L), biosorbent dosage (10–70 g/L) and temperature (283–323 K). This paper also incorporated the kinetics, isotherms and thermodynamics. Process optimization was carried out using central composite design for the most influenced four parameters. The optimum pH was obtained at 5. The maximum biosorption has occurred at a concentration of 20 mg/L and at a dosage of 35 g/L. The lagergren first order kinetics was well represented and suited for the present experimentation. Among the three isotherms studied Freundlich isotherm suited well with a high correlation coefficient.

Keywords
Bromo Cresol Purple, Biosorption, Gelidium Cartilagineum, Isotherms, Kinetics, Optimization, FTIR, Central Composite Design.

I. Introduction

Water is a prominent and promising barrier for ecological balance. World wide water distribution scenario shows that only 2 % belongs to fresh water and the remaining comprises salt water only **[1,2]**. Among this 2 % fresh water, the distribution is like this: Ice 87 %, Ground water 12 % and Rivers and lakes 1 %. The only remaining trace amounts of potable drinking water is also even being polluted by the industrial activities now a days due to the greedy needs of humans. As the human race is being acquainted with the advantages of technology, the growth of industrial sector has also enhanced, which inturn increased the surface pollution. The pollutants were being settled slowly and polluting the ground water quality **[3]**. This can be stopped and eradicated using different techniques **[4,5,6]**. After surveillance of different methods which are expensive and leaves harmful chemicals, Biosorption has been very promising now a days to treat and solve the above problem **[7,8,9]**. The present experimentation was carried out in order to evaluate the potential and power of red algae powder (*Gelidium Cartilagineum*) for the removal of novel dye Bromo Cresol Purple for the first time.

II. Materials And Methods

The materials and methods consists of the following steps: Reagents and materials, Preparation of the biosorbents and Studies on equilibrium biosorption process.

A. Reagents and materials

All the chemicals used in this investigation were of analytical grade and used without further purification. BCP was used as the source of dye and all the solutions were made with distilled water. The solution of BCP dye was made from a stock solution containing 1000 mg of BCP dye in 1litre. The pH of dye solution was adjusted to the desired value by addition of 0.1M HCL and 0.1M NaOH solutions.

B. Preparation of the Biosorbent:

Gelidium cartilagineum algae was collected from Tenneti Park, Jodugullapalem beach in Visakhapatnam and was washed with water to remove dust and soluble impurities and dried in sun light till the algae became crispy and colorless. The dried algae were finely powdered and sized by passing it through a set of sieves ranging from 300 to 75 mesh sizes. The powder of 53, 75, 105, 125 and 152 micron meters were separated and stored in dry bottles with double cap and used as biosorbent.

C. Studies on equilibrium biosorption process

The biosorption was carried out in a batch process by adding a pre-weighed amount of the Gelidium cartilagineum algae powder to a known volume of aqueous solution for a predetermined time interval in an orbital shaker. The procedures adopted to evaluate the effects of various parameters via. Agitation time, pH, initial concentration, biosorbent dosage and temperature of the aqueous solution on the biosorption of BCP dye were evaluated using single step optimization process. Further optimized and checked using Central Composite Design.

III. Results And Discussion

A. Characterization of Gelidium cartilagineum powder

1. (a) FTIR spectrum of untreated Gelidium cartilagineum powder

FTIR spectrum for treated powder is shown in fig 1(a). a broad band at 573.85, 596.03, 617.25 and 663.54 cm^{-1} is due to the presence of C-Br stretch bands from alkyl halides. The broad absorption peaks at around 758.06 cm^{-1} indicates the presence of Aromatic C–H bending group. The bands at 855.47, 876.68 and 937.44 cm^{-1} are due to the Al–O–H bending bonds. The bands at 1012.67 and 1024.25 cm^{-1} denotes the presence of C–F stretch bands from alkyl halides. The bands at 1037.75, 1053.18 and 1080.18 cm^{-1} are due to the presence of C–F stretch bonds. The band at 1148.66 cm^{-1} suggests the presence of A.romatic C–H bending bond. Similarly the bands at 1211.35, 1233.53, 1242.21, 1319.37, 1339.62 and 1363.73 cm^{-1} are due to the presence of =C–H bending alkenes **[10–16]**.

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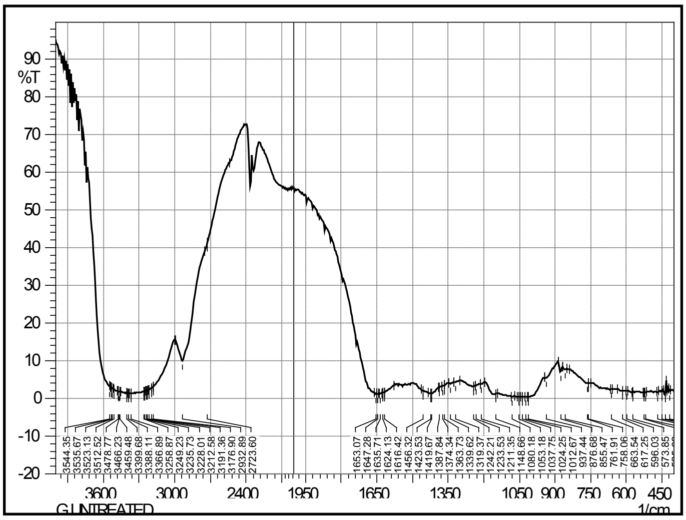

Fig. 1 (a) FTIR spectrum of untreated Gelidium cartilagineum powder

3.1.1 (b) FTIR spectrum of treated Gelidium cartilagineum powder

FTIR spectrum for treated powder is shown in fig 5.17(b). A broad band at 617.25 cm^{-1} suggests the presence of C–Br stretch bands from alkyl halides. The band at 712.73 cm^{-1} is characteristic of Aromatic C–H bending bond. The band at 1495.86 cm^{-1} is due to the presence of Amine N–H stretch group. The bands at 3096.85, 3127.71, 3229.94, 3245.37, 3266.59 and 3287.81 cm^{-1} are the indication for the presence of Amine N–H stretching bonds. The bands at 3299.38, 3355.32,3362.07and3372.68cm^{-1}contain ≡ CH stretch bonds.

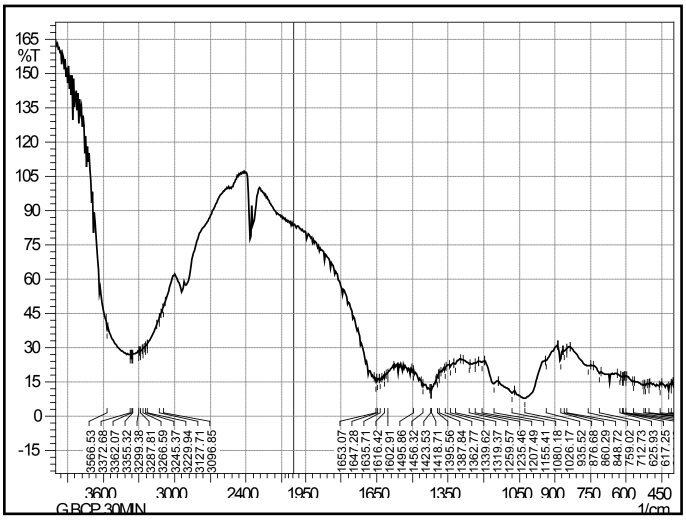

Fig. 1 (b) FTIR spectrum of treated Gelidium cartilagineum powder

The shifts in FTIR peaks are shown in table-1 and in turn confirm that bisorption was achieved.

Table 1 : Shift of FTIR peaks between untreated and BCP dye treated Gelidium cartilagineum powder

Peaks in untreated powder, cm^{-1}	Peaks in treated powder, cm^{-1}	Description
573.85		C–Br stretch bands from alkyl halides
596.03		
617.25	617.25	
663.54	625.93	
758.06	712.73	Aromatic C-H Bending
761.91	759.02	C–Cl stretch alkyl halides
855.47	848.72	Al-O-H bending
876.68	860.29	
937.44	876.68	
1012.67	935.52	
1024.25		C–F stretch bands from alkyl halides
1037.75	1026.17	C-F stretch
1053.18		
1080.18	1080.18	
1148.66	1155.41	Aromatic C-H Bending
1211.35	1207.49	=C–H bend alkenes
1233.53	1235.46	=C–H bend alkenes
1242.21	1259.57	=C–H bend alkenes
1319.37	1319.37	=C–H bend alkenes
1339.62	1339.62	=C–H bend alkenes
1363.73	1362.77	=C–H bend alkenes
1374.34	1387.84	C–N stretch aliphatic amines
1387.84	1395.56	C-O single bond
1419.67	1418.71	S = O and C–S–O bands from ester sulfonate
1423.53	1423.53	C–O stretching from benzene ring
1456.32	1456.32	Amine N-H Stretch
1616.42	1495.86	Amine N-H Stretch
1624.13	1602.91	Alkyl C-H Stretch: Alkane C-H bonds are fairly ubiquitous and therefore usually less useful in determining structure.
1635.71	1616.42	
1647.28	1635.71	
1653.07	1647.28	
2723.60	1653.07	
2932.89	3096.85	Amine N-H Stretch
3176.90	3127.71	
3191.36	3229.94	
3212.58	3245.37	
3228.01	3266.59	
3235.73	3287.81	

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3249.23	3299.38	≡ CH stretch
3258.87	**3355.32**	
3366.89	**3362.07**	
3388.11	**3372.68**	
3399.68		
3459.48		OH stretch
3466.23		
3478.77		
3512.52		
3523.13		
3535.67		
3544.35	3566.53	

3.1.2 X-Ray Diffraction

3.1.2 (a) XRD pattern spectrum of untreated Gelidium cartilagineum powder

X-ray diffractogram of the untreated *Gelidium Cartilagineum* powder is shown in the Fig. 2 (a). From the figure it can be observed that XRD pattern does show amorphous nature.

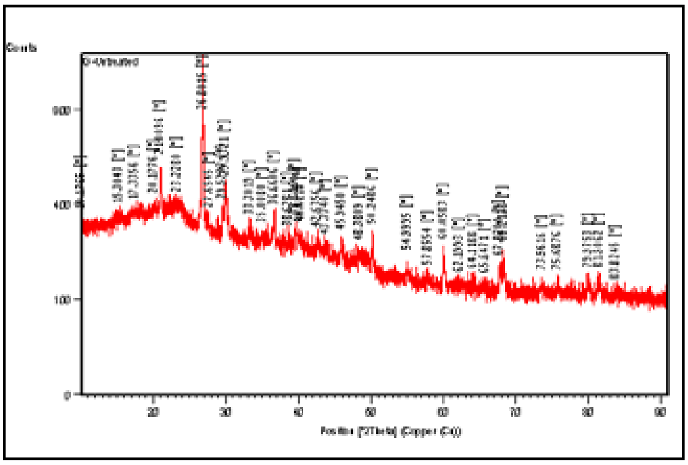


Fig. 2 (a) XRD spectrum of untreated Gelidium cartilagineum powder

3.1.2 (b) XRD pattern spectrum of treated Gelidium cartilagineum powder

X-ray diffractogram of the untreated *Gelidium Cartilagineum* powder is shown in the Fig. 2 (b). From the figure it can be observed that XRD pattern show more amorphous nature and increase in surface area and porosity **[17–23]**.

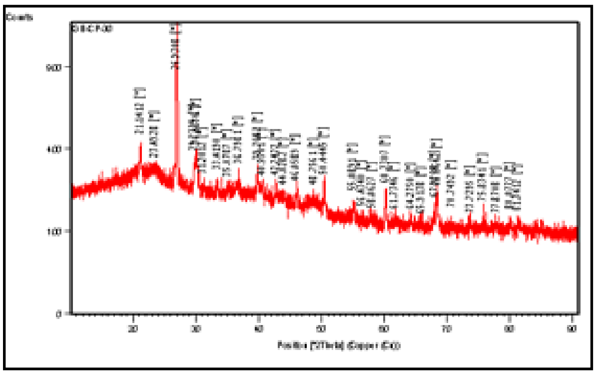


Fig. 2 (b) XRD spectrum of treated Gelidium cartilagineum powder

3.1.3 Scanning Electron Microscope

3.1.3 SEM spectrum of untreated and BCP dye treated Gelidium cartilagineum powder

The micrographs of untreated and treated Gelidium cartilagineum are shown in fig. 3 (a) & (b).

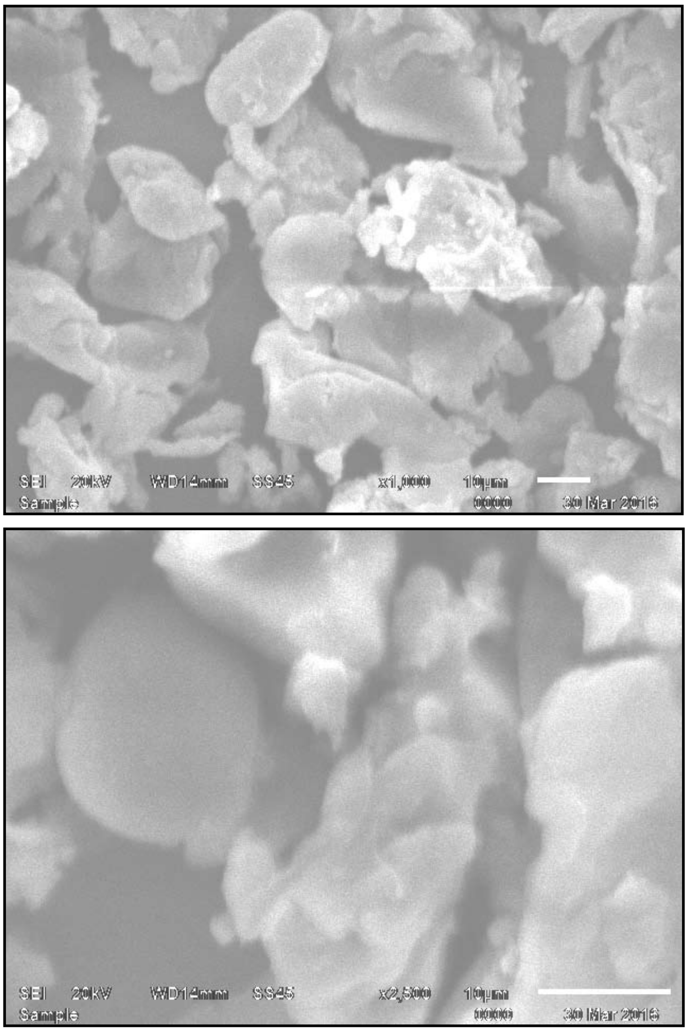
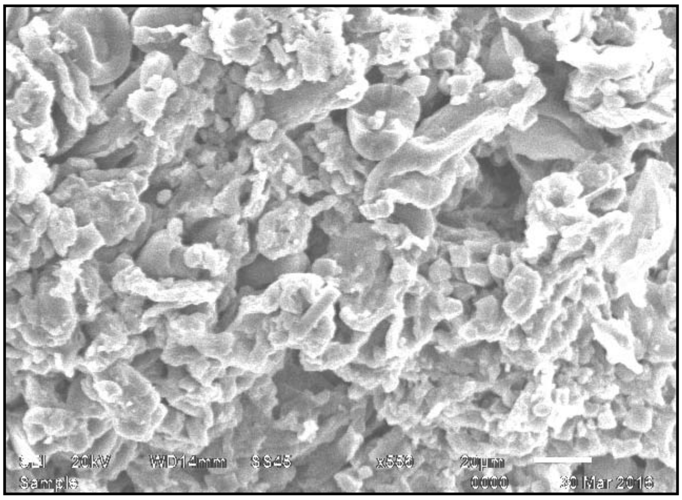


Fig. 3 (a) SEM spectrum of untreated Gelidium cartilagineum powder



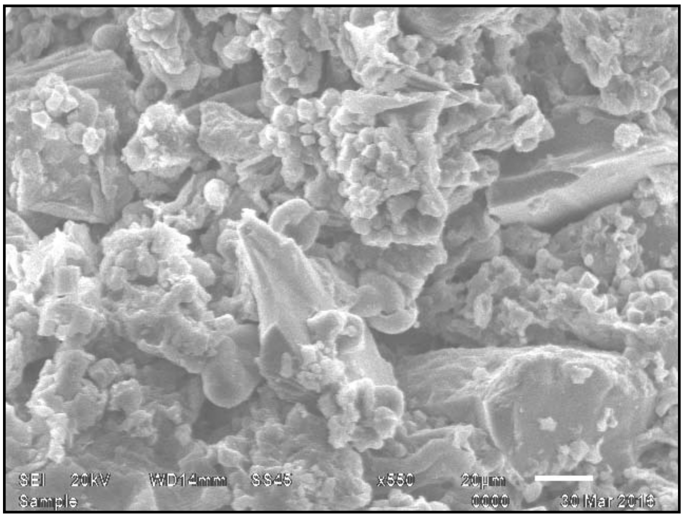


Fig. 3 (b) SEM spectrum of treated Gelidium cartilagineum powder

The micrographs of treated biomass show that the surface has irregular texture with globular, elongated grains and shiny particles over the surface of treated biomass which are absent in the untreated biomass. These elongated grains show that the dye particles are adhered onto the surface of algae. The clustered grains like morphology, on treated biosorbent denote increased active surface area **[24–30]**.

B. Equilibrium studies on biosorption of Bromo Cresol purple

1. Effect of agitation time

In order to determine the biosorption equilibrium time for Bromo Cresol purple ions, the agitation time was varied from 1 to 180 min and the results are shown in Fig. 4.

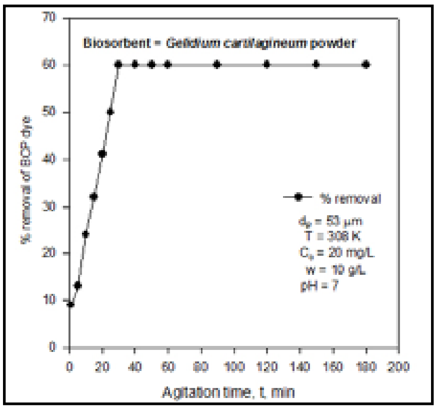


Fig. 4 : Effect of agitation time on biosorption of BCP dye

The % removal of Bromo Cresol purple was initially very fast and then slowly reached equilibrium in 30 min. At the start, the ions adsorbed and occupied selectively the active sites on the biosorbent. As the contact time increased the active sites on the adsorbents were filled **[31–37]**. The rate of adsorption became gradually slower and reached a plateau. The maximum % removal of Bromo Cresol purple at equilibrium time 30 min is 60 %.

2. Effect of pH

To evaluate the effect of pH on the biosorption of BCP dye ions onto Gelidium Cartilagineum, batch biosorption studies were carried out at different pH values ranging at 2, 3, 4, 5, 6, 7 and 8. Fig. 5 presents the effect of BCP dye removal on Gelidium Cartilagineum at different pH values. It is observed that initially when the pH is increased from 2 to 5, the % of BCP dye biosorption increased from 54 to 75 and further increase in pH to 8 resulted in decrease in % BCP dye biosorption to 53. The lower % of biosorption at low pH, is due to the competition between hydrogen and BCP dye ions for the biosorption sites **[38–44]**.

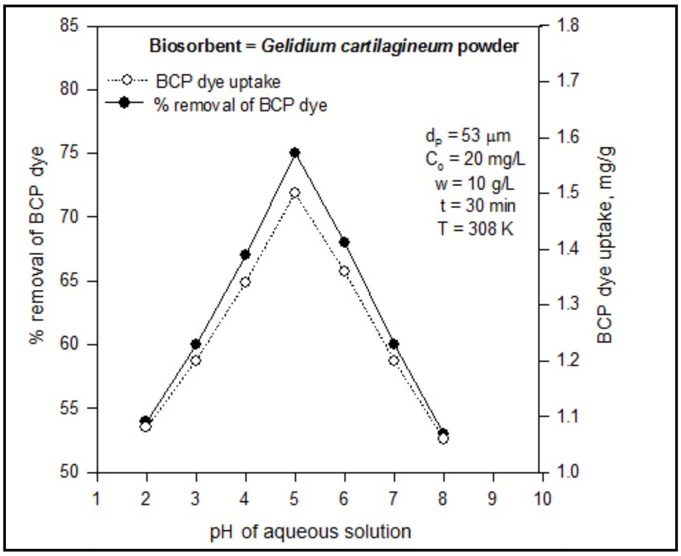


Fig. 5 Effect of pH on biosorption of BCP dye

3. Effect of initial BCP concentration

BCP dye sorption was studied in batch experiments using different initial BCP dye concentrations of 20, 50, 100, 150 and 200 mg/L. The results obtained given in Fig. 6 shows that BCP dye uptake increased and percentage biosorption of the

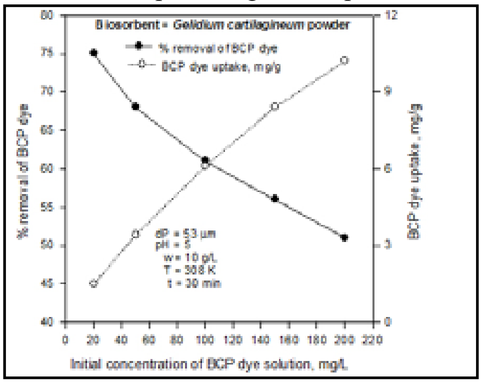


Fig. 6 Effect of initial concentration on % removal of BCP dye

BCP dye decreased with increase in initial BCP dye ion concentration in the studied range. This increase (1.5 to 10.2 mg/g) is probably due to higher interaction between BCP dye ions and the biosorbent. As expected the percentage biosorption of BCP dye ions on Gelidium Cartilagineum decreased from 75 to 51 % due to lack of sufficient active sites so as to accommodate much more dye available in the solution **[45–51]**.

4. Effect of biosorbent dosage

The effect of biomass dosage on the biosorption of BCP dye was studied using different biomass dosage ranging at 10, 20, 30, 40, 50, 60 and 70 g/L was shown in Fig. 7. It was apparent that the removal percentage of BCP dye increased (76 to 92%)

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with increasing biomass dosage due to the greater availability of exchangeable sites for the BCP dye ions, but the dye uptake per gram of biosorbent decreased (1.52 to 0.525714 mg/g). The decrease in the amount of BCP dye adsorbed with increasing biosorbent mass is due to split in the flux or the concentration gradient between solute concentrations in solution and on the biosorbent surface **[52–58]**.

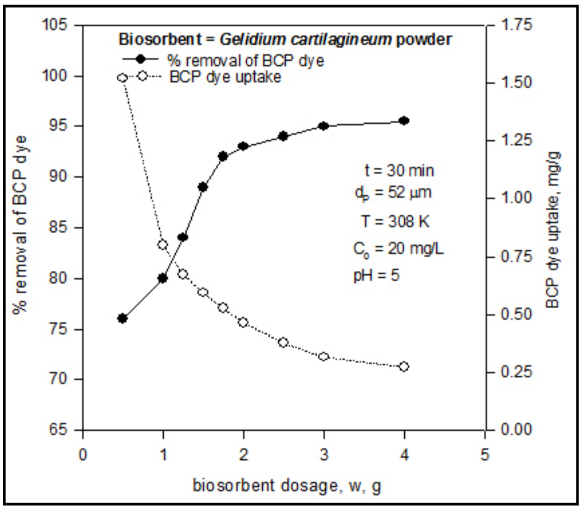

Fig. 7 : Effect of dosage on biosorption of BCP dye

5. Effect of temperature

Temperature dependence of the adsorption process is associated with several thermodynamic parameters.

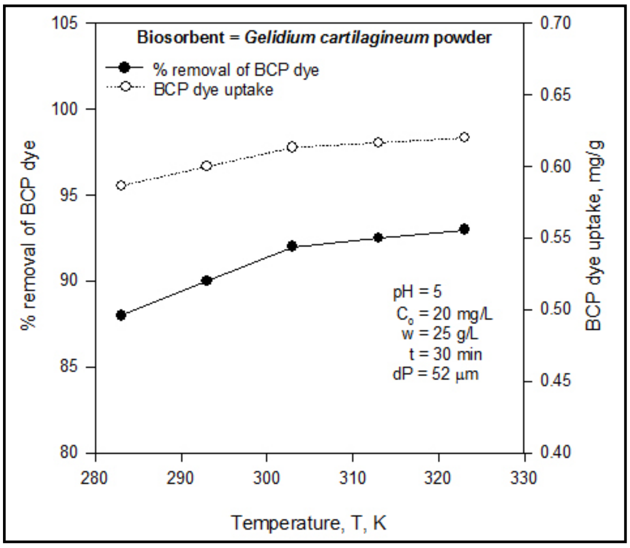

Fig. 8 : Effect of temperature on biosorption of BCP dye

The effect of temperature on the biosorption of BCP dye on the biomass is investigated at different temperatures (283, 293, 303, 313 and 323K) as given in Fig. 8. The increase in the uptake (0.5866 to 0.62 mg/g) with the rise in temperature may be due to the increase in chemical interaction between BCP dye ions and biosorbent surfaces or the increased rate of intraparticle diffusion of biosorbate ions into the pores of the biosorbent at a higher temperature as diffusion is an endothermic process **[59–65]**.

C. Kinetic studies

1. Lagergren-first-order kinetic model

In order to obtain the rate constant, ln (qe −qt) versus time was plotted (Fig.9). The intercept of the above plot should equal to lnqe However, if qe from intercept does not equal the equilibrium BCP dye uptake, then the reaction is not likely to be first-order, even if this plot has high correlation coefficient for the experimental data **[66–72]**.

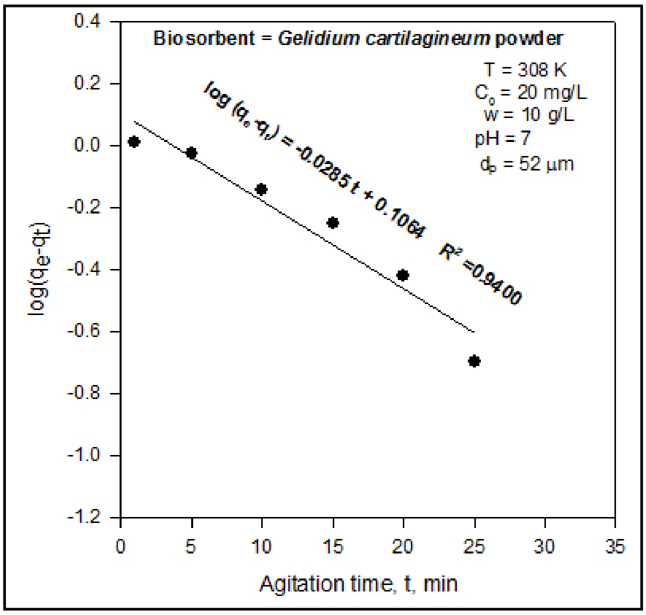

Fig. 9 : First order kinetics for biosorption of BCP dye

2. Pseudo-second-order kinetic model

Attempts were made to test the applicability of pseudo-second-order model. A plot t/qt versus t was shown in Fig. 10. R^2 value of 0.6842 was obtained. qe (1.5082) and k (0.0387) were estimated from this plot. It is observed that pseudo-second-order model also fitted well for the present data next to lagergren first order **[73–79]**.

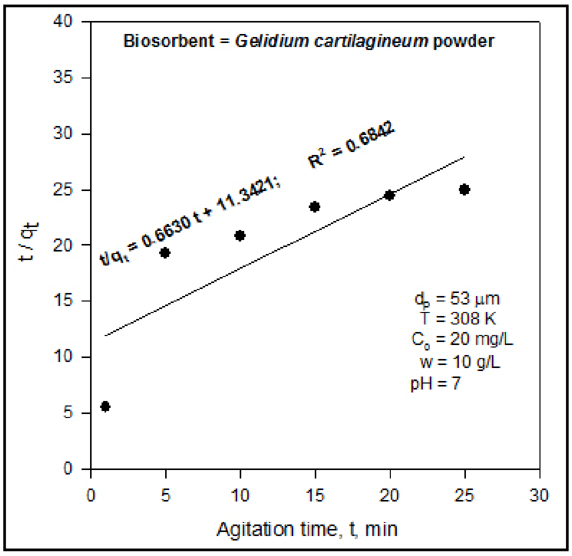

Fig. 10 : Second order kinetics for biosorption of BCP dye

D. Isotherm models

1. Langmuir Isotherm

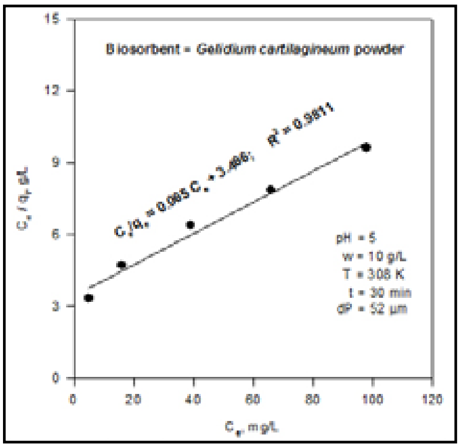

Fig. 11 : Langmuir isotherm for biosorption of BCP dye

Langmuir isotherm is drawn between (Ce/qe) and Ce, the slope {1/ (bqm)} and the intercept (1/b) are calculated for the present data and shown in Fig.11. The equation obtained 'n' Ce/qe = 0.065 Ce + 3.466 with a good linearity (correlation coefficient, R^2~0.9811) indicating strong binding of BCP dye to the surface of Gelidium cartilagineum powder **[80–86]**.

2. Freundlich Isotherm

Freundlich isotherm is drawn between ln Ce and ln qe, yielding equation is: ln qe = 0.6500 ln Ce - 0.6090; for the present data and is depicted in Fig. 12. The yielded equation has a correlation coefficient of 0.9975 **[87–93]**.

The 'n' value (0.6005) calculated from the above equations satisfies the condition of 0<n<1 indicating enthusiastic biosorption.

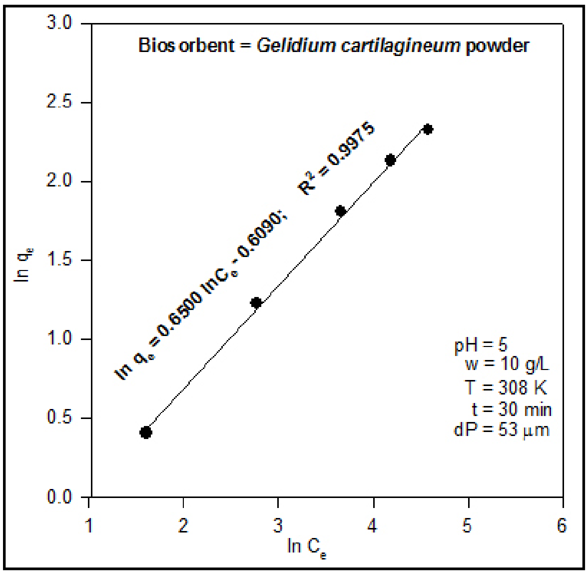

Fig. 12 : Freundlich isotherm for biosorption of BCP dye

3. Temkin Isotherm

Temkin equation is plotted between ln Ce and qe. The present data are analysed according to the linear form of Temkin isotherm and the linear plot is shown in Fig. 13. The equation obtained for BCP biosorption is: qe = 2.9123 ln Ce – 3.877 with a correlation coefficient 0.9578 **[94–100]**.

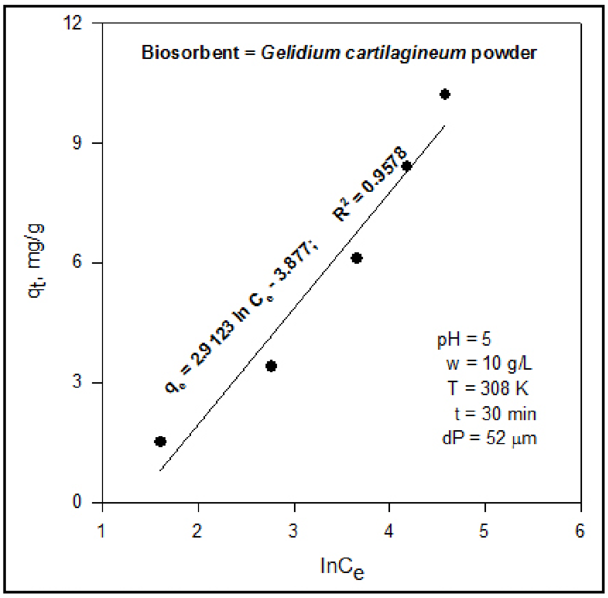

Fig. 13 : Temkin isotherm for biosorption of BCP dye

The best fit model is determined based on the linear regression correlation coefficient (R). From the Figs 11, 12 & 13, it is found that biosorption data are well represented by Freundlich isotherm with higher correlation coefficient of 0.9975, followed by Langmuir and Temkin isotherms with correlation coefficients of 0.9811 and 0.9578 respectively

E. Thermodynamics

1. Vant Hoff's Plot

Experiments are conducted to understand the biosorption behavior varying the temperature from 283 to 323 K. The Vant Hoff's plots indicating the effect of temperature on biosorption of BCP dye is shown in fig. 14. From the obtained slope and intercept, the calculated values procured are as follows: ΔG = -8920.65, ΔH = 11.43084 and ΔS = 29.00026 **[101–107]**.

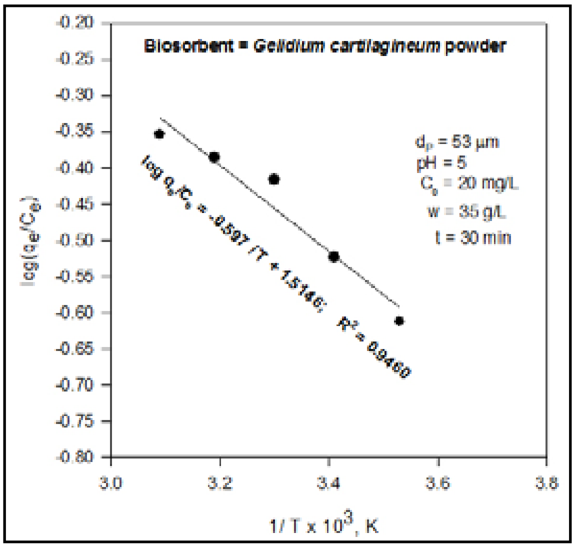

Fig 14 Vant Hoff's plot for % biosorption of BCP dye

F. Optimization using Response Surface Methodology (RSM):

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1. Optimization of the selected parameters using CCD

From the results of preliminary experimental runs, the four variables (pH, initial BCP dye concentration, biosorbent dosage and temperature) have been identified as the potential variables for the percentage biosorption of BCP dye. A summary of the independent variables and their range and levels was presented in Table 2.

Regression equation for the optimization of biosorption is:

% biosorption of BCP dye (Y) is function of pH (X_1), initial concentration (X_2), dosage (X_3), and Temperature (X_4).

Table 2 : Experimental range and levels of the independent variables

Independent variables	Range and level				
	–2	–1	0	+1	+2
pH (X_1)	3	4	5	6	7
Initial BCP dye concentration (X_2),mg/L	10	15	20	25	30
Biosorbent dosage (X_3),g/L	25	30	35	40	45
Temperature (X_4), K	293	298	303	308	313

A 2^4 – factorial central composite experimental design, with eight axial points ($\alpha=\sqrt{4}$) and six replications at the center points (no= 6) leading to a total number of 30 experiments (Table 3) was employed for the optimization of the parameters **[108–115]**.

Table 3 : CCD for BCP dye biosorption by Gelidium Cartilagineum powder

Sl No	pH	Conc, mg/L	Dosage, g/L	Temp, K	% Biosorp
1	4	15	30	293	89.42
2	4	15	30	313	90.62
3	4	15	40	293	90.22
4	4	15	40	313	91.2
5	4	25	30	293	86.58
6	4	25	30	313	88.26
7	4	25	40	293	88.12
8	4	25	40	313	89.48
9	6	15	30	293	89.66
10	6	15	30	313	91.26
11	6	15	40	293	91.28
12	6	15	40	313	92.62
13	6	25	30	293	87.22
14	6	25	30	313	89.22
15	6	25	40	293	89.52
16	6	25	40	313	91.22
17	3	20	35	303	84.92
18	7	20	35	303	86.78
19	5	10	35	303	91.72
20	5	30	35	303	87.38
21	5	20	25	303	90.18
22	5	20	45	303	93.08
23	5	20	35	283	90.32
24	5	20	35	323	93.32
25	5	20	35	303	95.22
26	5	20	35	303	95.22
27	5	20	35	303	95.22
28	5	20	35	303	95.22
29	5	20	35	303	95.22
30	5	20	35	303	95.22

Multiple regression analysis of the experimental data resulted in the following equation for the biosorption of BCP dye:

$$Y = -806.482 + 19.458\,X_1 + 1.112\,X_2 + 2.727\,X_3 + 5.157\,X_4 - 2.337\,X_1^2 - 0.056\,X_2^2 - 0.036\,X_3^2 - 0.008\,X_4^2 + 0.017\,X_1X_2 + 0.039\,X_1X_3 + 0.009\,X_1X_4 + 0.007\,X_2X_3 + 0.002\,X_2X_4 - 0.001\,X_3X_4$$

The parity plot (Fig. 15) showed a satisfactory correlation between the experimental and predicted values of percentage removal of BCP dye indicating good agreement of model data with the experimental data.

The maximum percentage biosorption of BCP dye is indicated by the surface confined in the smallest curve (circular or elliptical) of the contour plot.

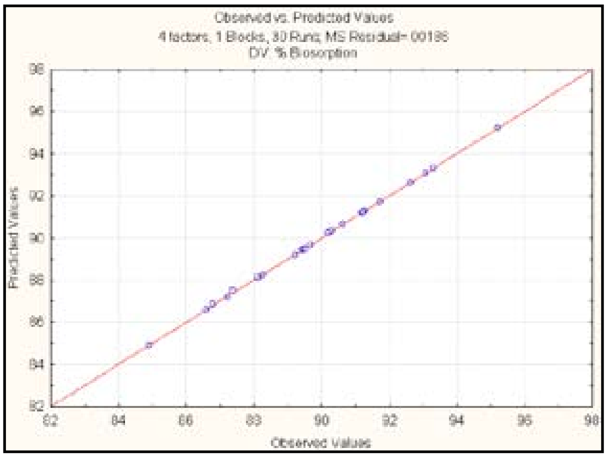
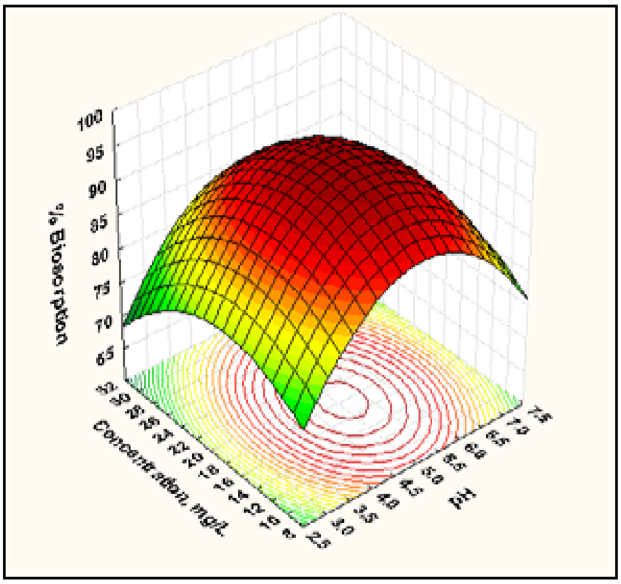


Fig. 15 : Parity plot showing the distribution of experimental vs. predicted values of percentage biosorption of BCP dye

The optimal set of conditions for maximum percentage biosorption of BCP dye is pH = 5.1225, initial BCP dye concentration = 18.3335 mg/L, biosorbent dosage = 36.8407 g/L, and temperature = 307.1192 K. The extent of biosorption of BCP dye at these optimum conditions was 95.71146 %. It is evident that experimental values of % biosorption are in close agreement with that of predicted by Central Composite Design. Experiments are conducted in triplicate with the above predicted optimal set of conditions and the % biosorption of BCP dye is 93 %, which is closer to the predicted % biosorption.



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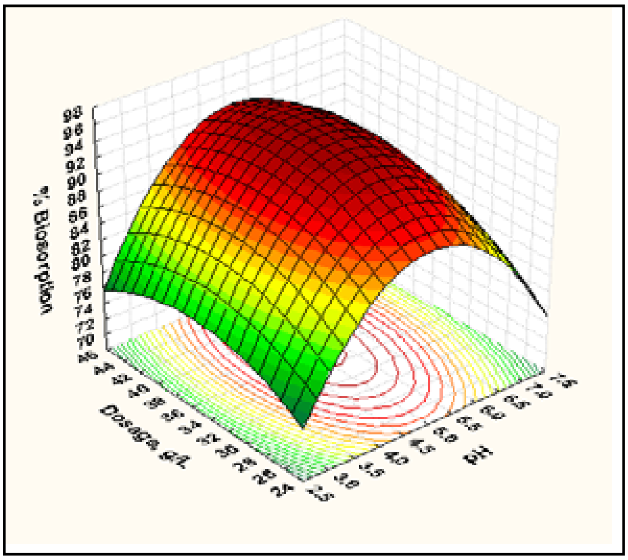
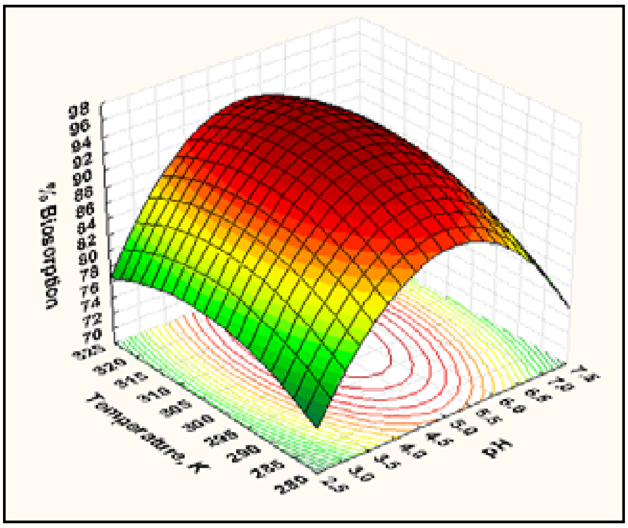


(b)



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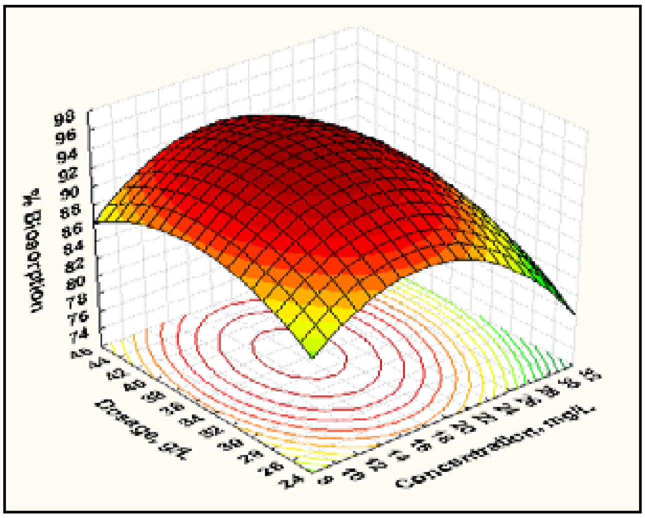
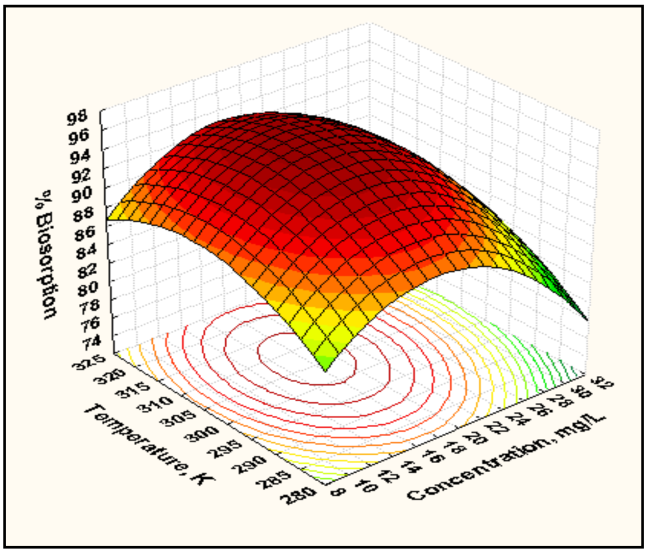
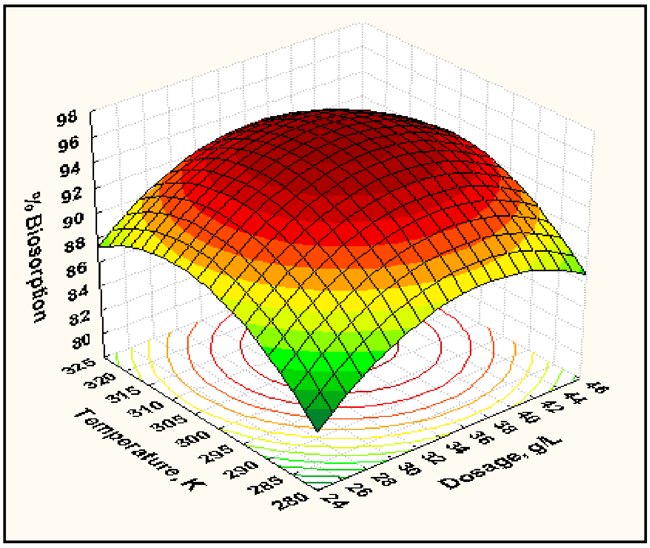


(d)



(e)



(f)

Figs. 16 (a) to (f) represents the Surface Contour plots for the optimization of % biosorption of BCP dye

Table-4 presents the comparison of dye uptake capacities of various biosorbents with those of present investigation.

Table : 4 Dye uptakes for different biosorbents

Author	Biosorbent	qt, mg/g
Aseel M. Aljeboree et al **[116]**	coconut shell activated carbon	58.5
Sheikha S. Ashour **[117]**	steam-activated carbons developed from date pits	42.1
N. Rajamohan **[118]**	activated water hyacinth roots	13.46
P. N. Palanisamy et al **[119]**	Activated carbon from Euphorbia tirucalli L wood	181.81
N. Rajamohan et al **[120]**	activated plant biomass	112.34
G.Vijayakumar et al **[121]**	natural adsorbent perlite	60.976
Present investigation	Gelidium Cartilagineum	15.1048

IV. Conclusions

The aim of this investigation is to determine the capability and capacity of Gelidium Cartilagineum powder performance for the removal of Bromo Cresol Purple dye and yielded the following conclusions. The equilibrium agitation time for BCP dye biosorption is 30 min. With an increase in the initial concentration of BCP dye (20 to 200 mg/L) in the aqueous solution, the percentage biosorption of BCP dye from the aqueous solution is decreased (75 to 51 %). Optimum pH value is obtained at 5. The CCD optimized conditions are: w = 36.8407 g/L, pH = 5.1225, Co = 18.3335 mg/L and T = 307.1192 with % biosorption of 95.71146%. The biosorption of BCP dye is better described by lagergren first order kinetics (R^2 = 0.94). The experimental data are well represented by Freundlich isotherm (R^2 = 0.9975). The thermodynamic investigation reveals: the endothermic nature of biosorption (ΔH = 11.43084 J/mole), irreversibility of biosorption (ΔS = 29.0006 J/mole-K) and increased randomness at the solid/solution interface and the spontaneity and feasibility of biosorption (ΔG = –8920.65 J/mole). Hence the above said Gelidium Cartilagineum powder is highly effective and efficient biosorbent and is capable of removing novel Bromo Cresol Purple dye.

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