

Studies on Biosorption of Crystal Violet Dye with *Jania Rubens*

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Abstract

*In this study dried powdered of *Jania rubens*, component were used for the removal of Crystal violet dye from aqueous solution by using a new biosorbent in a batch biosorption technique. The characterization of the biosorbent was performed by using FTIR and XRD techniques. The parameters investigated includes, agitation time, biosorbent size, pH, initial concentration of dye, dosage of biosorbent and temperature. The Kinetic study incorporated Lagergren first order and pseudo second order models. The study also included thermodynamics and isotherms like Langmuir, Freundlich and Temkin. The experimental data was correlated for regression analysis and the data was very well fitted.*

Keywords - Crystal violet dye, *Jania rubens*, RSM.

I. INTRODUCTION

The crystal violet (CV) dye is a synthetic cationic dye and transmits violet color in aqueous solution. It is also known as Basic Violet 3, gentian violet and methyl violet 10B, belonging to the group of triarylmethane [1]. This dye is used extensively in the textile industries for dyeing cotton, wool, silk, nylon, in manufacture of printing inks and also the biological stain, a dermatological agent in veterinary medicine [1-2]. The CV is toxic and may be absorbed through the skin causing irritation and is harmful by inhalation and ingestion. In extreme cases, can lead to kind CV failure, severe CV irritation leading to permanent blindness and cancer [3-4]. Therefore, removal of this dye from water and wastewater is of great importance.

Various methods of treatment exploited through the years by industries for removing colorants include physicochemical, chemical, and biological methods, such as flocculation, coagulation, precipitation, adsorption, membrane filtration, electrochemical techniques, ozonation, and fungal decolorization [5]. However, due to the fact that effluents contain different dyes, and these dyes contain complex structures, is very difficult to treat using conventional methods [6].

Jania rubens, the slender-beaded coral weed, is a species of red seaweeds. “*Jania*” comes from the latin « Janus », double-head God from Roman mythology, watchman of Gods house. “*Rubens*” means of red colour. This alga is named like this because of its dichotomous ramifications and its colour. *Jania*

Rubens is a calcified red alga from 15 to 40 mm high, and has a rose-red colouration, although in strongly illuminated areas this can be slightly yellowish white. It grows slender rose-pink fronds that form rounded bunches reaching 5 cm long. *Jania rubens* has a thallus formed from cylindrical filaments; the fronds are erect and jointed with particularly thin branches. The ramification is dichotomous with the branches tapering near the end; swellings found at the joints are the reproductive bodies. Textile industries consume large volumes of water and chemicals for the wet processing of textiles. The presence of very low concentrations of dyes in effluent discharged from these industries is highly visible and undesirable [7]. Due to their chemical structure, dyes are resistant to fading when exposed to light, water and chemicals [8, 9]. Dyes, usually, have a synthetic origin and complex aromatic molecular structures, which make them more stable and difficult to biodegrade.

Dyes can be classified as follows [10]:

- Anionic: direct, acid, and reactive dyes.
- Cationic: basic dyes.
- Non-ionic: disperse dyes.

Brightly colored, water-soluble, reactive, acidic dyes are the most problematic, as thCV tend to pass through conventional treatment systems unaffected [11]. Various physical, chemical and biological methods have been used for the treatment of dye containing wastewater. Some Chemical oxidations, such as Fenton reagent, ozone, UV plus H₂O₂ or NaOCl, result in aromatic ring cleavage, which may generate chemical sludge or by-products that are likely to be even more toxic [12]. Aerobic biological treatment is known to be ineffective for dye removal [12], but anaerobic bioremediation enables water soluble dyes to be decolorized [13]. Although ion exchange resins can be regenerated easily, the high cost hinders their wide application for the treatment of dye-bearing wastewater. Consequently, various types of (bio) sorbents, which are able to bind dye molecules and be easily regenerated, have been extensively searched and tested [14,15]. A suitable sorbent has to meet the following criteria [16]:

- (1) high affinity and capacity for target compounds,
- (2) regeneration possible,
- (3) safe and economically viable treatment/disposal of regenerate,
- (4) tolerance for a wide range of wastewater parameters, and

(5) usable for all or nearly all-reactive dyes. Due to their cost-effectiveness, biosorbents have gained much attention. However, most are non-regeneratable throwaway products, such as bagasse pith [17], eucalyptus bark [18], and so on. Good sorption capacities for reactive dyes (60–420 mg g⁻¹) were found for quaternized organic materials, such as cellulose [19], sugarcane bagasse [20], rice husk [21] and coconut husk [22] however, no successful regeneration has been reported here either.

II. EXPERIMENTAL PROCEDURE

The present experimentation is carried out both batch-wise and column, on biosorption of crystal violet dye from aqueous solutions on the biosorbent –Jania Rubens powder. The experimental procedure consists of the following steps:

- 2.1 Preparation of the biosorbent
- 2.2 Characterization of biosorbent
- 2.3 Preparation of the stock solutions
- 2.4 Studies on Equilibrium Biosorption Process

A. Preparation of the biosorbent

Jania Rubens was collected from intertidal region of the Visakhapatnam coast. The collected biosorbent was washed with water several times until the dirt particles are removed and finally washed with distilled water. The biosorbent was dried in sun light for fifteen days, cut into small pieces, powdered and sieved. In the present study, the obtained powder was used as biosorbent without any pretreatment.

B. Characterization of biosorbent

Biosorption of crystal violet using Jania Rubens has many affecting factors which include characterization (FTIR, XRD, SEM), Biosorbents were characterized by FTIR spectrometry using Spectrum GX of Perkin Elmer, XRD patterns were recorded from 10 to 700 For SEM studies, the dried powders and the corresponding loaded powders were first coated with ultra-thin film of gold by an ion sputter JFC-1100 and then were exposed under a Japanese make electron microscope (JEOL, JXA8100) equilibrium studies (agitation time, biosorbent size, pH, initial concentration, biosorbent dosage, temperature), Isotherms (Langmuir, Freundlich, Temkin), Kinetics (Lagergren First Order, Pseudo Second Order), Thermodynamics (Entropy, Enthalpy and Gibb’s Free Energy) and Optimization using Central Composite Design. XRD patterns were recorded from 10 to 700.

C. Preparation of stock solution:

The standard stock solution of crystal violet dye (1000 mg/L) was prepared by dissolving 1.0 g of 99.9 % analytical grade. Crystal violet dye in 1000 mL of distilled water. The concentration of dye in the aqueous solution was varied from 20 to 200 mg/L by diluting the stock solutions with required quantity of

deionized water. The pH of the working solution was adjusted using either 0.1 N HCl or 0.1N NaOH.

2.3 Studies on Equilibrium Biosorption Process:

The biosorption was carried out in a batch process by adding a pre-weighed amount of the Jania Rubens 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, biosorbent size, pH, initial concentration, biosorbent dosage and temperature of the aqueous solution on the biosorption of Indigo carmine dye were evaluated using single step optimization process.

Table I

Experimental conditions for biosorption of crystal violet dye

S.No	Parameter	Values Investigated
1.	Agitation time, t, min	5, 10, 15, 20, 25, 30, 30, 50, 60, 90, 120, 150 and 180
2.	pH of the aqueous solution	2, 3, 3, 5, 6, 7 and 8
3.	Initial dye concentration, C ₀ , mg/L	20, 50, 100, 150 and 200
4.	Initial Biosorbent dosage, w, g/L	0.5,1,1.25,1.5,1.75,2,2.5,3,4

III. RESULTS AND DISCUSSIONS

A. Effect of agitation time

The below discussions are about the effects of various parameters on the CV dye by Jania Rubens biosorption. The experiment is carried out by varying time from 1 to 180 minutes. The fig. 3.1 shows the agitation time against the % biosorption and it is found to increase up to 40 min. At 60 min of agitation the max 75 % of biosorption is attained and becomes constant after 60min by indicating the attainment of equilibrium (75%)

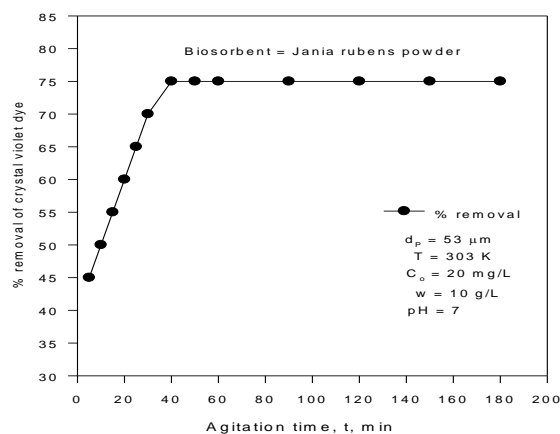


Fig 3.1. Effect of time on % biosorption of CV dye

B. Effect of biosorbent size

Fig 3.2 indicates the function of the particle size and % biosorption of CV dye by Jania Rubens. The biosorbent size is increased from 53 to 152 μm and its percentage is decreased from 75% to 50%. As the surface area of the biosorbent enhances and extra number of active sites on the biosorbent are available to the biosorbate and the size of the particle decreases

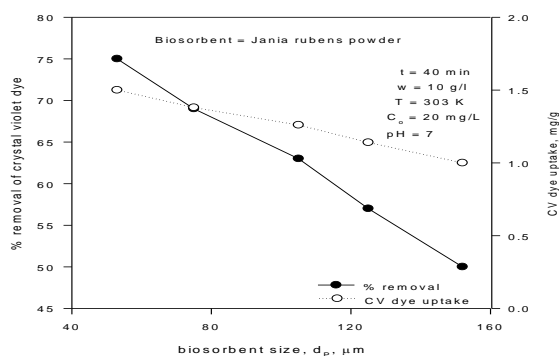


Fig 3.2. Effect of size on % biosorption of CV dye

C. Effect of pH

The fig 3.3 is drawn to show the effect of pH of aqueous solution on percentage biosorption of CV dye by Jania rubens powder. As pH is increased from 2 to 5 and the % biosorption is increased from 67 to 82% as well as the pH is decreased from 5 to 8 and the % biosorption is decreased from 82 to 70%. The electro static interaction between biosorbent and biosorbate is the principle is the driving force for dye biosorption. As the interaction because the greater then biosorption of dye will be more. For forming part of the surface functional group, the CV dye by Jania rubens powder replace H⁺ ions bound to the biosorbent with an interaction. Participation of =C-H of alkene or arena and symmetric -SO₃ stretching in which the predominant contributors in dye uptake are symmetric bending of CH₃ group. C≡N presenting in the poly acrylo nitrile and thio cyanate (-SCN) which are directly involved in the biosorption and further followed by thio cyanate, iso thio cyanate, diazo in the aromatic combination groups. Hence the conclusions were reported when dye ions were adsorbed in the case of orange peel

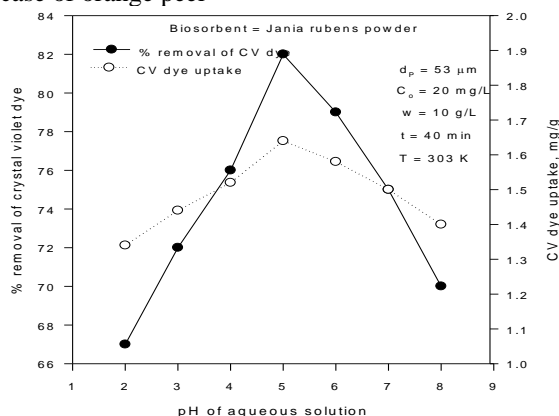


Fig 3.3. Effect of pH on % biosorption of CV dye

D. Effect of initial concentration of CV dye by Jania rubens powder

The below graph shown the function of initial concentration of CV dye by Jania rubens powder with the % biosorption of CV dye by Jania rubens powder and it is decreased from 82% to 55% as the initial concentration CV dye by Jania rubens powder while increasing aqueous solution from 20 to 200 mg/L. The uninterrupted number of freely active sites on the biosorbent can be attributed to the increase in the amount of biosorbate is the behavior observe fig 3.4

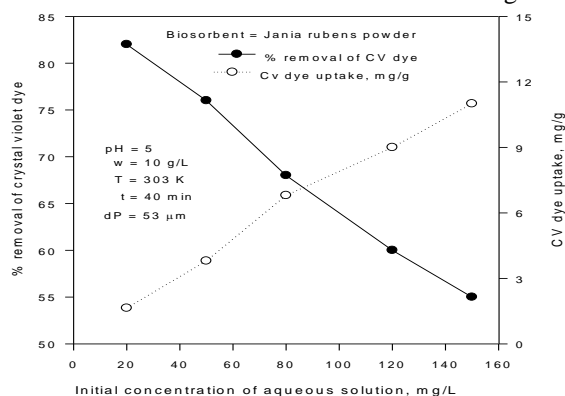


Fig 3.4 Effect of initial concentration on % biosorption of CV dye

E. Effect of biosorbent dosage

The fig 3.5 is drawn against biosorbent dosage and the % biosorption of CV dye by Jania rubens powder. The increase in biosorbent dosage with the increase in % biosorption. As dosage is increased from 0.5 to 4 gm/L, the % biosorption increases from 82 to 94.5% for a biosorbent size of 53 μm. For dye removal of the number of available sites where this behavior is obvious and it would be more as the amount of biosorbent increases

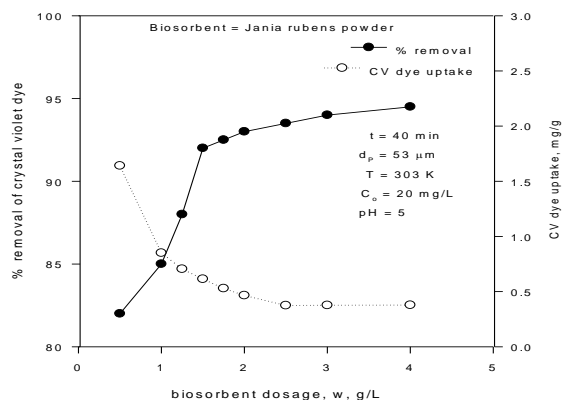


Fig 3.5. Effect of dosage on % biosorption of CV dye

F. Effect of temperature

On the equilibrium dye uptake was significant on the effect of temperature. The fig 3.6 shows the effect of changes in temperature on the CV dye by Jania rubens powder uptake. This system is an endothermic process which indicates that the biosorption of dyes and its capacity increased at higher temperatures. The experiment is carried out by varying temperature

from 283 to 323K. The creation of new active sites or this may be attributed at higher temperatures to increase penetration of reactive dyes inside micro pores. At higher temperature in the case of CV dye by jania rubens powder to be achieved the formation of more than 1 molecular layer on the surface of jania rubens powder

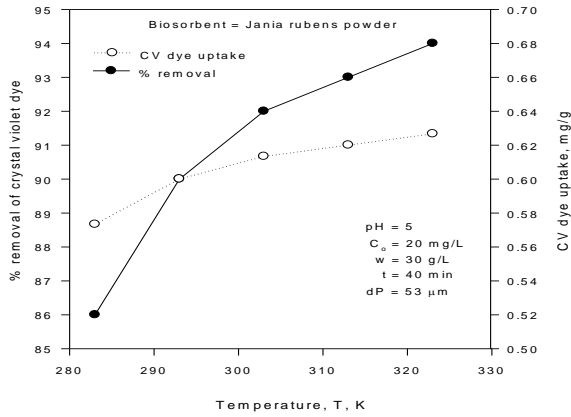


Fig 3.6. Effect of temperature on % biosorption of CV dye

G. Isotherms

1. Langmuir Isotherm:

Langmuir isotherm, drawn in fig. 3.7, for the present data has yielded the equation:

$$(C_e / q_e) = 0.06869 C_e + 2.2666$$

$$R^2 = 0.9870 \text{ ----- (1)}$$

The correlation coefficient value of 0.9870 indicates strong binding of CV dye on to the biosorbent

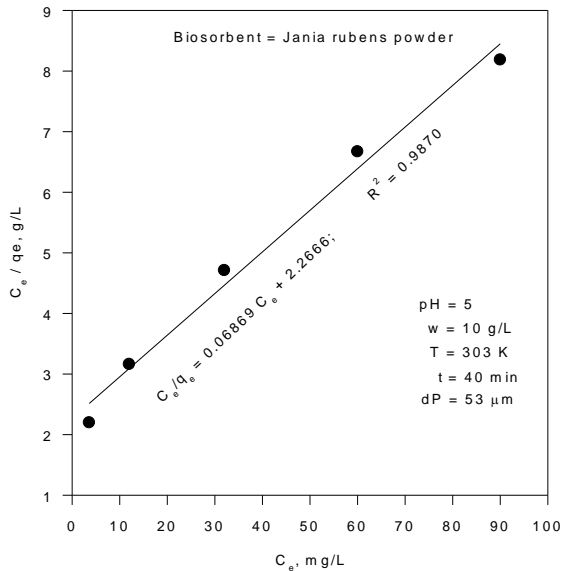


Fig. 3.7 Langmuir isotherm for biosorption of CV dye

2. Freundlich Isotherm:

Fig. 3.8, drawn between ln C_e and ln q_e, has resulted the equation:

$$\ln q_e = 0.5895 \ln C_e - 0.1975 \text{ ----- (2)}$$

The equation has a correlation coefficient of 0.9926. The 'n' value of 0.5895 satisfies the condition of 0 < n

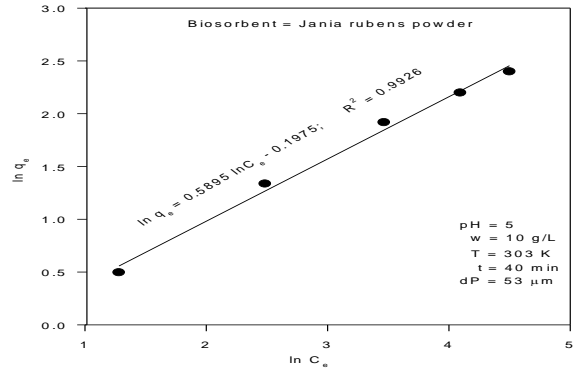


Fig. 3.8 Freundlich isotherm for biosorption of CV dye

3. Temkin Isotherm:

The present data are analysed according to the linear form. The linear plot of Temkin isotherm is shown in fig. 3.9. The equation obtained for CV dye biosorption is:

$$q_e = 0.3377 \ln C_e + 0.9876 \text{ ----- (3)}$$

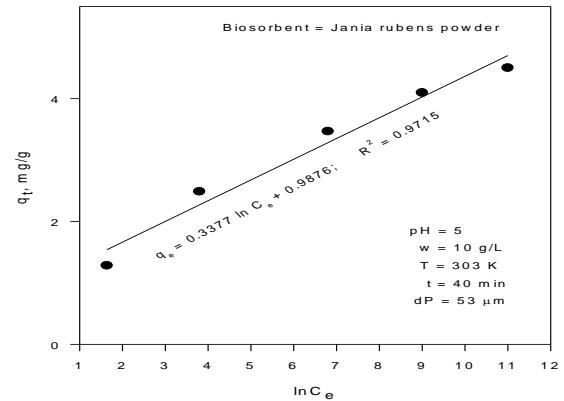


Fig. 3.9 Temkin isotherm for biosorption of CV dye

With a correlation coefficient 0.9715. The isotherm constants of the three isotherms are compiled in table-II. The equilibrium data are well explained by Langmuir isotherm (0.9870), Temkin (0.9926) and Freundlich isotherm (0.9715)

Table II

Isotherm constants (linear method)

Langmuir isotherm	Freundlich isotherm	Temkin isotherm
qm= 14.55816mg/g	Kf= 0.82078mg/g	AT= 18.62471L/mg
KL = 0.030305	n = 0.5895	bT = 7459.704
R ² = 0.9870	R ² = 0.9926	R ² = 0.9715

Kinetics of biosorption

The prediction of biosorption rate gives important information for designing batch biosorption systems. Information on the kinetics of solute uptake is required for selecting optimum operating conditions for full-scale batch process. The kinetics of the biosorption data was analysed using two kinetic models, lagergren-first-order and pseudo-second-order. These models correlate solute uptake, which

are important in predicting the reactor volume. The experimental data are tested for Lagergren first order rate equation and pseudo second order rate equation. Lagergren plot of $\log(q_e - qt)$ vs agitation time (t) is shown in fig. 3.10.

$$\log(q_e - qt) = -0.03397 t + 0.09542, \quad R^2 = 0.9743 \dots\dots\dots (4)$$

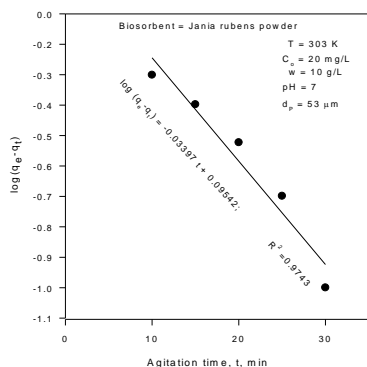


Fig. 3.10 First order kinetics for biosorption of CV dye

a) Pseudo second order rate equation

Pseudo second order kinetics plot between 't' vs 't/qt' for biosorption of CV dye is drawn in fig. 3.11. summarizes rate constant values for first and second order rate equations. It is noted that both first and second order rate equations explain the biosorption interactions.

$$t/qt = 0.6290 t + 3.4108, \quad R^2 = 0.9834 \dots\dots\dots (5)$$

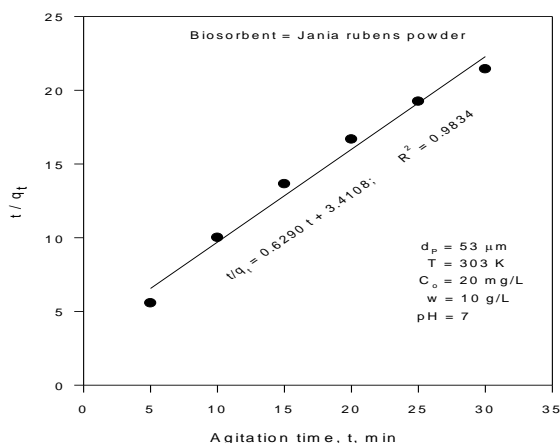


Fig. 3.11 Second order Kinetics for biosorption of CV dye

H. Thermodynamics:

A series of thermodynamic parameters - change in Gibbs free energy (ΔG) change in enthalpy (ΔH) and change in entropy (ΔS) are determined. ΔG value of -14707.7 J/mole indicates that biosorption of CV dye by Jania rubens powder could take place spontaneously. Higher temperatures have benefitted biosorption and increased the equilibrium biosorption capacity. Positive ΔH of 17.31285 J/mole indicates endothermic nature of biosorption while

positive $\Delta S = 49.59736$ J/mole-K demonstrates the affinity of Jania rubens powder to CV dye

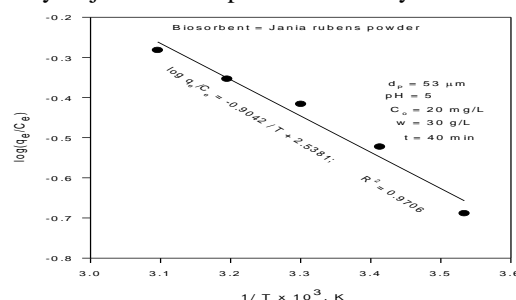


Fig.3.12 Vant Hoff's plot for biosorption of CV dye

I. Optimization using Response Surface Methodology (RSM):

A. Optimization using CCD

In the present study, the levels of four process input variables for % biosorption are shown in table-IV.

Table – IV
Levels of different process variables in coded and uncoded form for % biosorption of CV dye using Jania rubens powder

Variable	Name	Range of levels				
		-2	-1	0	1	2
X1	pH of aqueous solution	3	4	5	6	7
X2	Initial concentration, Co, mg/L	10	15	20	25	30
X3	Biosorbent dosage, w, g/L	35	40	45	50	55
X4	Temperature, T, K	28	30	31	32	33

The parameters that have greater influence over the response are to be identified so as to find the optimum condition for the biosorption of CV dye. For optimization of medium constituents, the regression equation is: % biosorption of CV dye is a function of pH (X1), Co (X2), w (X3), and T (X4). The variations in the corresponding coded values of four parameters and response are presented in table-3.13 depending on experimental runs and predicted values proposed by CCD design. The following equation represents multiple regression analysis of the experimental data: $Y = -2056.89 + 44.52 X1 + 2.93 X2 + 14.77 X3 + 10.94 X4 - 2.15 X1^2 - 0.09 X2^2 - 0.09 X3^2 - 0.02 X4^2 - 0.33 X1X2 - 0.51 X1X3 + 0.03 X1X4 - 0.04 X2X3 + 0.01 X2X4 - 0.01 X3X4$ ----- (5)

Table - V
Results from CCD for CV dye biosorption by Jania rubens powder

Critical values; Variable: % Removal

Solution: maximum

Predicted value at solution: 97.43419

	Observed	Critical	Observed
pH	3.0000	5.2030	7.0000
Concentration	10.0000	20.0844	30.0000
Dosage	0.5000	1.8451	2.5000
Temperature	283.0000	306.3282	323.0000

	pH	Conc	Dos	Temp	% Rem	Predic
1	4	15	1	293	83.59	83.64583
2	4	15	1	313	85.22	85.19167
3	4	15	2	293	85.19	85.24167
4	4	15	2	313	87	86.96250
5	4	25	1	293	78.28	78.32500
6	4	25	1	313	80.12	80.09583
7	4	25	2	293	80.79	80.84583
8	4	25	2	313	82.82	82.79167
9	6	15	1	293	83.08	83.12500
10	6	15	1	313	84.72	84.69583
11	6	15	2	293	86.19	86.24583
12	6	15	2	313	88.02	87.99167
13	6	25	1	293	85.52	85.47917
14	6	25	1	313	87.32	87.27500
15	6	25	2	293	89.48	89.52500
16	6	25	2	313	91.52	91.49583
17	3	20	1.5	303	69.38	69.42917
18	7	20	1.5	303	77.58	77.61250
19	5	10	1.5	303	83.98	84.02917
20	5	30	1.5	303	82.18	82.21250
21	5	20	0.5	303	89.18	89.21250
22	5	20	2.5	303	94.98	95.02917
23	5	20	1.5	283	89.18	89.21250
24	5	20	1.5	323	92.68	92.72917
25	5	20	1.5	303	96.58	96.60000
26	5	20	1.5	303	96.58	96.60000
27	5	20	1.5	303	96.58	96.60000
28	5	20	1.5	303	96.58	96.60000
29	5	20	1.5	303	96.58	96.60000
30	5	20	1.5	303	96.58	96.60000

Experimental conditions [Coded Values] and observed response values of central composite design with 24 factorial runs, 6- central points and 8- axial points. Agitation time fixed at 40 min and biosorbent

size at 53 µm The results of eq. 5.17 are presented in the form of ANOVA. From the Fisher’s F-test and a very low probability value (Pmodel>F=0.000000), the ANOVA of the model clearly explains that the model is highly significant (Refer table 3.15). It shows that the treatment differences are significant

	SS	df	MS
(1)pH (L)	101.024	1	101.0241
pH (Q)	912.847	1	912.8470
(2)Concentration(L)	4.824	1	4.8241
Concentration(Q)	311.311	1	311.3110
(3)Dosage (L)	50.344	1	50.3441
Dosage (Q)	34.342	1	34.3424
(4)Temperature(L)	19.476	1	19.4760
Temperature(Q)	54.257	1	54.2571
1L by 2L	59.367	1	59.3670
1L by 3L	2.235	1	2.2350
1L by 4L	0.000	1	0.0000
2L by 3L	0.801	1	0.8010
2L by 4L	0.040	1	0.0400
3L by 4L	0.040	1	0.0400
Error	0.016	15	0.0011
Total SS	1334.411	29	

The model is reduced to the following form by removing insignificant term (X2).

$$Y = -2056.89 + 44.52 X_1 + 2.93 X_2 + 14.77 X_3 + 10.94 X_4 - 2.15 X_1^2 - 0.09 X_2^2 - 0.09 X_3^2 - 0.02 X_4^2 - 0.33 X_1 X_2 - 0.51 X_1 X_3 + 0.03 X_1 X_4 - 0.04 X_2 X_3 + 0.01 X_2 X_4 - 0.01 X_3 X_4 \text{ ----- (6)}$$

The regression coefficient value of 0.99996 indicates that 0.004 % of the total variations are not satisfactorily explained by the model . The statistical significance of the ratio of mean square due to regression and mean square due to residual error are tested. It is proved from that table that, the F-statistics value for entire model is higher. i.e., % biosorption of CV dye can be adequately explained by the model equation. Generally P values lower than 0.05 indicates that the model is considered to be statistically significant at 95% confidence level. The % biosorption prediction from the model is shown in table-3.14. It is implied from table-5.15 that all the squared terms of the variables are significant compared to the linear terms. Among the interaction terms, all the terms (P < 0.05) are highly significant on biosorption capacity.

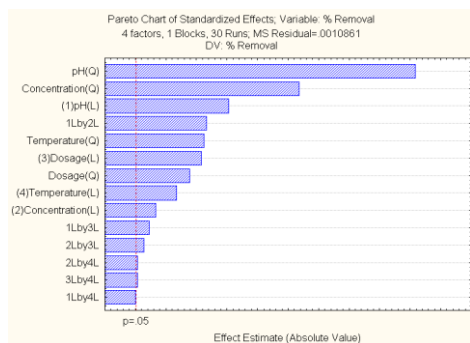


Fig. 3.13 Pareto Chart

The optimal set of conditions for maximum percentage biosorption of CV dye is pH = 5.8809, initial CV dye concentration = 20.0533 mg/L, biosorbent dosage = 44.3682 g/L, and temperature = 304.2994 K. The extent of biosorption of CV dye at these optimum conditions was 95.8056 %. 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 CV dye is 93 %, which is closer to the predicted % biosorption.

3.9.2 Interpretation of residual graphs:

Fig. 3.14 shows normal probability plot of residual values. The experimental values are in good agreement with predicted values with minimum error.

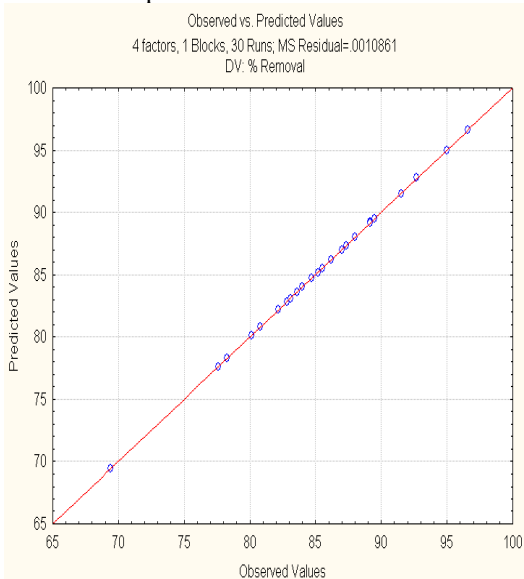


Fig. 3.14 Normal probability plot for % biosorption of CV dye

3. Interaction effects of biosorption variables:

Figs.3.15(a) to (f) depict the three-dimensional view of response surface plots. The % biosorption of biosorbent is maximal at low and high levels of the variables but there is a region where increasing/decreasing trend in % biosorption is not observed.

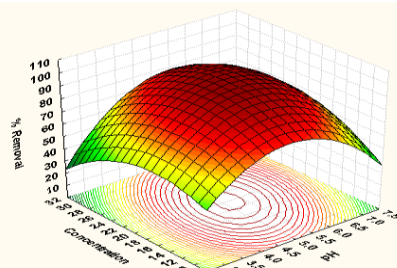


Fig. 3.15 (a) Surface contour plot for the effects of pH and initial concentration of CV dye on % biosorption

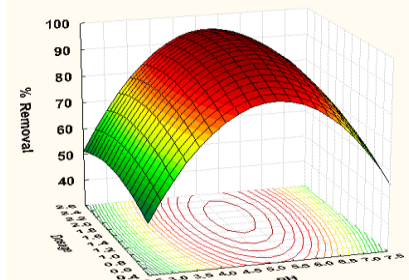


Fig. 3.15 (b) Surface contour plot for the effects of pH and dosage on % biosorption of CV dye

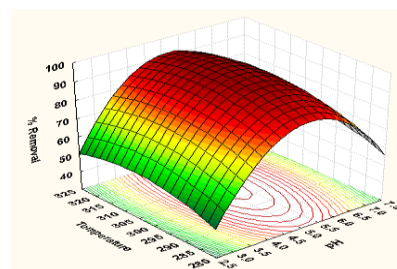


Fig. 3.15 (c) Surface contour plot for the effects of pH and Temperature on % biosorption of CV dye

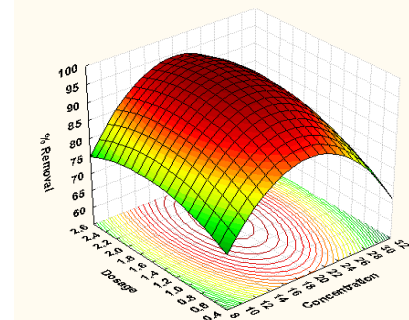


Fig. 3.15 (d) Surface contour plot for the effects of initial concentration and Dosage on % biosorption of CV dye

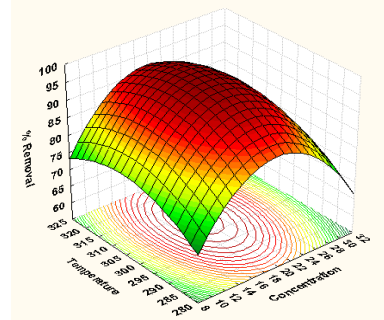


Fig. 3.15 (e) Surface contour plot for the effects of initial concentration and Temperature on % biosorption of CV dye

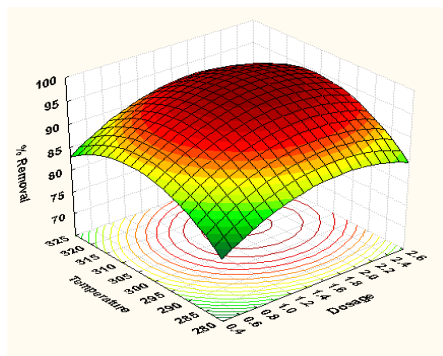


Fig.3.15 (f) Surface contour plot for the effects of Dosage and Temperature on % biosorption of CV dye

IV CONCLUSIONS

The equilibrium agitation time for CV dye biosorption is 40 minutes. The optimum dosage for biosorption is 30 g/L. Maximum extent of biosorption is noted at pH = 5. From the predicted values of RSM results, maximum biosorption of CV dye (85.03392 %) is observed when the processing parameters are set as pH = 5.9549, w = 30.3234 g/L, Co = 19.7484 mg/L and T = 303.3380 K.

The investigation also reveals the:

1. Endothermic nature of biosorption as ΔH is positive (17.3128 J/mole)
2. Spontaneity of the biosorption as ΔG is negative (-14707.7 J/mole)
3. Irreversible nature of biosorption as ΔS is positive (48.59736)

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