

Studies on Removal of Orange G Dye Using Carpentry Waste as Sorbent in Synthetic Waste Water

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Abstract: - The objective of this study is to minimize the threat to the environment by utilizing carpentry waste powder for the removal of Orange G dye from synthetic waste water. In recent time rising an invincible interest to detect a cost-effective and eco-friendly material for the removal of hazardous chemicals from contaminated water. The biosorption studies are carried out in a batch process varying with six parameters agitation time, biosorbent size, biosorbent dosage, initial concentration of Orange G dye in the aqueous solution, pH of aqueous solution and temperature of the process. From the experimentation it is revealed that 35 g/L of carpentry waste powder of 53 μ m size is enough to remove 64 % of 20 mg/L concentration of Orange G dye from 50mL of aqueous solution in 40 min.. A significant increase in percentage removal of Orange G dye is observed as pH value increases from 2 to 6 and the percentage removal is maximum at pH = 6. The percentage removal decreases beyond that pH. Freundlich and Langmuir models are applied to describe the equilibrium isotherms. The kinetic studies shows that the biosorption of Orange G dye onto Carpentry waste powder follows second order kinetics. Various thermodynamic parameters such as change in enthalpy, entropy and gibb's free energy are also determined. It was found that the biosorption is exothermic, irreversible and spontaneous

Keywords: - (Carpentry waste powder, Orange G dye, Biosorption).

I INTRODUCTION

Water pollution is a worldwide problem due to its negative impacts for human health, plants, animals and uncovered materials [1]. Industrialization and urbanization are extraordinarily attributed the contamination of water. Since, an extensive number of organic and industrial effluents has been brought into the condition that has expanded water and land contamination issues. Water contamination because of dyeing industry is the matter of incredible worry since huge amount of effluent is released into the water bodies. Central pollution control board has recorded the dyeing industries as one of the intensely polluting industries [2]. The textile effluent is exceptionally harmful in nature as it contains high suspended solids, COD, color and synthetic substances alongside high convergence of heavy metals like Cu, Cd, Zn Ni and Pb. The dye wastewater pollutes the surface and ground water, in this way making it unfit for water system and drinking [3].

Modern industrial developments have brought about age of expansive measure of waste water containing poisonous toxins. A portion of the impacts of dye bearing waste water are

aesthetic pollution of environment and also carcinogenicity due to their degradation products. Numerous strategies like electrochemical coagulation, reverse osmosis, nano filtration, adsorption activated minerals and so forth, are utilized for the expulsion of dye from waste water.

Adsorption has been observed to be an effective and economic process for the treatment of dyeing industry effluent. Exploration of good low cost adsorbent may contribute to the sustainability of the environment and offer promising benefits for the commercial purpose in future. Some of the low cost adsorbents are and waste plant materials like rice husk , peanut hull , orange waste, palm shell, coconut shell squander, tropical wood , saw dust, palm shell, corncob, eucalyptus bark, pine saw dust, pistachio shells, Babool wood and so on. The expense of activated carbon arranged from waste materials are negligible when compared with industrial activated carbon [4].

II EXPERIMENTAL PROCEDURE

2.1 Reagents and materials:

All the chemicals used in this investigation were of analytical grade and used without further purification. Solutions of Orange G dye were made from a stock solution containing 1000mg of Orange G dye powder in 1litre. pH of Orange G dye solution was adjusted to the desired value by addition of 0.1 M HCL and 0.1 M NaOH solutions.

2.2 Preparation of the biosorbent:

Carpentry waste was taken from a carpentry shop and is dried under sun light in order to remove moisture content present in it. After that it is finely powdered and sized by passing it through a set of sieves ranging from 152 to 53 mesh sizes. The powder of 53-152 μ m fractions was separated and used as an adsorbent.

2.3 Preparation of the 1000mg/L of Orange G dye solution:

Orange G dye stock solution of 1000 mg/L was prepared by dissolving 1 g of Orange G dye in 1000 ml of distilled water. It produces 1000 ppm dye solution. From the dye stock solution (1000 ppm) various concentrations of dyes were prepared by suitable dilutions. 100 ppm dye solution was prepared by diluting 100 ml of 1000 ppm dye stock solution with distilled water in 1000 ml volumetric flask up to the mark. Similarly solutions with different dye concentrations such as 20, 50, 100, 150 and 200 ppm were prepared.

III RESULT AND DISCUSSION

In the present investigation, the potential of *carpentry waste powder* as a biosorbent for removal of Orange G dye present in an aqueous solution is investigated. The effects of various parameters on biosorption of Orange G dye are studied. The measured data consists of initial and final concentration of Orange G dye, agitation time, biosorbent size, and biosorbent dosage, pH of the aqueous solution and temperature of the aqueous solution. The experimental data are obtained by conducting batch experiments.

A. Effect of agitation time:

The equilibrium agitation time is determined by plotting the % biosorption of Orange G dye against agitation time as shown fig. 5.1 for the interaction time intervals between 5 min to 180 min and the % biosorption is 10 % to 64 % and dye uptake is 0.2 mg/g to 1.28 mg/g. The % biosorption is increased briskly up to 40 min reaching 64 % and dye uptake 1.28 mg/g. Beyond 40 min, the % biosorption is constant indicating the attainment of equilibrium conditions .

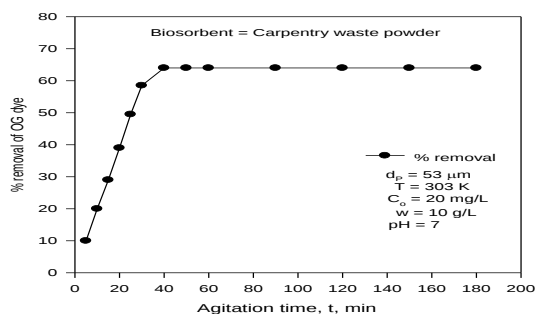


Figure 1 Effect of agitation time on % removal of orange G dye

The maximum percentage of biosorption is attained at 40 minutes. The percentage biosorption of Orange G dye becomes constant after 40 min. Therefore, all other experiments are conducted at this agitation time [5].

B. Effect of biosorbent size:

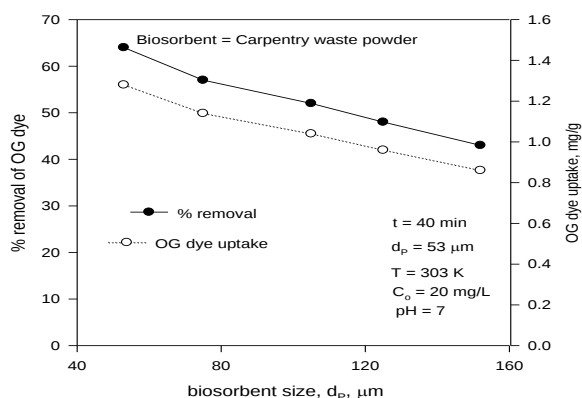


Figure 2 Effect of biosorbent size on % removal of orange G dye

The percentage biosorption is decreased from 64 % to 43 % and dye uptake is decreased from 1.28 mg/g to 0.86 mg/g as the biosorbent size decreases from 153 μm to 53 μm. This phenomenon is expected, as the size of the particle decreases, surface area of the biosorbent increases; thereby the number of active sites on the biosorbent also increases [6].

C. Effect of pH:

The effect of pH of aqueous solution on % biosorption of Orange G dye is shown in fig.5.3. The % biosorption of Orange G dye is increased from 47 % to 70 % and dye uptake increased from 0.94 mg/g to 1.4 mg/g as pH is increased from 2 to 6 and decreased beyond the pH value of 6. % biosorption is decreased from pH 6 to 8 reaching 70 % from 58 % and dye uptake is 1.4 mg/g to 1.16 mg/g. Low pH depresses biosorption due to competition with H⁺ ions for appropriate sites on the biosorbent surface. However, with increasing pH, this competition weakens and Orange G dye ions replace H⁺ ions bound to the biosorbent [7].

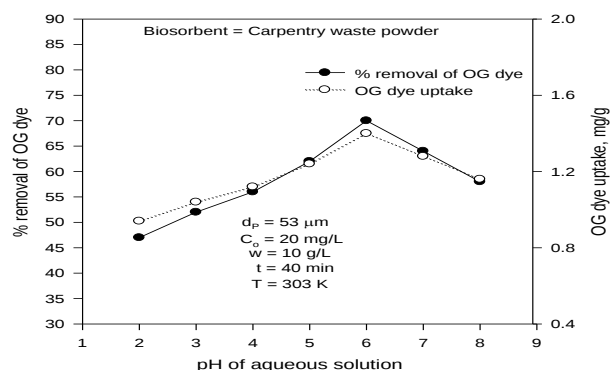


Figure 3 Effect of pH on % removal of orange G dye

D. Effect of initial concentration of Orange G dye:

The percentage biosorption of Orange G dye is decreased from 70 % to 43.7 % and dye uptake is increased from 1.4 mg/g to 8.74 mg/g with an increase in C₀ from 20 mg/L to 200 mg/L. Such behavior can be attributed to the increase in the amount of biosorbate to the unchanging number of available active sites on the biosorbent [8] .

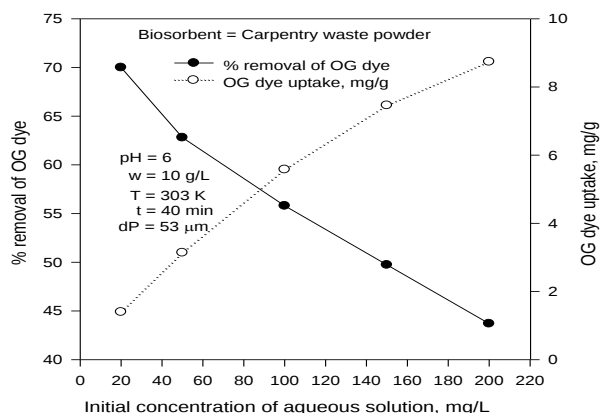


Figure 4 Effect of concentration on % removal of orange G dye

E. Effect of biosorbent dosage:

The biosorption of Orange G increased from 70 % to 89 % and the dye uptake is decreased 1.4 mg/g to 0.506 mg/g with an increase in biosorbent dosage from 10 g/L to 35 g/L. The change in percentage biosorption of Orange G dye is marginal from 89 % to 95 % when 'w' is increased from 35 to 80 g/L. Hence all other experiments are conducted at 35 g/L dosage [9].

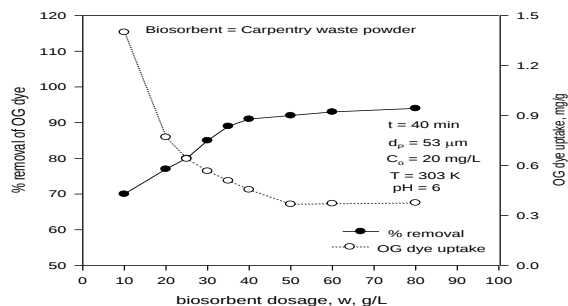


Figure 5 Effect of dosage on % removal of orange G dye

F. Effect of Temperature:

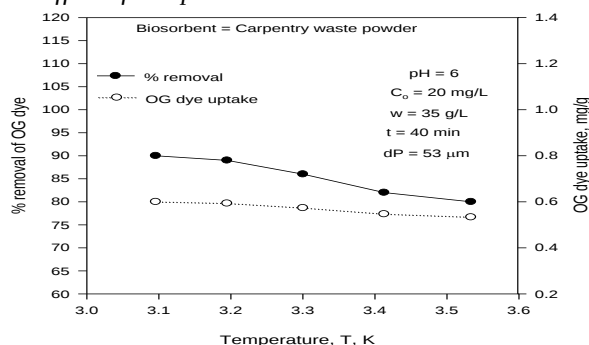


Figure 6 Effect of temperature on % removal of orange G dye

The effect of changes in the temperature on the Orange G uptake is shown in Fig.6. The maximum percentage of biosorption and maximum dye uptake is attained at 303k. So 303k is taken as optimum temperature [10].

G. Isotherms

The isotherms are characterized by the certain constants the values of which express the surface properties and affinity of the sorbent and can also be used to compare biosorptive capacity of biosorbent for different dyes. Out of several isotherm model equations five model equations were applied for this study. They are, Langmuir, Freundlich, and Temkin isotherms are obtained at room temperature.

Langmuir isotherm is drawn for the present data and shown in Fig.7. The equation obtained ' n ' $C_e/q_e = 0.0781 C_e + 4.2114$ with a good linearity (correlation coefficient, $R^2 \sim 0.9934$) indicating strong binding of Orange G dye ions to the surface of Carpentry waste powder [11]

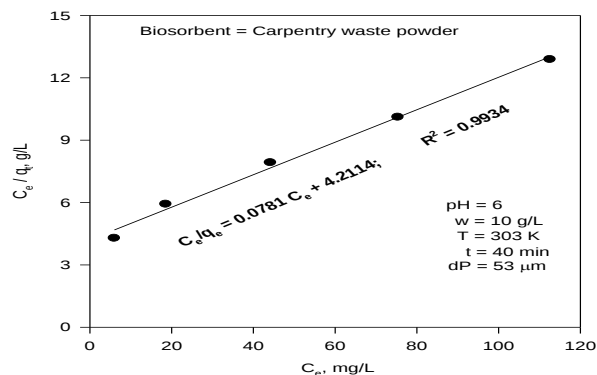


Figure 7 Langmuir isotherm for % removal of orange G dye

Freundlich isotherm is drawn between $\ln q_e = 0.6343 \ln C_e - 0.7509$; $\log C_e$ and $\log q_e$ in Fig.8 for the present data. The resulting equation has a correlation coefficient of 0.9933. [12].

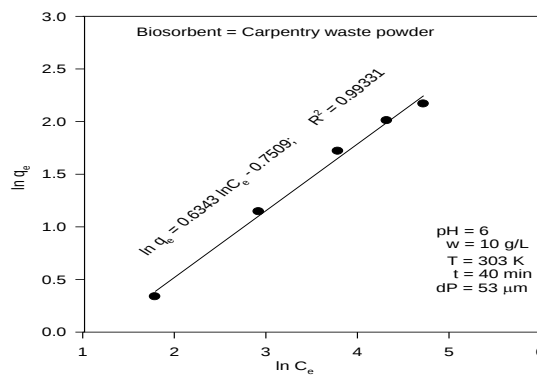


Figure 8 Freundlich isotherm for % removal of orange G dye

The present data are analysed according to the linear form of Temkin isotherm and the linear plot is shown in Fig.9. The equation obtained for Orange G biosorption is: $q_e = 2.5375 \ln C_e - 3.6429$ with a correlation coefficient 0.9731.

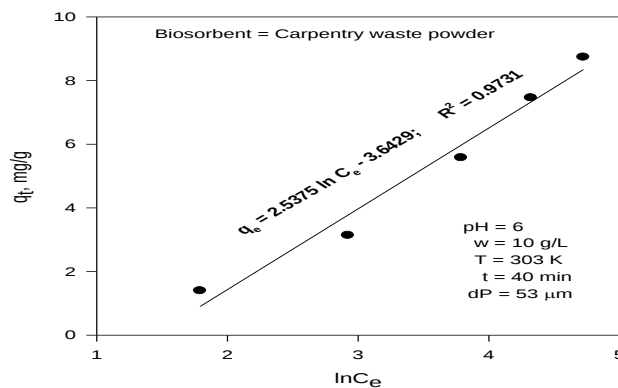


Figure 9 Temkin isotherm for % removal of orange G dye

Table - 1 Isotherm Constants

Langmuir isotherm	Freundlich isotherm	Temkin isotherm
$q_m = 12.8041 \text{ mg/g}$	$K_f = 0.471942 \text{ mg/g}$	$A_T = 0.237966 \text{ L/mg}$
$R_L = 0.899872$	$n = 0.70906$	$b_T = 992.7653$
$R^2 = 0.9934$	$R^2 = 0.9933$	$R^2 = 0.9731$

The best fit model is determined based on the linear regression correlation coefficient (R^2). From the Figs 7, 8 & 9, it is found that biosorption data are well represented by Langmuir isotherm with higher correlation coefficient of 0.9934, followed by Freundlich and Temkin isotherms with correlation coefficients of 0.9933 and 0.9731 respectively [13].

H. Biosorption kinetics:

The kinetics of the biosorption data was analyzed using two kinetic models, Lagergren first order and pseudo second order.

Plot of $\log(q_e - q_t)$ versus 't' gives a straight line for first order kinetics, facilitating the computation of adsorption rate constant (K_{ad}). [14]

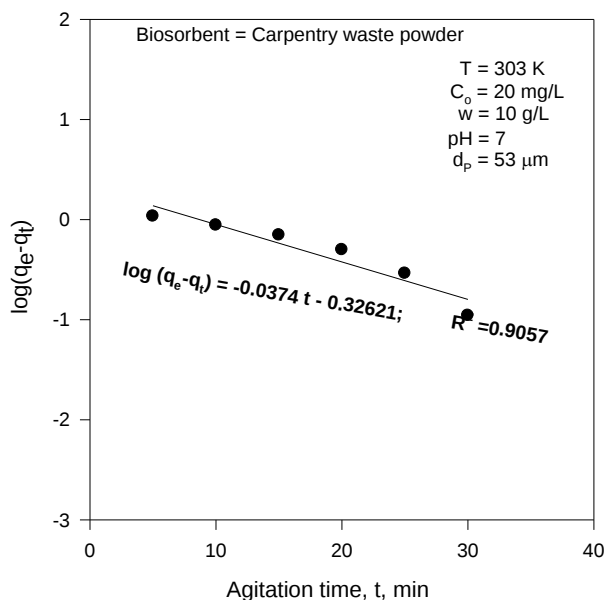


Figure 10 First order kinetics for % biosorption of orange G dye

If the pseudo second order kinetics is applicable, the plot of (t/q_t) versus 't' gives a linear relationship that allows computation of q_e and K .

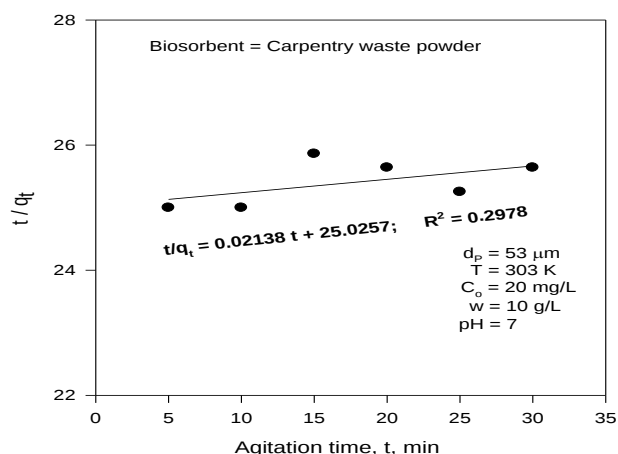


Figure 11 Second order kinetics for % biosorption of orange G dye.

Table – 2 Equations and rate constants

Order	Equation	Rate constant	R^2
Lagergren first order	$\log(q_e - q_t) = -0.0374 t - 0.32621$	$0.086132 \text{ min}^{-1}$	0.9057
Pseudo second order	$(t/q_t) = 0.02138 t + 25.0257$	$1.83 \text{E-}05 \text{ g/(mg-min)}$	0.2978

Based on the regression coefficient obtained from fig.10&11, I notice that the Lagergren first order model suits.

I. Thermodynamics of biosorption:

Biosorption is temperature dependant. In general, the temperature dependence is associated with three thermodynamic parameters namely change in enthalpy of biosorption (ΔH), change in entropy of biosorption (ΔS) and change in Gibbs free energy (ΔG).

The Vant Hoff's equation is

$$\log(q_e/C_e) = \Delta H/(2.303 RT) + (\Delta S/2.303 R)$$

Experiments are conducted to understand the biosorption behavior varying the temperature from 283 to 323 K. the plots indicating the effect of temperature on biosorption of Orange G dye for different initial dye concentrations are shown in fig.12. The Vant Hoff's plots for the biosorption data obtained at various initial concentrations of the Orange G dye are shown in fig.12. The values are $\Delta G = -12891.4$, $\Delta H = 16.69363$ and $\Delta S = 41.90926$. [15]

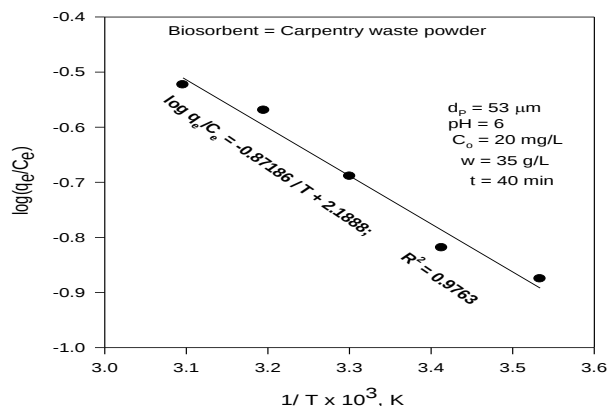


Figure 12 Vant Hoff's plot for biosorption

J. Optimization using Response Surface Methodology (RSM)(CENTRAL COMPOSITE DESIGN):

RSM consists of a group of empirical techniques devoted to the evaluation of relationship existing between a cluster of controlled experimental factors and measured responses according to one or more selected criteria.

The parameters that have greater influence over the response are to be identified so as to find the optimum condition for the biosorption of Orange G Dye. The quadratic model is used in the present study, to relate four independent variables and percentage biosorption of Orange G dye. The regression equation for is % biosorption of Orange G dye (Y) is function of pH (X_1), C_o (X_2), w (X_3) and T (X_4). Experimental conditions [Coded Values] and observed response values of central composite design with 2^4 factorial runs, 6- central points and 8- axial points.

The following equation represents multiple regression analysis of the experimental data for the biosorption of Orange G Dye:

$$Y = -4611.59 + 70.83 X_1 + 3.71 X_2 + 312.08 X_3 + 27.54 X_4 - 5.53 X_1^2 - 0.21 X_2^2 - 76.20 X_3^2 - 0.05 X_4^2 + 0.25 X_1 X_2 - 0.28 X_1 X_3 - 0.03 X_1 X_4 + 0.26 X_2 X_3 + 0.01 X_2 X_4 - 0.15 X_3 X_4 \dots(1)$$

Table-3 Results from CCD for biosorption of Orange G dye using carpentry waste powder

	Observed	Predicted	Resids
1	70.20000	70.20750	-0.007500
2	70.49000	70.45500	0.035000
3	72.78000	72.82167	-0.041667
4	71.59000	71.61417	-0.024167
5	67.48000	67.47000	0.010000
6	69.38000	69.50750	-0.127500
7	71.40000	71.38917	0.010833
8	72.02000	71.97167	0.048333
9	71.02000	71.06167	-0.041667
10	70.28000	70.30917	-0.029167
11	73.50000	73.39083	0.109167

12	71.18000	71.18333	-0.003333
13	73.30000	73.29417	0.005833
14	74.38000	74.33167	0.048333
15	76.90000	76.92833	-0.028333
16	76.50000	76.51083	-0.010833
17	67.20000	67.15750	0.042500
18	72.52000	72.55083	-0.030833
19	70.08000	70.08417	-0.004167
20	72.69000	72.67417	0.015833
21	70.58000	70.53250	0.047500
22	75.29000	75.32583	-0.035833
23	74.02000	74.03417	-0.014167
24	73.89000	73.86417	0.025833
25	91.98000	91.98000	0.000000
26	91.98000	91.98000	0.000000
27	91.98000	91.98000	0.000000
28	91.98000	91.98000	0.000000
29	91.98000	91.98000	0.000000
30	91.98000	91.98000	0.000000

Table-4 ANOVA of Orange G biosorption for entire quadratic model

Source of variation	SS	Df	Mean square (MS)	F-value	P > F
Model	2051.118	14	146.5054	45782.93	0.00000
Error	0.048	15	0.0032		
Total	2051.166				

df- degree of freedom; SS- sum of squares; F- factor F; P- probability. $R^2=0.99467$; R^2 (adj):0.99264.

In general, the Fischer's 'F-statistics' value ($F=MS_{\text{model}}/MS_{\text{error}}$), where MS (mean square) with a low probability 'P' value indicates high significance of the regression model. The ANOVA of the regression model demonstrates that the model is highly significant, as is evident from the Fisher's F-test ($F_{\text{model}} = 45782.93$) and a very low probability value ($P_{\text{model}} > F=0.000000$). More ever, the computed F-value ($F_{0.05}(14,15) = MS_{\text{model}} / MS_{\text{error}} = 146.5054 / 0.0032$) is greater than the tabular F-value ($F_{0.05}(14,15)$ tabulars = 3.56) at the 5% level, indicating that the treatment differences are significant.

The optimal set of conditions for maximum percentage biosorption of Orange G is pH = 6.1305, biosorption dosage (w) = 1.7820 g/L, Temperature (T) = 302.9058 and initial Orange G concentration (C_o) = 20.4112 mg/L. The extent of biosorption of Orange G calculated at these optimum conditions is 92.17151 %. The experimental values are in good agreement with predicted values.

Among the interaction terms, all the terms ($P < 0.05$) are insignificant on the biosorption capacity. The following two figures show the pareto chart and normal probability plot

(NPP) of residual values. It could be seen that the experimental points are reasonably aligned suggesting normal distribution.

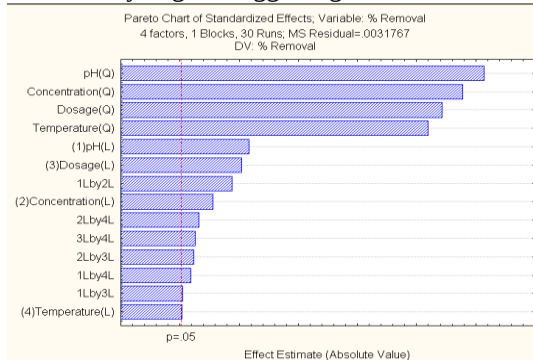


Figure 13 Pareto Chart

Interpretation of residual graphs:

Normal probability plot (NPP) is a graphical technique used for analyzing whether or not a data set is normally distributed to greater extent. The difference between the observed and predicted values from the regression is termed as residual. Fig. 5.14 exhibits normal probability plot for the present data. It is evident that the experimental data are reasonably aligned implying normal distribution [16,17].

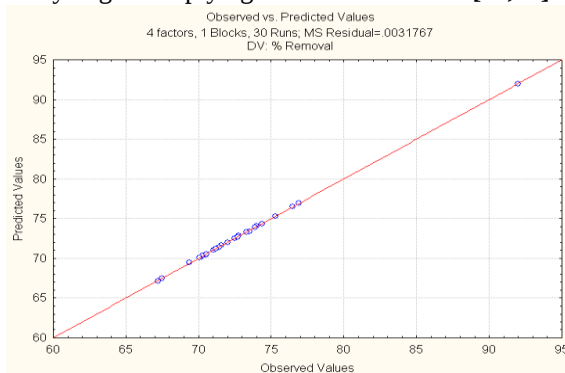
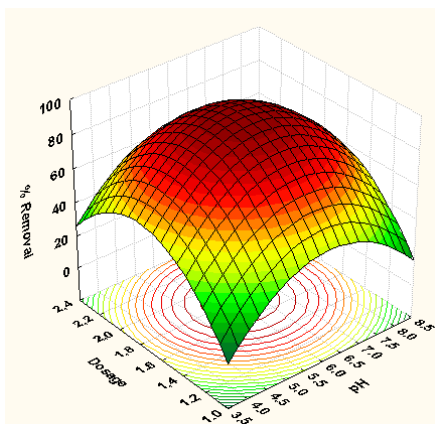
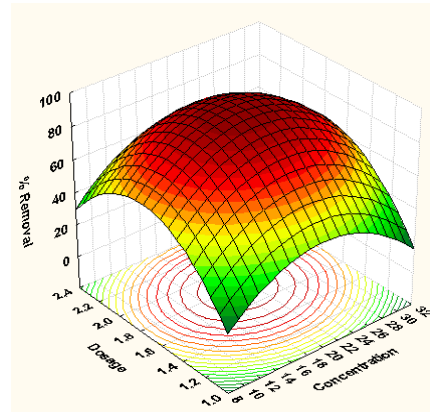


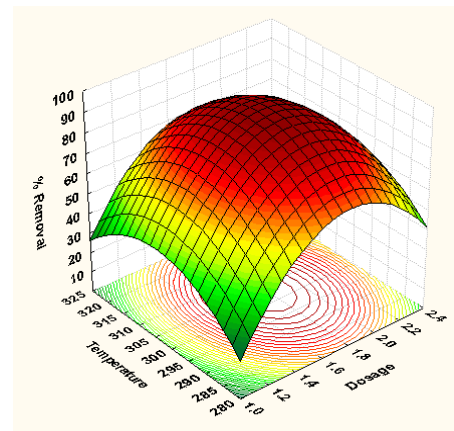
Figure 14 Normal probability plot for % biosorption of Orange G dye



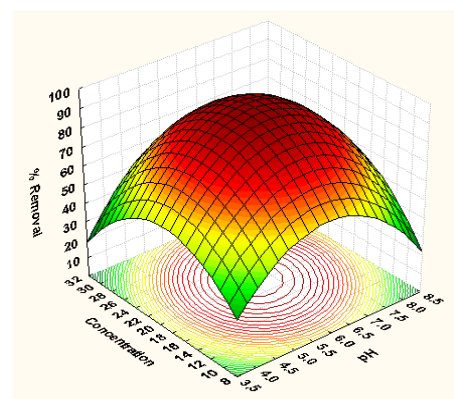
(a) Surface contour plot for the effects of dosage and pH of Orange G dye on % biosorption



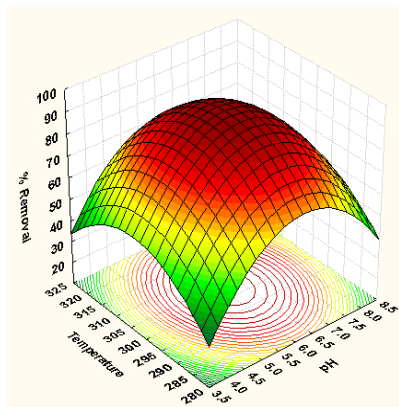
(b) Surface contour plot for the effects of dosage and concentration of Orange G dye on % biosorption



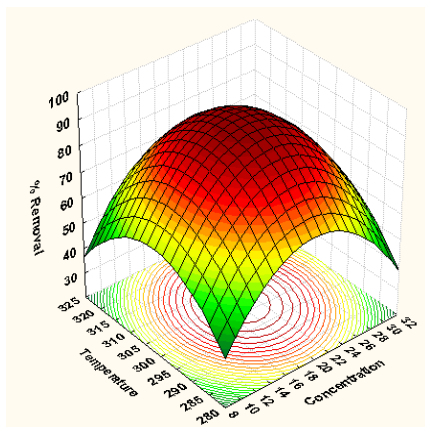
(c) Surface contour plot for the effects of Temperature and dosage of Orange G dye on % Biosorption



(d) Surface contour plot for the effects of Concentration and pH of orange G dye on % biosorption



(e) Surface contour plot for the effects of Temperature and pH of Orange G dye on % biosorption



(f) Surface contour plot for the effects of Temperature and initial Concentration of Orange G dye on % biosorption

It is evident from response surface contour plots that the % biosorption is minimal at low and high levels of the variables. This behavior confirms that there is a presence of optimum for the input variables in order to maximize % biosorption. The role played by all the variables is so vital in % biosorption of Orange G dye and seen clearly from the plots. The predicted optimal set of conditions for maximum % biosorption of Orange G are:

Biosorbent dosage	=	1.7820 g/L
Initial Orange G dye ion concentration	=	20.4112 mg/L
pH of aqueous solution	=	6.1305
Temperature	=	302.9058
% biosorption of Orange G dye	=	92.17151

The experimental optimum values are compared with those predicted by CCD in table-5. The experimental values are in close agreement with those from and CCD

Table-5 Comparison between optimum values from CCD and experimentation

Variable	CCD	Experimental
Biosorbent dosage, w, g/L	1.7820	1.75
Initial Orange G concentration, mg/L	20.4112	20
pH of aqueous solution	6.1305	6
Temperature	302.9058	303
% biosorption	92.17151	90

K. Characterization Studies:

Fourier Transform Infra-Red Spectroscopy (FTIR):

Infrared spectroscopy belongs to the group of molecular vibrational spectroscopies which are molecule-specific and give direct information about the functional groups, their kind, interactions and orientations. The shift of the bands and the changes in signal intensity allow the identification of the functional groups involved in dye sorption.

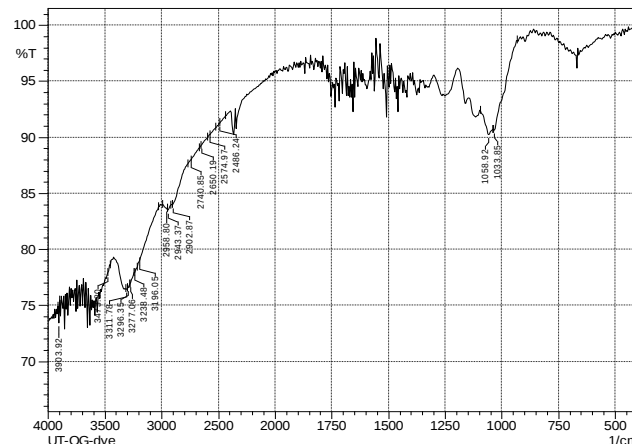


Figure 15 FTIR spectrum of Carpentry waste powder before biosorption

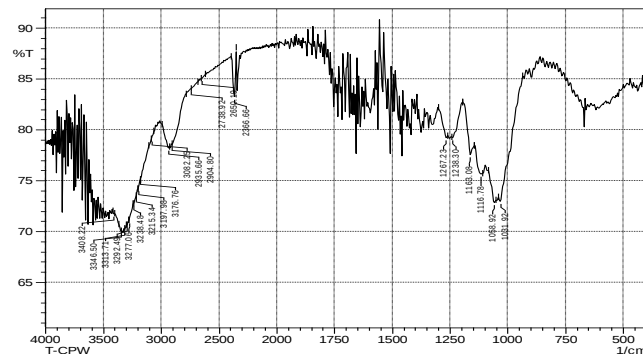


Figure 16 FTIR spectrum Carpentry waste powder after biosorption

Table-6 Shift of FTIR peaks for untreated and Carpentry waste powder treated orange G dye

Sl No	Peaks in untreated powder, cm ⁻¹	Peaks in biosorbent Treated Orange G powder	Bond and Functional group Description
1	-----	1031.92	-C-C-group
2	1033.85	-----	-C-C-group
3	1058.92	1058.92	-C-C-group
4	-----	1116.78	-CH ₂ -O-CH ₂ linkage
5	-----	1163.08	C-O stretching
6	-----	1238.30	C-O stretching
7	-----	1267.23	-SO ₃ stretching
8	-----	2366.66	Phosphate ester group
9	2486.24	-----	Phosphate ester group
10	2574.97	-----	Phosphate ester group
11	2650.19	2650.19	CH ₂ stretching vibrations
12	-----	2738.92	CH ₂ stretching vibrations
13	2740.85	-----	CH ₂ stretching vibrations
14	2902.87	-----	CH ₂ stretching vibrations
15	-----	2904.80	CH ₂ stretching vibrations
16	-----	2935.66	CH ₂ stretching vibrations
17	2943.37	-----	CH ₂ stretching vibrations
18	2958.80	-----	CH ₂ stretching vibrations
19	-----	3082.25	=C-H of alkene or arene
20	-----	3176.76	=C-H of alkene or arene
21	3196.05	-----	bounded -OH and -NH groups
22	-----	3197.98	bounded -OH and -NH groups
23	-----	3215.34	bounded -OH and -NH groups
24	3238.48	3238.48	bounded -OH and -NH groups
25	3277.06	3277.06	bounded -OH and -NH groups
26	-----	3292.49	
27	3296.35	-----	
28	3311.78	-----	
29	-----	3313.71	
30	-----	3346.50	Hydroxyl(-OH) stretching or amine (-NH ₂) stretching
31	-----	3408.22	Hydroxyl(-OH) stretching or amine (-NH ₂) stretching
32	3473.80	-----	Hydroxyl(-OH) stretching or amine (-NH ₂) stretching
33	3903.92	-----	Hydroxyl(-OH) stretching or amine (-NH ₂) stretching

X-Ray Diffraction:

XRD FOR ORANGE G DYE UNTREATED WITH CARPENTRY WASTE POWDER:

XRD patterns of untreated powder are shown in figs.17 & 18. XRD patterns shown in figs. 5.17(a) & (b) do not indicate sharp peaks, less crystallinity and exhibit little amorphous nature. The peaks at 2θ values of 0.1399, 0.9107, 0.8925, 0.8925, 0.6311 the presence of O₂Si,

Cr₂CuSe₄, C, C, C and their corresponding d-values are 2.1723, 0.2674, 0.1671, 0.1671, 0.1671.

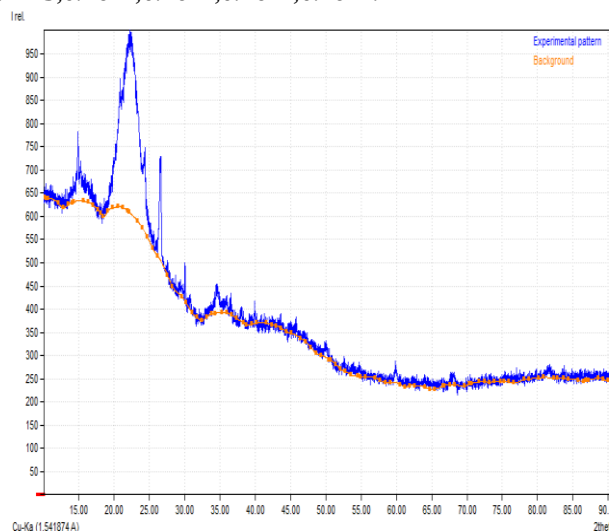


Figure 17 XRD pattern of orange G dye untreated Carpentry waste powder

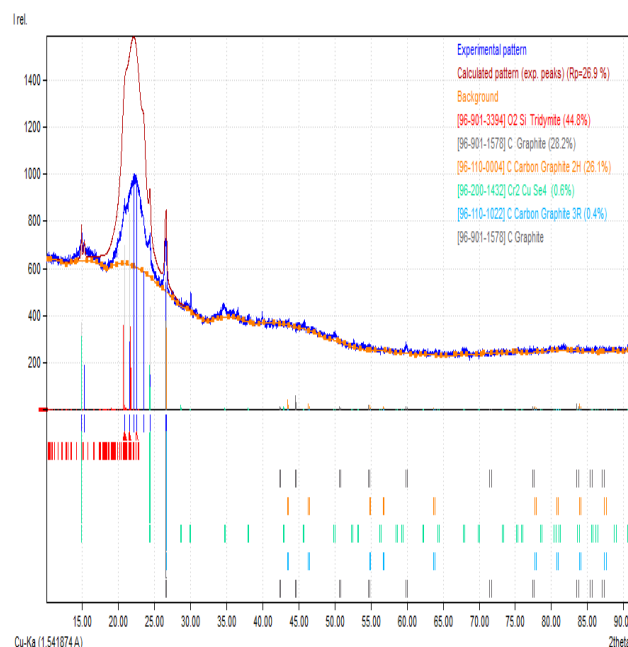


Figure 18 XRD pattern of orange G dye untreated Carpentry waste powder with matching compounds

XRD for orange G dye treated with carpentry waste powder:

XRD patterns for treated powder [Figs.19 & 20] exhibit good crystallinity, more amorphous nature and increase in surface area and porosity. The peaks at 2θ values of 0.6872, 0.9261, 0.8811, 0.4702 and 0.6070 corroborate the presence of C₂N₂Zn, B₆Li₂, O₂Si, Cl₃CrH₁₂O₆, AsH₂LiO₄ and their corresponding d-values are 0.1671, 1.1697, 2.4731, 1.9718, 1.7044.

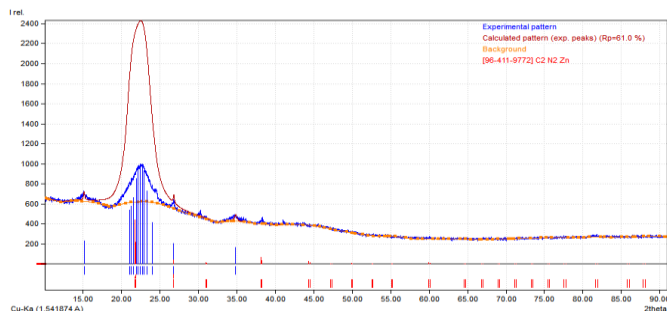


Figure 19 XRD pattern of orange G dye untreated Carpentry waste powder

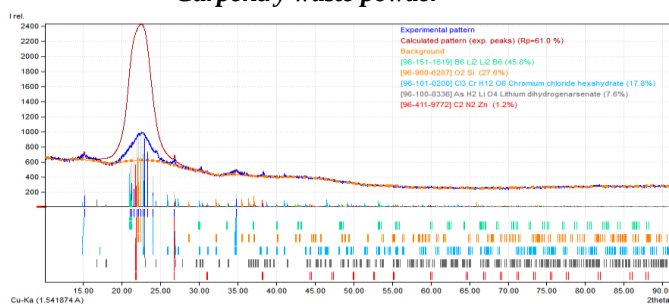


Figure 20 XRD pattern of orange G dye treated Carpentry waste powder with matching compounds

Scanning Electron Microscope (SEM):

SEM is a useful technique in the study of both the natural sorbent morphology and its modification derived from sorbate interactions. SEM is an electron microscope which provides images of the sample surface by scanning it with a high energy beam of electrons. The electron interactions with the atoms of the sample produce signals that contain information about topography, morphology, and composition of the sample surface.

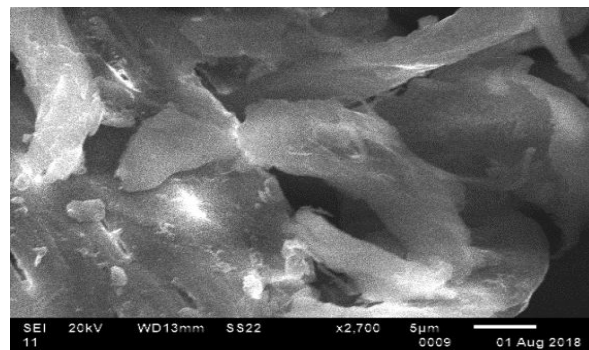
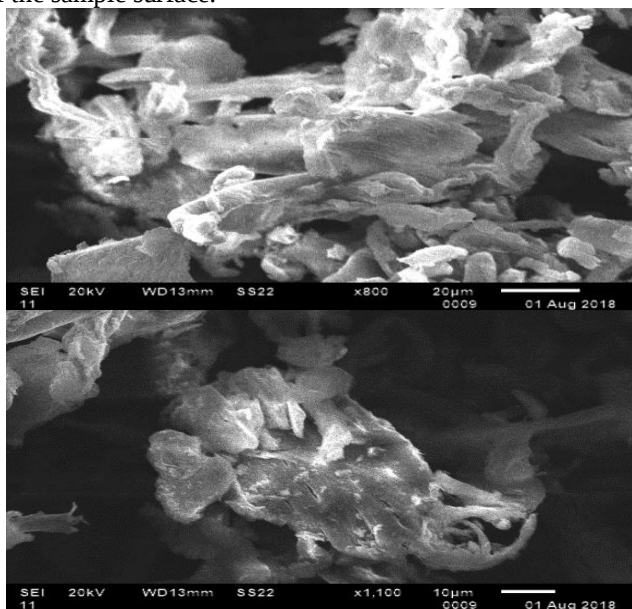


Figure 5.18 (a) SEM pattern of orange G dye untreated carpentry waste powder

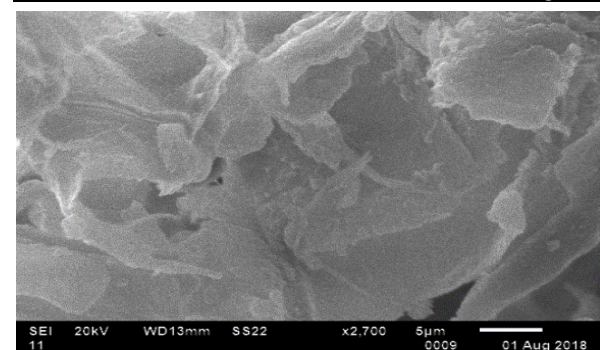
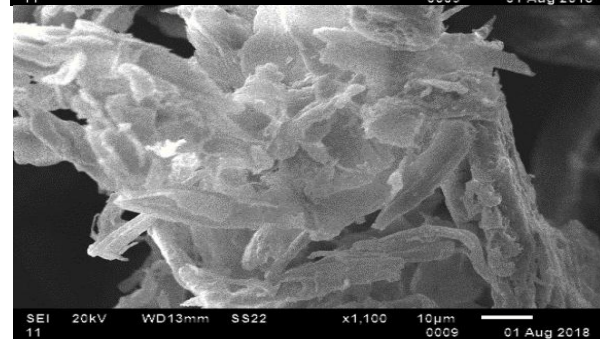
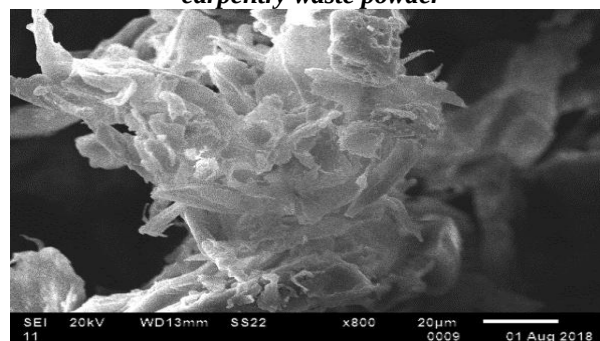


FIGURE 5.18 (b) SEM pattern of orange G dye treated carpentry waste powder

IV CONCLUSION

The aim of this investigation is to determine the suitability of carpentry waste powder as a biosorbent for the removal of Orange G dye from aqueous solutions. The equilibrium, kinetic and thermodynamic studies are carried out for biosorption of Orange G dye experimentally and

theoretically. The equilibrium agitation time for Orange G dye biosorption is 40 minutes. The percentage biosorption of Orange G dye decreased with the increase in biosorbent size from 53 μm (64 %) to 152 μm (43 %). Percentage biosorption of Orange G dye from the aqueous solution increases significantly with increase in pH from 2 (47 %) to 6 (70 %). The optimum dosage for biosorption is 35 g/L. The maximum uptake capacity of 0.573333 mg/g is obtained at 303 K. The maximum biosorption of Orange G dye (92.17151%) onto *carpentry waste* powder is observed when the processing parameters are set as: pH = 6.1305, w = 1.7820g/L and C_0 = 20.4112 mg/L using CCD. The investigation also reveals the endothermic nature of biosorption as ΔH (16.69363) is positive, irreversible nature of biosorption as ΔS (41.90926) is positive and spontaneity of biosorption as indicated by negative ΔG (ΔG = -1289.4 J/mole). Hence the above said *carpentry waste* powder is effective and efficient biosorbent and is capable of removing Orange G dye.

ACKNOWLEDGMENT

I am very thankful Andhra University, for providing the facilities to carry out this work. I express my deep sense of gratitude and sincere thanks to **Dr. CH. A. I. Raju**, Professor, Department of Chemical Engineering, Andhra University College of Engineering for her guidance and encouragement during the project work.

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