ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
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International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 5, Issue 4, April 2018

Studies on Biosorption of Methyl Red Dye With Pterocladia Lucida Powder and Optimization Throught Central Composite Design

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Abstract: Agile Industrialization resulted in the enormous discharge of dyes and heavy metals into waste water. These wastes inturn degrade the natural habitat of the living species. Biosorption, the best practice for the removal of dyes and heavy metals is considered for the present investigation. Pterocladia Lucida (red algae) powder is used for the removal of methyl red dye from aqueous solution. 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: MB, Pterocladia lucida, RSM, CCD

I. INTRODUCTION

Water plays a vital and essential role in our ecosystem [1]. It is one of the most important substances on earth. All plants and animals must have water to survive. With two thirds of the earth's surface covered by water and the human body consists of 75 percent of it [2], after pollution is a major global problem which requires ongoing evaluation and revision of water resource policy at all levels (international down to individual aquifers and wells). *Water pollution* is the *contamination* of *water* bodies (e.g. lakes, rivers, oceans, aquifers and groundwater) [6, 7, 8]. This form of environmental degradation occurs when *pollutants* are directly or indirectly discharged into *water* bodies without adequate treatment to remove harmful compounds [5]. It has been suggested that water pollution is the leading worldwide cause of deaths and diseases [3, 4] and that it accounts for the deaths of more than 14,000 people daily [3]. An estimated 580 people in India die of water pollution related illness every day. Several treatment methods have been employed the *methods* used include physical *processes* such as *filtration*, sedimentation, and distillation; biological *processes* such as slow sand filters or biologically active carbon; chemical *processes* such as flocculation and chlorination and the use of electromagnetic radiation such as ultraviolet light [9,10]. These types of different methods are very expensive and very harmful chemicals are used in water treatment. So as to avoid the environmental effects and to solve the above problem biosorption has been very promising now a day to treat. The present experiment was carried out on studies on removal of Methyl red dye using fresh water algae and optimization through Central Composite Design.

II. EXPERIMENTAL PROCEDURE

The present experimentation is carried out both batch-wise and column, on biosorption of Methyl red dye from aqueous solutions on the biosorbent – Pterocladia Lucida powder.

The experimental procedure consists of the following steps:

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

2.1. Preparation of the bisorbent

Pterocladia Lucida leaves was collected from Jodugulla palem beach, near tenneti park, Visakhapatnam. The collected biosorbent was washed with water several times

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ISSN 2394-3785 (Online)
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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.

2.2 Characterization of biosorbent

Biosorption of MR dye using pterocladia lucida powder 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, JXA-8100). 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.

2.3 Preparation of stock solution

The standard stock solution of MR dye (1000 mg/L) was prepared by dissolving 1.0 g of 99.9 % analytical grade Methyl Red 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.

2.4 Studies on Equilibrium Biosorption Process

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

TABLE 1

EXPERIMENTAL CONDITIONS FOR BIOSORPTION OF MR DYE

S.No.	Parameter	Values Investigated
1	Agitation time, t, min	5, 10, 15, 20, 25, 30, 40, 50, 60, 90, 120, 150 and 180
2	pH of the aqueous solution	2, 3, 4, 5, 6, 7 and 8
3	Initial dye concentration, Co, mg/L	20, 50, 100, 150 and 200
4	Initia Biosorbent dosage, w, g/L	10, 20, 25, 30, 35, 40, 50, 60 and 80
5	Temperature, K	283, 293, 303, 313 and 323

III. RESULTS AND DISCUSSION

Biosorption of MR dye onto pterocladia lucida powder Biosorption of MR dye using pterocladia lucida powder has many affecting factors which include characterization (FTIR, XRD, SEM), 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.

3.1 Characterization of pterocladia lucida powder

FTIR spectrum of untreated and MR dye treated pterocladia lucida powder:

FTIR measurements for untreated pterocladia lucida powder is shown in fig. 1 (a). FTIR spectra of MR loaded pterocladia lucida biomass is shown in fig. 1 (b). The band at 1201.70 cm-1 is also shifted to 1202.67 cm-1 due to the slight participation in MR dye loading by overlapping of C–O stretching. The band peak at 1418.71 cm-1 is shifted to 1419.67 and 1430.28 cm-1 respectively after treatment due to C–N stretching bonds.

ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
Available online at www.ijartet.com

International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
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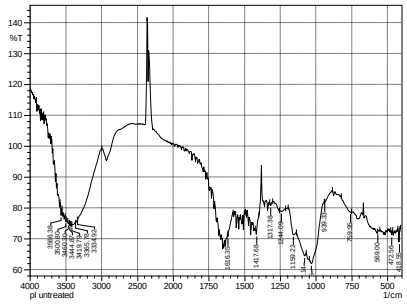

Fig.1 (a) FTIR spectrum of MR dye untreated with pterocladia lucida powder

The new peaks are observed after biosorption at 3328.31, 3357.25 and 3385.22 cm-1 due to the involvement of the stretching vibration bands og hydroxyl and –OH stretching or –NH2 stretching functional groups respectively in the ion-exchange process.

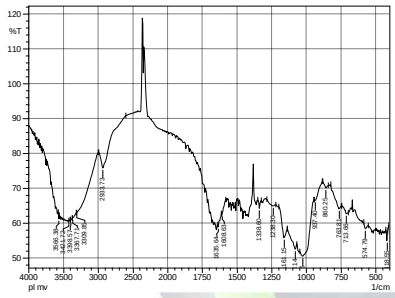

Fig. 1 (b) FTIR spectrum of MR dye treated pterocladia lucida powder

Further, the peaks at 3502.88 and 3523.13 cm-1, attributed to O-H stretching modes and are not seen in untreated biomass.

3.2 X-Ray Diffraction:

XRD patterns shown in figs 2 (a) & (b) for untreated powder do not show very keen or pointed and distinct peaks and exhibits roughly amorphous nature. The peaks at 2θ values of 0.7748, 0.7273, 0.7273, 0.7159 and 0.7035 corroborate the presence of Fe2H474K44, Eu8K16.5O206, As6ClCS3.9, H168K3Li5.5 and C40K13O368 (ICDD files). Their corresponding d-values are 5.5771, 5.1148, 5.8082, 6.4302 and 6.6466.

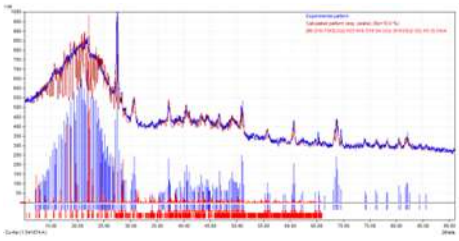

Fig. 2 (a) XRD pattern of MR dye untreated pterocladia lucida powder

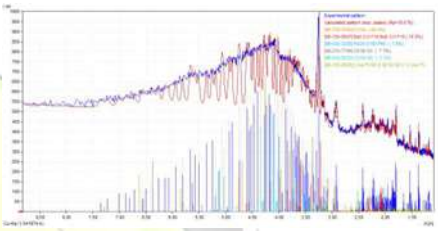

Fig. 2 (b) XRD pattern of MR dye untreated pterocladia lucida powder with matching compounds

3.2.2 XRD Pattern of MR dye treated pterocladia lucida powder

XRD patterns of treated MR dye, shown in figs 2 (c) & (d), show very violent and intense and well defined peaks and exhibits precisely amorphous nature. The peaks at 2θ values of 0.8662, 0.8333, 0.7447, 0.7717 and 0.7310 corroborate the presence of Ba8S15Sn4, Fe39Sb9Se4, As14Cs4Zn, Cs2O7S2 and Br10Te8U2. Their corresponding d-values are 3.5370, 3.1420, 4.1371, 3.8977 and 3.7049 respectively.

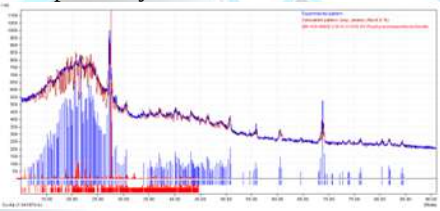

Fig. 2 (c) XRD pattern of MR dye treated pterocladia lucida powder

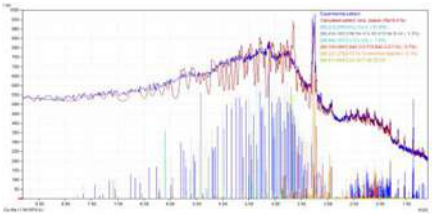

Fig. 2 (d) XRD pattern of MR dye treated pterocladia lucida powder with matching compounds

ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
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International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 5, Issue 4, April 2018

3.3 Scanning Electron microscope (SEM):

The SEM micrographs of pterocladia lucida powder before and after biosorption are analyzed. The SEM images in fig. 3 (a) show that the algae powder is less uneven and porous.

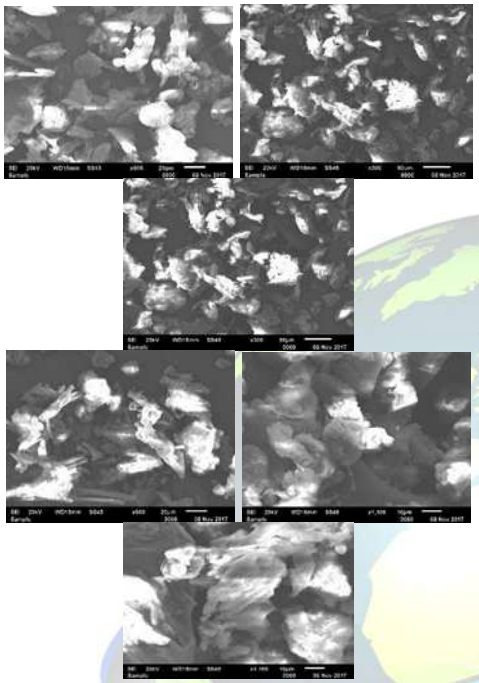

Fig. 3 (a) SEM pattern of untreated pterocladia lucida powder

3.3.2 SEM pattern of treated pterocladia lucida powder

The surface area analysis after MR dye loading confirms the increased surface area and porosity. The surface has a greater potential to biosorb MR dye. After biosorption as per fig. 3 (b), the particles have granular, complex, uneven and porous surface texture that is not found in the original untreated pterocladia lucida powder.

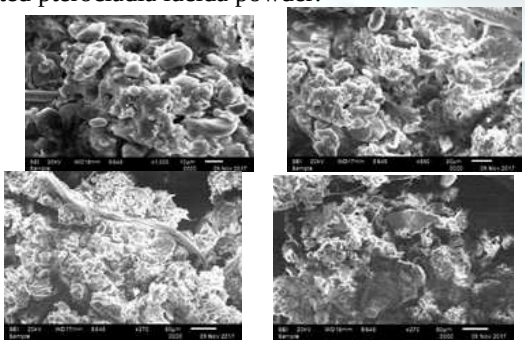

Fig. 3 (b) SEM pattern of treated pterocladia lucida powder with MR dye

3.4 Equilibrium in studies on biosorption of MR dye

3.4.1 Effect of agitation time:

The effect of agitation time on the removal of MR dye onto pterocladia lucida powder was studied at dosage of 0.5g/L. The percentage biosorption is shown against agitation time in fig. 4. The maximum percentage of biosorption is attained at 30 min of agitation and becomes constant after 30 min. indicating the attainment of the equilibrium (58 %) [11-20].

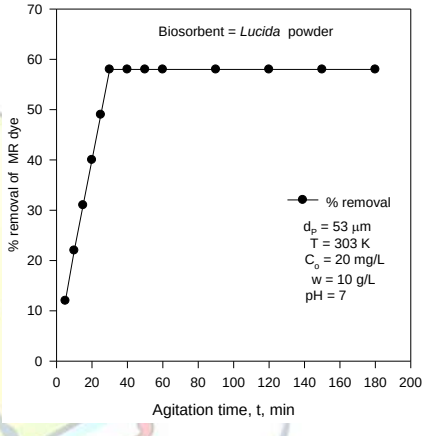

Fig. 4 Effect of agitation time on % biosorption of MR dye

3.4.2 Effect of biosorbent size:

Fig. 5 indicates percentage biosorption of MR dye as a function of particle size. The percentage biosorption is increased from 28 % to 58 % as the biosorbent size is decreased from 152 to 53 µm [21-30].

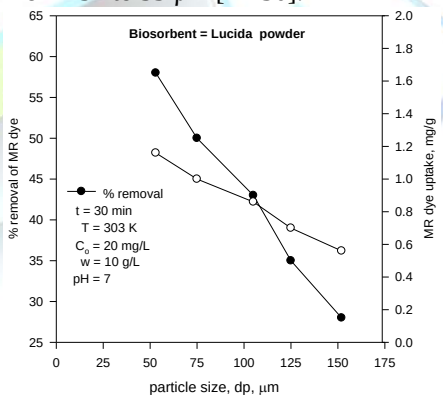

Fig. 5 Effect of biosorbent size on % biosorption of MR dye

3.4.3 Effect of pH:

The effect of pH of aqueous solution on percentage biosorption of MR dye is drawn in fig. 6. The percentage biosorption is increased from 56 % to 75 % as pH is

ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
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International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
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increased from 2 to 5. The percentage biosorption is decreased from 75 % to 50 % as pH increases from 5 to 8. The maximum value is obtained at pH 5 [31–40].

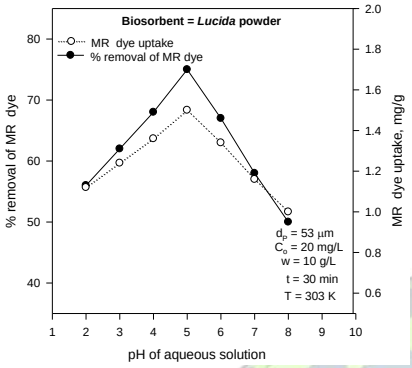

Fig. 6 Effect of pH on % biosorption of MR dye

3.4.4 Effect of initial concentration of MR dye:

A graph is drawn in fig. 7 with percentage biosorption of MR dye as a function of initial concentration of MR dye. The percentage biosorption is decreased from 75 % to 51 % as the initial concentration of MR dye in the aqueous solution increases from 20 mg/L to 200 mg/L [41–50].

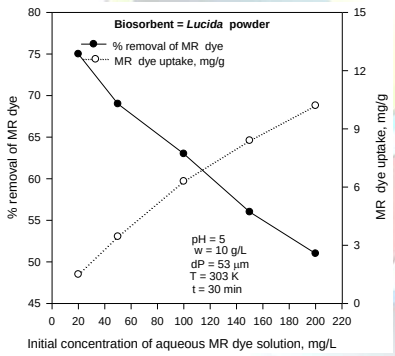

Fig. 7 Effect of initial concentration for the biosorption of MR dye

3.4.5 Effect of biosorbent dosage:

The percentage biosorption of MR dye is drawn against biosorbent dosage in fig. 8 For a biosorbent size of 53µm, percentage biosorption increases from 75 % to 85 %, as dosage is increased from 10 to 25 g/L [51–60].

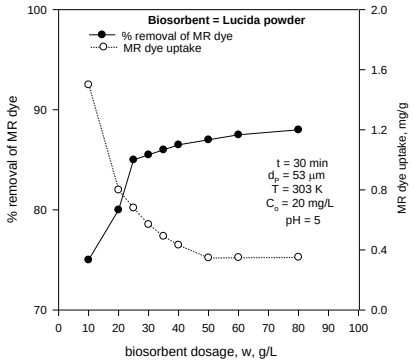

Fig. 8 Effect of biosorbent dosage on % biosorption of MR dye

3.4.6 Effect of temperature:

The effect of temperature on the equilibrium dye uptake was significant. The effect of changes in the temperature on the MR dye uptake is shown in Fig. 9. The biosorption capacity of dye is increased at higher temperatures, which indicates that biosorption of dyes in this system is an endothermic process [61–70].

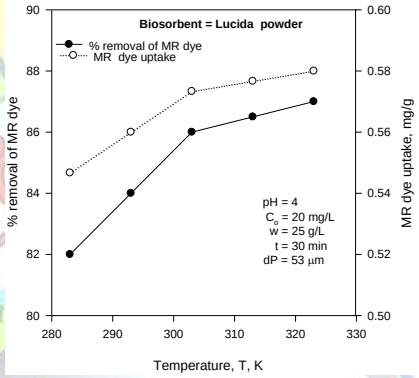

Fig. 9 Effect of temperature for the biosorption of MR dye

3.5 Isotherms

3.5.1 Langmuir Isotherm:

Langmuir isotherm, drawn in fig. 10, for the present data has yielded the equation:

(Ce/qe) = 0.06627 Ce + 3.2969 R2 = 0.9922 ------- (1)

The correlation coefficient value of 0.9922 indicates strong binding of MR dye [71–80] on to the biosorbent.

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ISSN 2394-3785 (Online)
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International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 5, Issue 4, April 2018

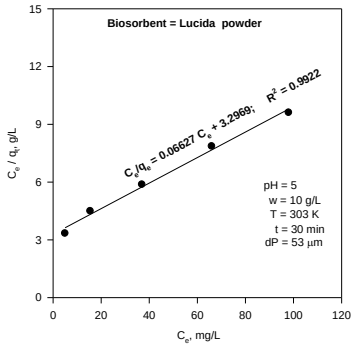

Fig. 10 Langmuir isotherm for biosorption of MR dye

3.5.2 Freundlich Isotherm:

Fig. 11, drawn between ln Ce and ln qe, has resulted the equation:

ln qe = 0.6488 ln Ce – 0.5848

------ (2)

The equation has a correlation coefficient of 0.9932. The 'n' value of 0.6488 satisfies the condition of 0<n<1, indicating favorable biosorption [81–90].

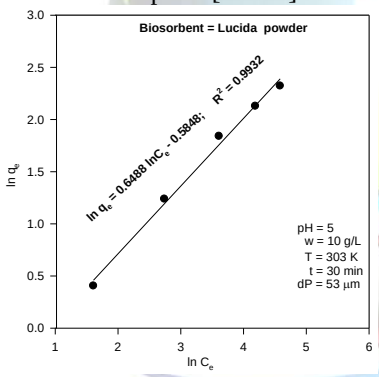

Fig. 11 Freundlich isotherm for biosorption of MR

3.5.3 Temkin Isotherm:

The present data are analysed according to the linear form. The linear plot of Temkin isotherm [91–100] is shown in fig. 12. The equation obtained for MR dye biosorption is:

qe = 2.9244 ln Ce – 3.8185

------ (3)

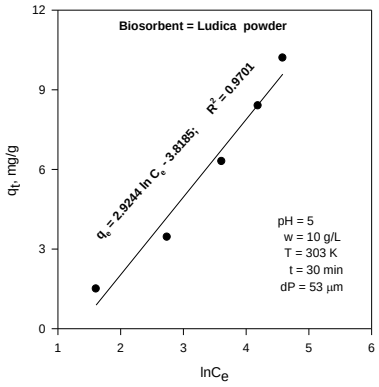

Fig. 12 Temkin isotherm for biosorption of MR dye

With a correlation coefficient 0.9701. The isotherm constants of the three isotherms are compiled in table-5.11. The equilibrium data are well explained by Langmuir isotherm (0.9922), Temkin (0.9701) and Freundlich isotherm (0.9932).

3.6 Kinetics:

Lagergren plot of log (qe-qt) vs agitation time (t) is shown in fig. 13 and pseudo second order kinetics plot between 't' vs 't/qt' for biosorption of MR dye is drawn in fig. 14. summarizes rate constant values for first and second order rate equations [101–120].

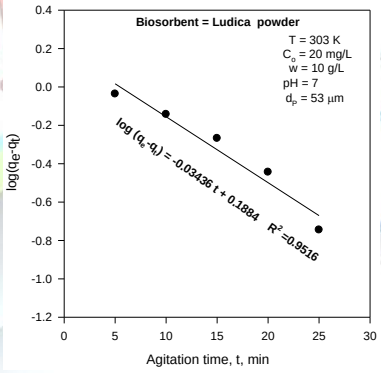

Fig. 13 First order kinetics for biosorption of MR dye

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International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 5, Issue 4, April 2018

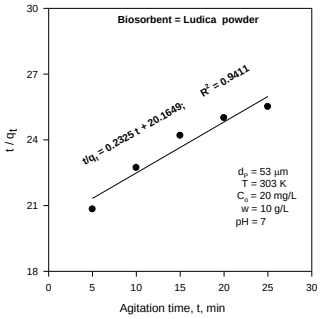

Fig. 14 Second order Kinetics for biosorption of MR dye

3.7 Thermodynamics:

A series of thermodynamic parameters - change in Gibbs free energy (ΔG) change in enthalpy (ΔH) and change in entropy (ΔS) are determined (Fig. 15). ΔG value of -3279.75 J/mole indicates that biosorption of MR dye by pterocladia lucida powder could take place spontaneously. Positive ΔH of 7.43100 J/mole indicates indicates endothermic nature of biosorption while positive ΔS = 10.84877 J/mole-K demonostrates the affinity of pterocladia lucida powder to MR dye [121–130].

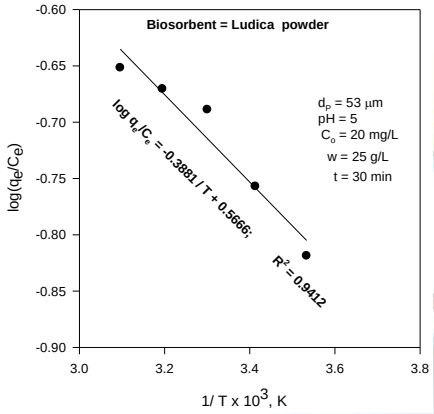

Fig. 15 Vant Hoff's plot for biosorption of MR dye

3.8 Optimization using Response Surface Methodology (RSM):

3.8.1 Optimization using CCD

In the present study, the levels of four process input variables for % biosorption are shown in table–2.

TABLE – 2

LEVELS OF DIFFERENT PROCESS VARIABLES IN CODED AND UN-CODED FORM FOR

% BIOSORPTION OF MR DYE USING PTEROCLADIA LUCIDA POWDER

Variable	Name	Range and 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	15	20	25	30	35
X4	Temperature, K	283	293	303	313	323

The % biosorption of MR dye (Y) 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 depending on experimental runs and predicted values proposed by CCD design. The following equation represents multiple regression analysis of the experimental data:

Y = –1104.08 + 39.41 X1 + 2.92 X2 + 2.37 X3 + 6.72 X4 – 2.19 X12 –0.05 X22 – 0.04X32 – 0.01 X42 – 0.012 X1X2 – 0.08 X1X3 – 0.04 X1X4 + 0.02 X2X3 - 0.00 X2X4 – 0.00 X3X4 ----- (4)

TABLE - 3

RESULTS FROM CCD FOR MR DYE BIOSORPTION BY PTEROCLADIA LUCIDA POWDER

Run No.	X1, pH	X2, Co	X3, w	X4, T	% biosorption of MR dye	
					Experimental	Predicted
1	-1	-1	-1	-1	81.52000	81.49750
2	-1	-1	-1	1	84.62000	84.60583
3	-1	-1	1	-1	81.78000	81.78583
4	-1	-1	1	1	84.58000	84.55417
5	-1	1	-1	-1	83.72000	83.67250
6	-1	1	-1	1	86.18000	86.24083
7	-1	1	1	-1	86.12000	86.09083
8	-1	1	1	1	88.32000	88.31917
9	1	-1	-1	-1	86.42000	86.39583
10	1	-1	-1	1	87.88000	87.89417
11	1	-1	1	-1	85.22000	85.14417
12	1	-1	1	1	86.28000	86.30250
13	1	1	-1	-1	86.22000	86.23083
14	1	1	-1	1	87.22000	87.18917

ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
Available online at www.ijartet.com

International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 5, Issue 4, April 2018

15	1	1	1	-1	87.12000	87.10917
16	1	1	1	1	87.72000	87.72750
17	-2	0	0	0	80.08000	80.09667
18	2	0	0	0	84.38000	84.40333
19	0	-2	0	0	84.48000	84.52000
20	0	2	0	0	88.12000	88.12000
21	0	0	-2	0	86.88000	86.88667
22	0	0	2	0	87.68000	87.71333
23	0	0	0	-2	84.88000	84.95667
24	0	0	0	2	88.72000	88.68333
25	0	0	0	0	91.00000	91.00000
26	0	0	0	0	91.00000	91.00000
27	0	0	0	0	91.00000	91.00000
28	0	0	0	0	91.00000	91.00000
29	0	0	0	0	91.00000	91.00000
30	0	0	0	0	91.00000	91.00000

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. 1 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 4). It shows that the treatment differences are significant.

TABLE-4

ANOVA OF MR DYE BIOSORPTION FOR ENTIRE QUADRATIC MODEL

Source of variation	SS	df	Mean square(MS)	F-value	P > F
Model	252.933	14	18.0666	9926.70	0.000
Error	0.0273	15	0.00182		
Total	252.9603				

df-, degree of freedom; SS- sum of squares; F- factor F; P- probability

R2=0.99219; R2 (adj):0.9849:

It is predicted from table-5 that the larger the value of t and smaller the value of P, the more significant is the corresponding coefficient term. The't' and 'P' values are analyzed from table-5.16 to predict the response. It is found that X1, X2, X3, X4, X12 , X22 , X32 , X42 X1X2, X1X3, X1X4, X2X3, X2X4 have high significance to explain the individual and interaction effects of input variables on biosorption of MR dye.

TABLE-5

REGRESSION COEFFICIENTS

Term	Regression	Standard error	T(15)	p
Mean/Intercept	-1104.08	8.011570	-137.81	0.000000
Dosage, w, g/L (L)	39.41	0.340305	115.81	0.000000
Dosage, w, g/L (Q)	-2.19	0.008146	-268.54	0.000000
Conc, Co, mg/L (L)	2.92	0.067659	43.196	0.000000
Conc, Co, mg/L (Q)	-0.05	0.000326	-143.63	0.000000
pH (L)	2.37	0.068061	34.755	0.000000
pH (Q)	-0.04	0.000326	-113.55	0.000000
Temperature, T, K (L)	6.72	0.050126	134.135	0.000000
Temperature, T, K (Q)	-0.01	0.000081	-128.28	0.000000
1L by 2L	-0.12	0.002133	-54.850	0.000000
1L by 3L	-0.08	0.002133	-36.098	0.000000
1L by 4L	-0.04	0.001067	-37.739	0.000000
2L by 3L	0.02	0.000427	49.928	0.000000
2L by 4L	-0.00	0.000213	-12.658	0.000000
3L by 4L	-0.00	0.000213	-7.970	0.000001

ainsignificant ($P \geq 0.05$)

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

$Y = -1104.08 + 39.41\ X1 + 2.92\ X2 + 2.37\ X3 + 6.72\ X4 - 2.19\ X12 - 0.05\ X22 - 0.04X32 - 0.01\ X42 - 0.012\ X1X2 - 0.08\ X1X3 - 0.04\ X1X4 + 0.02\ X2X3$ ------- (5)

The regression coefficient value of 0.99996 indicates that 0.004 % of the total variations are not satisfactorily explained by the model [131–140]. 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 MR dye can be adequately

ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
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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 (Fig. 16). The % biosorption prediction from the model is shown in table-5. It is implied from table-5 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.

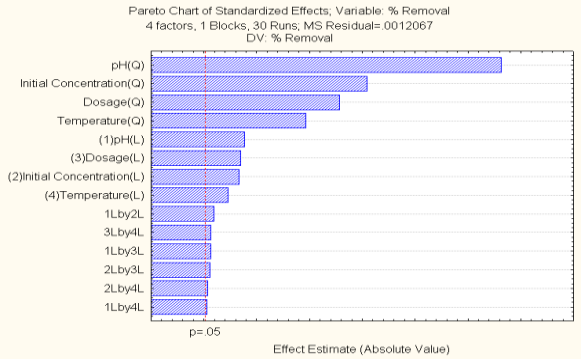

Fig. 16 Pareto Chart

5.12.2 Interpretation of residual graphs:

Fig. 17 shows normal probability plot of residual values. The experimental values are in good agreement with predicted values with minimum error.

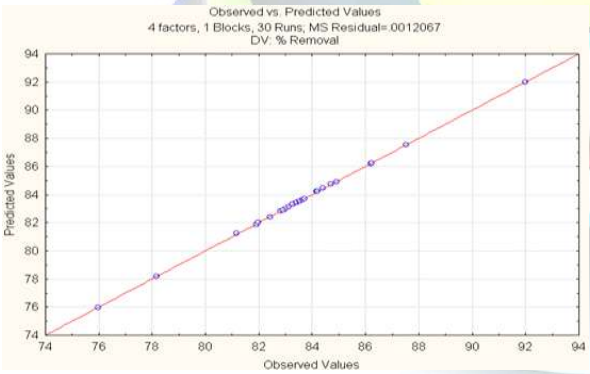

Fig. 17 Normal probability plot for % biosorption of MR dye

5.12.3 Interaction effects of biosorption variables:

Figs. 18 (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. The predicted optimum values for percentage biosorption of MR are:

pH = 5.1471,
Initial MR dye concentration = 21.8188 mg/L
Biosorbent dosage = 25.8401 g/L,
Temperature = 306.8712 K
% MR dye biosorption = 91.55044

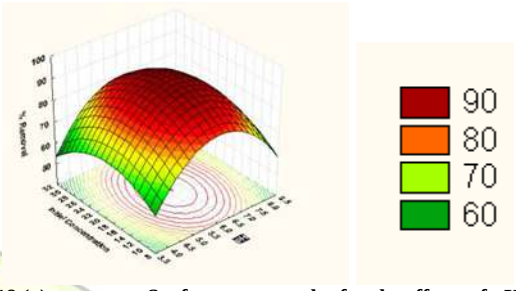

Fig. 18 (a) Surface contour plot for the effects of pH and initial concentration of MR dye on % biosorption

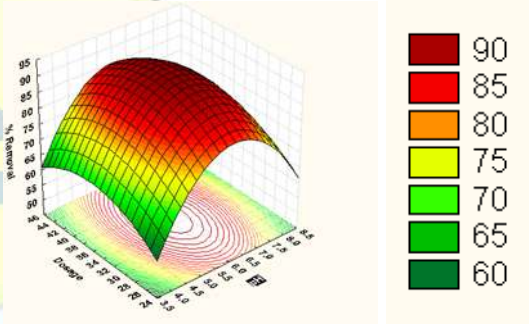

Fig. 18 (b) Surface contour plot for the effects of pH and dosage on % biosorption of MR dye

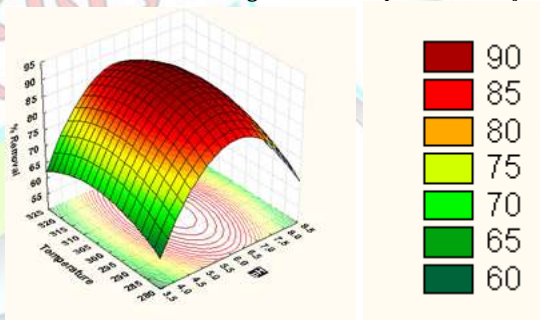

Fig. 18 (c) Surface contour plot for the effects of pH and Temperature on % biosorption of MR dye

ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
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International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 5, Issue 4, April 2018

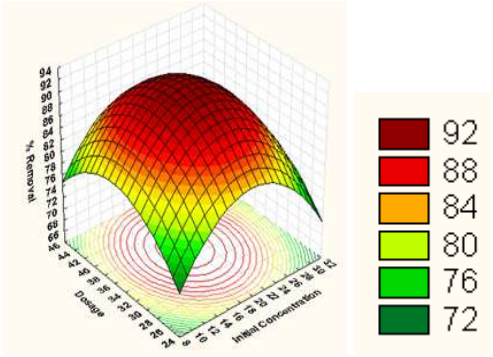

Fig. 18 (d) **Surface contour plot for the effects of initial concentration and Dosage on % biosorption of MR dye**

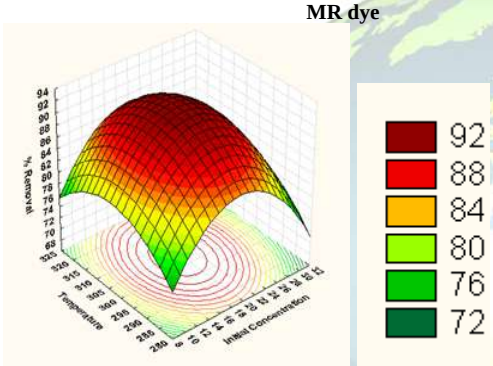

Fig. 18 (e) **Surface contour plot for the effects of initial concentration and Temperature on % biosorption of MR dye**

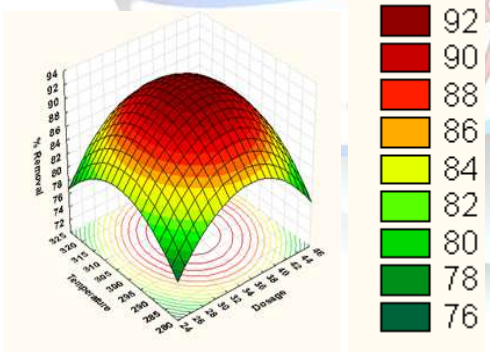

Fig. 18 (f) **Surface contour plot for the effects of Dosage and Temperature on % biosorption of MR dye**

TABLE – 6

COMPARISON BETWEEN OPTIMUM VALUES FROM CCD AND EXPERIMENTATION

Variable	CCD	Experimental value
pH of aqueous solution	5.1471	5
Initial MR dye concentration, mg/L	21.8188	20
Biosorption dosage, w, g/L	25.8401	30
Temperature, K	306.8712	303
% biosorption	91.55044	94.9

The present MR dye biosorption (Table 6) uptake capacities are compared in table–7 with those of other biosorbents.

TABLE – 7

MR DYE UPTAKE CAPACITIES FOR DIFFERENT BIOSORBENTS

Authors	Biosorbent	q_t, mg/g
Fatih Deniz et al [141]	agro-residue	40
A. Hebeish [142]	Sawdust	65.8
A.M.Ben Hamissa et al [143]	Agave americana (L.) fibres	21.41
Ana Méndezet al [144]	organic sludge from virgin pulp mill	435
Hameed BH et al [145]	novel agricultural waste adsorbent	119.05
Bao-E. Wang et al [146]	Aspergillus fumigatus beads	31.5
Bellir Karima et al [147]	Acid Activated Algerian Bentonite	56.34
P. Senthhilkumar et al [148]	silver wood sawdust carbon	29.107
R. Naveen Prasad et al [149]	Coir Pith	31.847
Sahadevan Renganathan et al [150]	Petiole	25.5
Present investigation	pterocladia lucida powder	15.0897

ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
Available online at www.ijartet.com

International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 5, Issue 4, April 2018

ACKNOWLEDGEMENT

The Author expresses his deep sense of gratitude to Andhra University and Department of Chemical Engineering for providing chemicals, equipment and laboratory facilities. The Author also expresses his sincere thanks to UGC-RGNF for providing financial support.

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