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ISOTHERMAL AND KINETICS OF BIOSORPTION CAPACITY OF AMIDO BLACK DYE USING CHAETOMORPHA ANTENNINA GREEN ALGAE POWDER AND OPTIMIZATION USING CENTRAL COMPOSITE DESIGN

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Abstract:
Biosorption is the property shown by idle, non-living substances of organic beginning to tie and to gather metal particles from watery arrangement. Rapid progress in industrialization in the last few years has increased the discharge of pollutants into the environment. Discard of municipal and other industrial wastes into water bodies causes water pollution. Industries release non-treated and/or incompletely treated wastewater into the environment which leads to water and soil pollution. Mostly pharmaceutical, textile, food, paper & pulp and cosmetic industries use synthetic dyes for the manufacturing process. In the present study by using biosorption removal of dye by using chaetomorpha antennnina and optimization was also incorporated using central composite design (CCD). The kinetics and isotherms studies are also studies along with thermodynamic study. Response Surface Methodology (RSM) is a proficient factual instrument for streamlining of various factors. To depict the reaction surfaces, a five-level, four-variable focal composite plan (CCD) was taken on in this review. The four autonomous factors and their levels for the 27 examinations in the CCD. Reaction Surface Methodology (RSM) is an assortment of measurable and numerical procedures valuable for creating, improving, and enhancing measures.

Keywords: Amido Black, Chaetomorpha Antennina, Biosorbent, Central Composite Design

1. INTRODUCTION

Various reports are available which show harmful effects of azo dyes on plants (plant growth and germination). Industrial pollutants are one of the biggest environmental problems due to their effect on groundwater and surface water along with human health. Various technologies such as advanced oxidation process, membrane filtration technique, microbial technologies, bio-electrochemical degradation, photocatalytic degradation have been reported for the treatment of dye industry wastewater. The composition of industrial wastewater is based on various types of raw materials, chemicals, organic-based compounds, and different types of dyes used in wet and dry processing phases. The industrial manufacturing procedure discards unsafe and colored dyes mostly azo dyes. Biosorption enjoys particular upper hands over the traditional strategies: the cycle doesn't deliver

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synthetic oozes, it very well may be exceptionally specific, more proficient, simple to work, and consequently financially savvy for the treatment of huge volumes of wastewaters containing low contamination fixations. The utilization of inactivated biomass is likewise invaluable as the cycle is liberated from supplement supply and there is no poisonousness requirements in the organic entity utilized.

II. EXPERIMENTAL PROCEDURE

A. Preparation of the Biosorbents

The proposed biosorbents are Chaetomorpha Antennina. This biosorbent is widely available and collected from the banks of the Godavari River in Rajahmundry, Andhra Pradesh, India. The collected plants are cleaned until the dirt is removed with distilled water. The cleaning is then continued with the process of drying the plants under the hot sun so that the plants become too dry so that they can only be torn apart by squeezing the palms of the hands. The dried leaves were then ground into powder and then sieved with various sizes such as 53 µm, 75 µm, 105 µm, 125 µm and 152 µm. So the sifted powder, without any preventive treatment, is used as a biosorbent

B. Preparation of 1000 Mg/L of Amido Black dye stock solution

Sources for the manufacture of dye stock solutions are Amido Black dye powders. Double distilled water was used for the preparation of the required solution. Amido Black stock solution with a concentration of 1000 mg/L was prepared by dissolving 1 g of Amido Black dye in 1 L of distilled water. Then the aquadest is poured up to the mark of the 1000 ml volumetric flask so that the solution is made or made. In the same way, all other solutions were also prepared whose concentrations varied accordingly [(20, 50, 100, 150, 200) mg/L].

C. Studies on Equilibrium Biosorption Process

The biosorption was carried out in a batch process by adding a pre-weighed amount of the Chaetomorpha Antennina green 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, which include characterization (FTIR, XRD), 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.

III. RESULTS AND DISCUSSIONS

In the present investigation, the prospectives of sorbents namely Chaetomorpha Antennina green algae powder were evaluated to estimate their performance for the decolorization of Amido Black dye present in aqueous solutions. The effects of parameters on decolorization of Amido Black dye were measured, data consisting of effect of agitation time, sorbent size, pH of the solution, initial concentration, sorbent dosage and temperature.

A. Sorption of Amido Black dye using Chaetomorpha Antennina green algae powder.

1. Equilibrium Isotherm Models

A). Langmuir isotherm is the most widely used simple two- parameter equation. This simple isotherm is based on following assumptions:

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- Adsorbate is chemically adsorbed at a fixed number of well- defined sites
- Each site can hold only one adsorbate species
- All sites are energetically equivalent
- There are no interactions between the adsorbate species

The Langmuir relationship is hyperbolic and the equation is:

$q_e/ q_m = K_LC_e/ (1+K_LC_e)$

The above equation can be rearranged as

$(C_e/q_e) = 1/(K_Lq_m) + C_e/q_m$

From the plots between (C_e/q_e) and C_e, the slope $\{1/ (K_Lq_m)\}$ and the intercept $(1/q_m)$ can be calculated. Further analysis of Langmuir equation can be made on the basis of separation factor $[R_L =1/ (1+K_LC_e)]$.

$0< R_L<1$	indicates	favorable adsorption
$R_L>1$	indicates	unfavorable adsorption
$R_L = 1$	indicates	linear adsorption
$R_L = 0$	indicates	irrepressible adsorption

Langmuir isotherm is drawn between C_e/q_e and C_e in fig. 1.1 for the present data. The resulting equation is

$$(C_e/q_e) = 0.0567\ C_e + 4.564$$

The (correlation coefficient of 0.9795) confirms strong binding of Amido Black ions to the surface of Chaetomorpha Antenninaa powder.

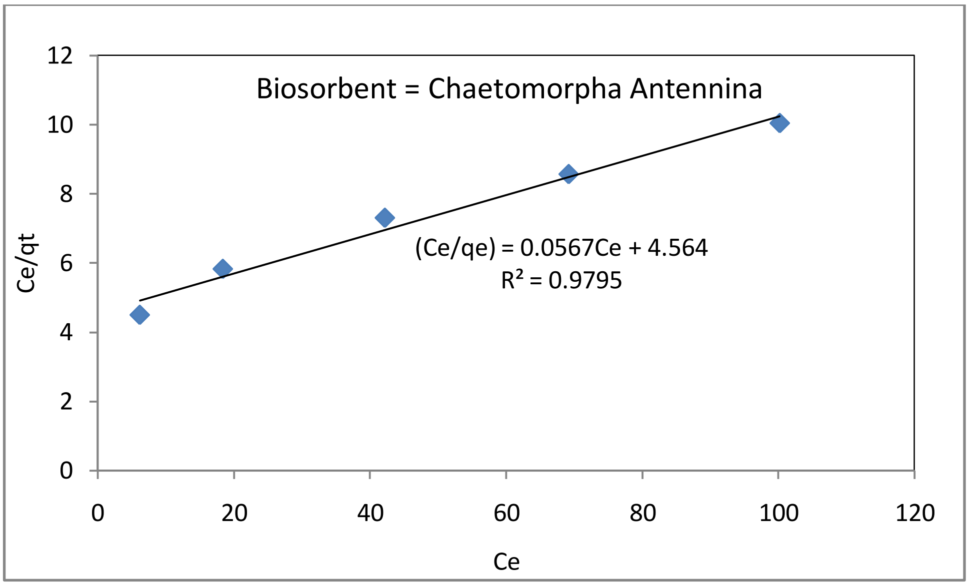

Fig. 1.1 Langmuir isotherm for removal of Amido Black

b). Freundlich isotherm

Freundlich presented an empirical adsorption isotherm equation that can be applied in case of low and intermediate concentration ranges. The Freundlich isotherm is given by

$q_e = K_fC_e^n$

Where $K_{f,}$ mg/g represents the adsorption capacity at dye equilibrium concentration and n represents the degree of dependence of adsorption

Taking LN on both sides, we get

$$\ln q_e = \ln K_f + n \ln C_e$$

Freundlich isotherm is drawn between $\ln C_e$ and $\ln q_e$ in fig. 1.2, resulted in the following equation

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$\ln q_e = 0.7168 \ln C_e - 0.9602$

The equation has a correlation coefficient of 0.9984. The 'n' value of 0.600508 indicates favorable biosorption satisfying the condition of 0<n<1.

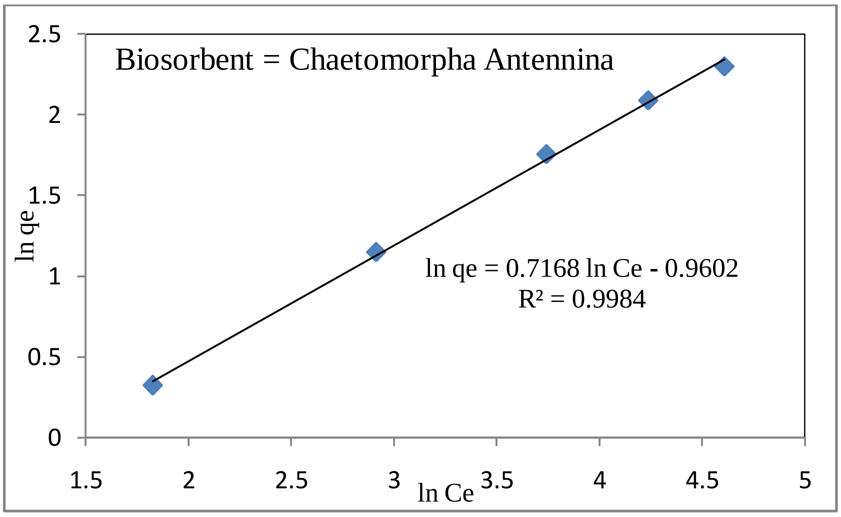

Fig.1.2 Freundlich isotherm for removal of Amido Black

c) Temkin isotherm:

Temkin and Pyzhev isotherm equation describes the behavior of many adsorption systems on heterogeneous surface and is based on the equation:

$q_e = RT \ln(A_T C_e)/b_T$

The linear form of Temkin isotherm is

$q_e = (RT/ b_T) \ln(A_T) + (RT/b_T) \ln(C_e)$

Where $A_T = \exp [b (0) \times b (1) / RT]$

Slope, $b (1) = RT/ b_T$

Intercept, $b (0) = (RT/ b_T) \ln (A_T)$

Plot between q_e and $\ln C_e$ is shown in fig. 1.3. The equation for Amido Black biosorption is

$q_e = 3.0636 \ln C_e – 4.9385$

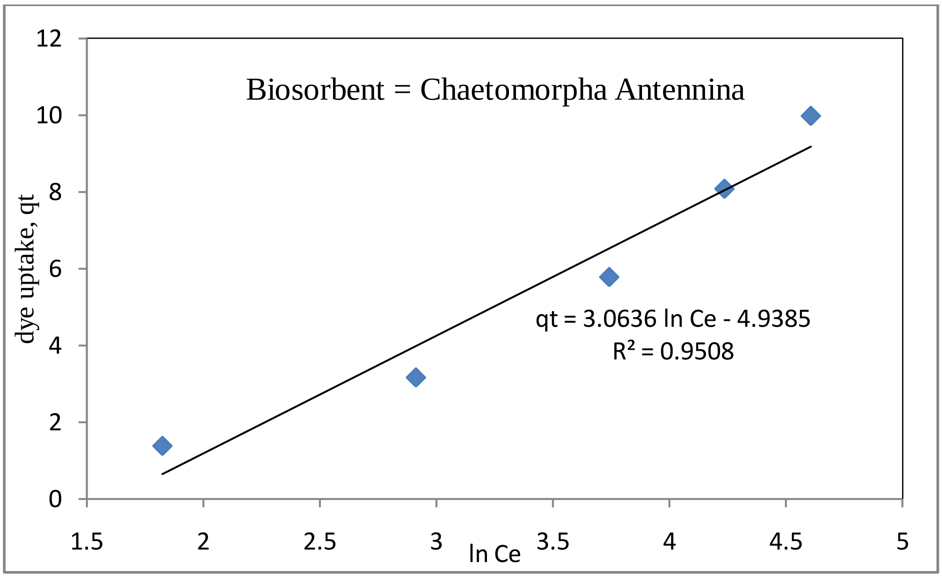

Fig. 1.3 Temkin isotherm for removal of Amido Black

The isotherm constants are compiled in table-1.1. It is found that biosorption data are well represented by Langmuir isotherm (R^2=0.9795), Temkin (R^2=0.9508) and Freundlich isotherms (R^2=0.9984).

Table - 1.1 Isotherm constants

Langmuir isotherm	Freundlich isotherm	Temkin isotherm
q_m = 17.6366 mg/g	K_f=0.3828 mg/g	A_T = 0.1994 L/mg
K_L = 0.0124	n = 0.7168	b_T= 822.2816
R^2 = 0.9284	R^2= 0.9984	R^2 = 0.9508

2. **Biosorption Kinetics**

Lagergren first order

Lagergren plot and pseudo second order kinetics plot for biosorption of Amido Black are drawn in figs. 1.4 & 1.5. Table-1.2 summarizes the 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 satisfactorily

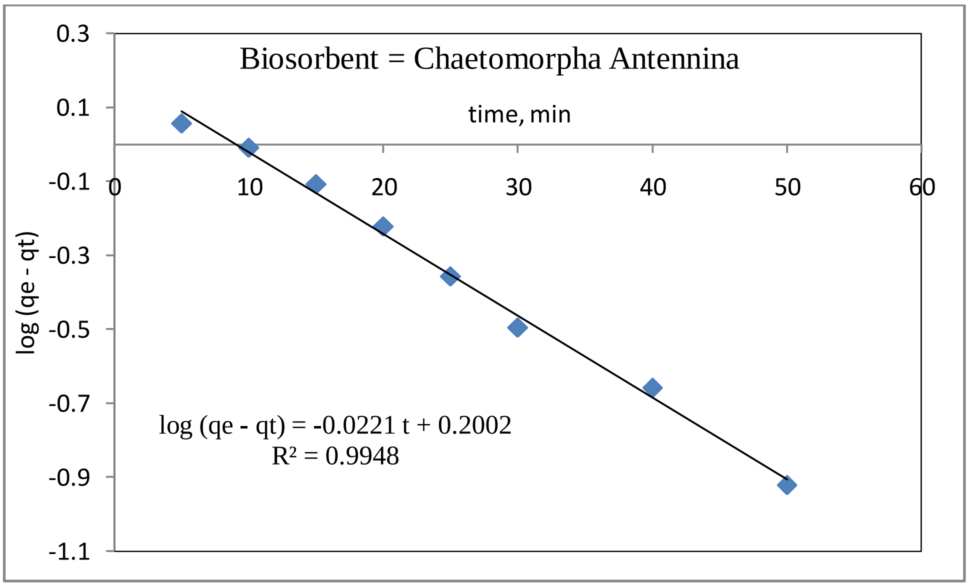

Fig.1.4. First order kinetics for removal of Amido Black

Pseudo second order

Second request rate conditions clarify the biosorption cooperation agreeably. The rate consistent qualities for second request rate conditions

$$t/qt = 0.3834t + 18.846,\ R^2=0.9344$$

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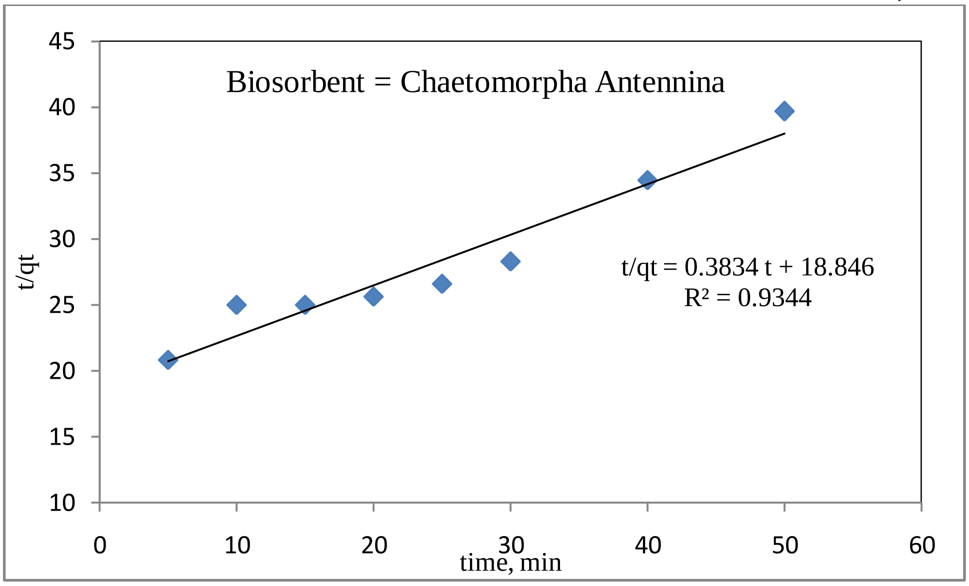

Fig.1.5. Second order Kinetics for removal of Amido Black

Table – 1.2 Equations and rate constants

Order	Equation	Rate constant	R^2
Lagergren first order	log (qe - qt) = -0.0221 t + 0.2002	0.0508 min^{-1}	0.9948
Pseudo second order	t/qt = 0.3834 t + 18.846	0.0078 g/ (mg-min)	0.9344

3. Thermodynamics of Sorption

Van'tHoff's plot is drawn in fig. 1.6. from the data, Gibbs free energy change (ΔG) is calculated to be -10319.6 J/mol for biosorption of Amido Black. The negative ΔG value indicates thermodynamically feasible and spontaneous nature of biosorption. The ΔH parameter is 13.6845 kJ/mol.K. The negative ΔH indicates the endothermic nature of biosorption. ΔS parameter is found to be 34.0129 J/mol K for Amido Black dye biosorption. The positive ΔS value suggests an increase in the randomness at the solid /solution interface during biosorption.

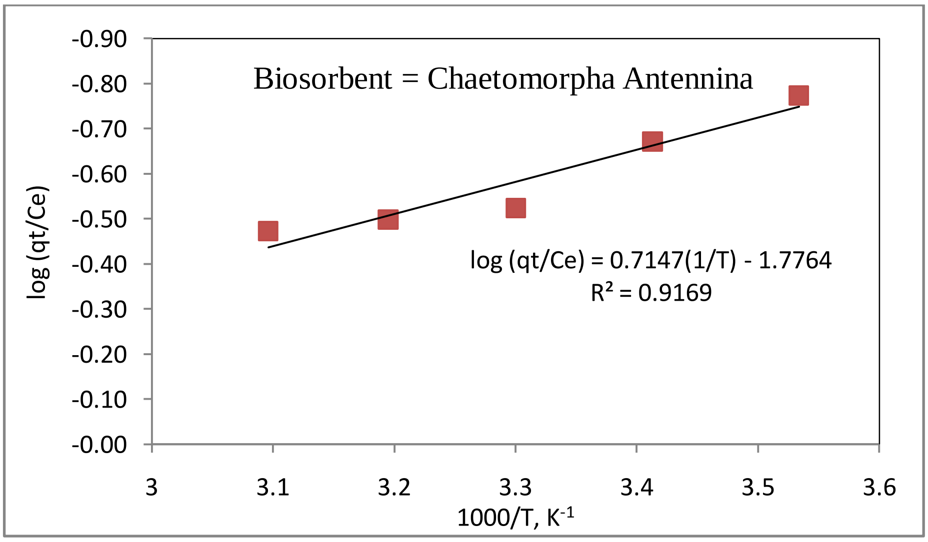

Fig.1.6. Vant Hoff's plot for removal of amido black

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4. Optimization using Response Surface Methodology (RSM)

A). Optimization of the Selected Parameters using CCD

The impacts of four autonomous factors (biosorbent dose, starting centralization of Amido Black in watery arrangement, pH and temperature) on Amido Black biosorption are examined utilizing Central Composite Design (CCD). The ideal conditions for the four autonomous factors on the degree of Amido Black biosorption are shaped inside the quadratic model. Levels of various cycle factors for rate biosorption are displayed in table–1.3.

Table–1.3 Levels of different process variables in coded and un-coded form for % Biosorption of Amido Black using Chaetomorpha Antennina powder

Variable	Name	Range and levels				
		-2	-1	0	1	2
X_1	Biosorbent dosage, w, g/L	25	30	35	40	45
X_2	Initial concentration, C_o, mg/L	10	15	20	25	30
X_3	pH of aqueous solution	5	6	7	8	9
X_4	Temperature, T, K	283	293	303	313	323

Relapse condition for the streamlining of biosorption is:

% Biosorption of Amido Black (Y) is capacity of measurements (X_1), beginning focus (X_2), pH of fluid arrangement (X_3), and Temperature of watery arrangement (X_4).

The different relapse examination of the trial information has yielded the accompanying condition:

$$Y = -2670.16+52.41X_1+3.93X_2 + 8.52X_3 + 15.61X_4 - 3.90X_1^2 - 0.13X_2^2 - 0.12X_3^2 - 0.03X_4^2 - 0.01X_1X_2 + 0.03X_1X_3 + 0.01X_1X_4 + 0.03X_2X_3 + 0.00X_2X_4 - 0.00X_3X_4 \text{-----} \quad (5.10)$$

Table-1.3 addresses the outcomes acquired in CCD. The reaction acquired as examination of difference (ANOVA) from relapse eq.1.10 is assembled in table–1.4. Fischer's 'F-insights' worth is characterized as MSmodel/MSerror, where MS is mean square. Fischer's 'F-insights' worth, having a low likelihood 'p' esteem, demonstrates high importance.

Table–1.4 Results from CCD for Amido Black biosorption by Chaetomorpha Antennina powder

Run No.	X_1, w	X_2, C_o	X_3, pH	X_4, T	% Biosorption of Amido Black	
					Experimental	Predicted
1	-1 (20)	-1 (15)	-1 (4)	-1 (293)	93.10	93.53571
2	-1 (20)	-1 (15)	-1 (4)	1 (313)	93.03	93.02117
3	-1 (20)	-1 (15)	1 (6)	-1 (293)	91.20	91.35117
4	-1 (20)	-1 (15)	1 (6)	1 (313)	91.71	91.55438
5	-1 (20)	1 (25)	-1 (4)	-1 (293)	92.01	92.16800
6	-1	1 (25)	-1 (4)	1 (313)	92.58	92.37071

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	(20)					
7	-1 (20)	1 (25)	1 (6)	-1 (293)	90.89	90.74071
8	-1 (20)	1 (25)	1 (6)	1 (313)	91.22	91.66117
9	1 (40)	-1 (15)	-1 (4)	-1 (293)	93.23	92.98650
10	1 (40)	-1 (15)	-1 (4)	1 (313)	93.15	93.20421
11	1 (40)	-1 (15)	1 (6)	-1 (293)	91.77	91.88421
12	1 (40)	-1 (15)	1 (6)	1 (313)	92.78	92.81967
13	1 (40)	1 (25)	-1 (4)	-1 (293)	92.30	92.36154
14	1 (40)	1 (25)	-1 (4)	1 (313)	93.25	93.29650
15	1 (40)	1 (25)	1 (6)	-1 (293)	91.81	92.01650
16	1 (40)	1 (25)	1 (6)	1 (313)	94.20	93.66921
17	-2 (10)	0 (20)	0 (5)	0 (303)	92.00	91.71979
18	2 (50)	0 (20)	0 (5)	0 (303)	93.00	93.17862
19	0 (30)	-2 (10)	0 (5)	0 (303)	93.30	93.15779
20	0 (30)	2 (30)	0 (5)	0 (303)	92.60	92.63962
21	0 (30)	0 (20)	-2 (3)	0 (303)	91.50	91.40462
22	0 (30)	0 (20)	2 (7)	0 (303)	89.60	89.59279
23	0 (30)	0 (20)	0 (5)	-2 (283)	93.56	93.24463
24	0 (30)	0 (20)	0 (5)	2 (323)	94.17	94.38279
25	0 (30)	0 (20)	0 (5)	0 (303)	96.45	96.48333
26	0 (30)	0 (20)	0 (5)	0 (303)	96.49	96.48333
27	0 (30)	0 (20)	0 (5)	0 (303)	96.50	96.48333
28	0 (30)	0 (20)	0 (5)	0 (303)	96.45	96.48333
29	0 (30)	0 (20)	0 (5)	0 (303)	96.50	96.48333
30	0 (30)	0 (20)	0 (5)	0 (303)	96.51	96.48333

Experimental conditions [Coded Values] and observed response values of central composite design with 2^4 factorial runs, 6- central points and 8- axial points. Agitation time fixed at 60 min and biosorbent size at 53 μm

Table–1.5

ANOVA of Amido Black biosorption for entire quadratic model

Source of variation	SS	Df	Mean square(MS)	F-value	P> F
Model	1046.875	14	74.7767	74776.7	0.00000
Error	0.015	15	0.001		

Total	1046.890

Df- degree of freedom; SS- sum of squares; F- factor F; P- probability.
R^2=0.98858; R^2 (adj):0.97792

A). 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. 1.7 exhibits normal probability plot for the present data. It is evident that the experimental data are reasonably aligned implying normal distribution.

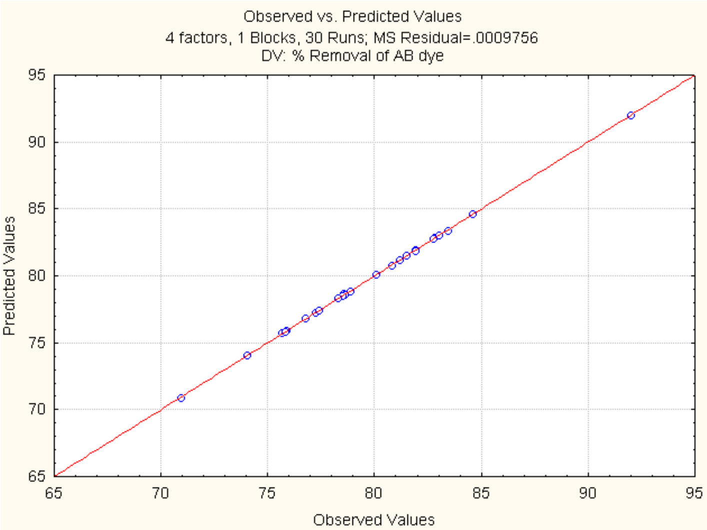

Fig. 1.7 Normal probability plot for % removal of Amido Black

5. Interaction effects of dye decolorization variables:

Three-dimensional view of response surface contour plots [Fig. 1.8 (a) to 1.8 (f)] exhibit % biosorption of the Amido Black using Chaetomorpha Antennina powder for different combinations of dependent variables. All the plots are delineated as a function of two factors at a time, imposing other factors fixed at zero level. It is evident from response surface contour plots that the % biosorption is minimal at low and high levels of the variables. This behavior conforms 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 Amido Black and seen clearly from the plots. The predicted optimal set of conditions for maximum % biosorption of Amido Black is:

Biosorbent dosage	=	35.7794 g/L
Initial Amido Black concentration	=	20.0617 mg/L
pH of aqueous solution	=	7.3595
Temperature	=	304.6548 K
% Biosorption of Amido Black	=	92.63301

The experimental optimum values are compared with those predicted by CCD in table-5.7 The experimental values are in close agreement with those from CCD.

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Table-1.7

Comparison between optimum values from CCD and experimentation

Variable	CCD	Experimental
pH of aqueous solution	7.3595	7
Biosorbent dosage, w, g/L	35.7794	35
Initial Amido Black concentration, mg/L	20.0617	20
Temperature, K	304.6548	303
% Biosorption	92.63301	91

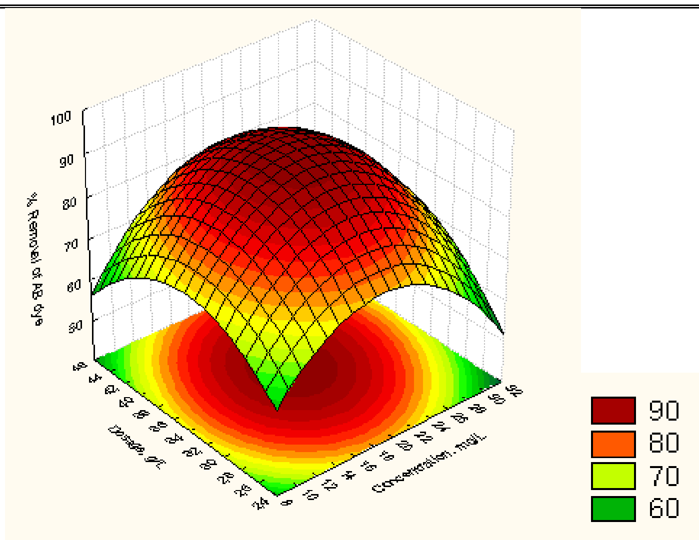

Fig. 1.8 (a) Surface contour plot for the effects of dosage and initial concentration of Amido Black on % removal

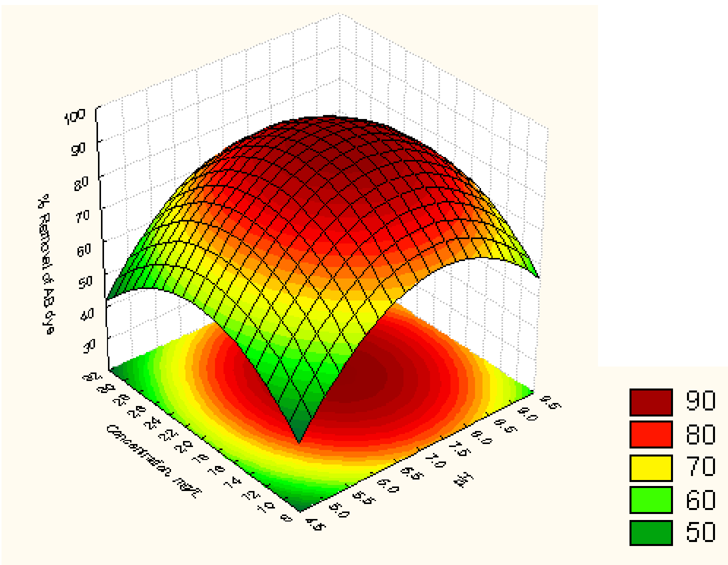

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Fig. 1.8 (b) Surface contour plot for the effects of pH and initial concentration of Amido Black in aqueous solution on % removal

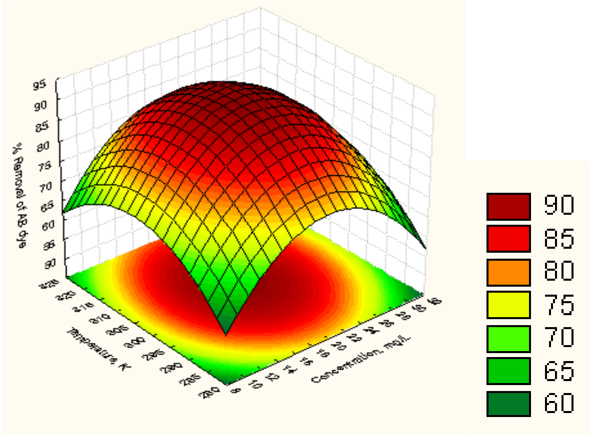

Fig. 1.8 (c) Surface contour plot for the effects of temperature and initialconcentration of Amido Black in aqueous solution on the % removal

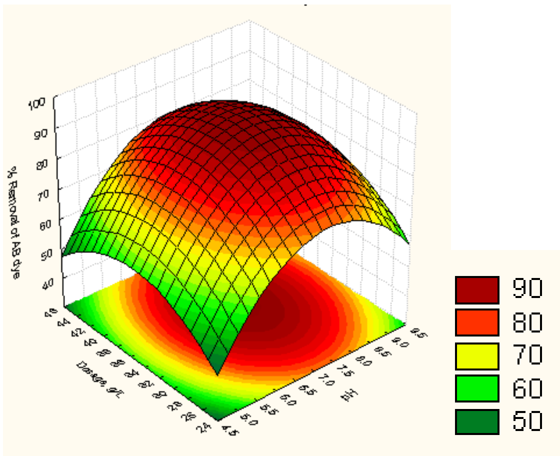

Fig. 1.8 (d) Surface contour plot for the effects of pH of aqueous solution and dosage on % removal of Amido Black

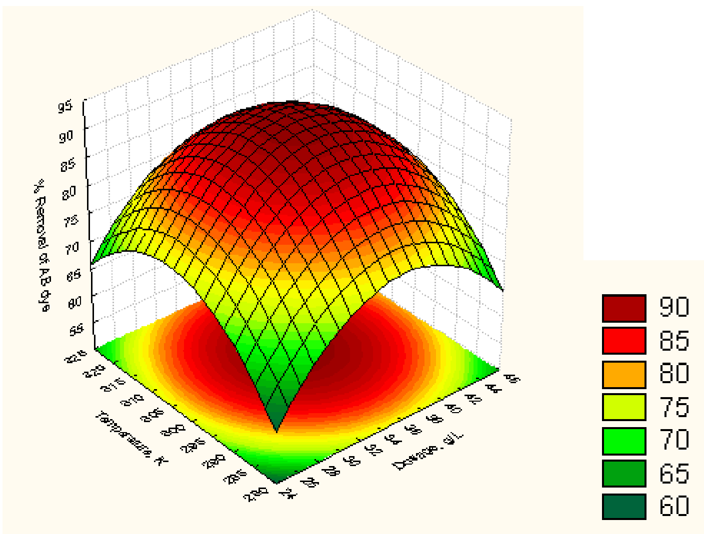

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Fig. 1.8 (e) Surface contour plot for the effects of temperature and dosage on % removal

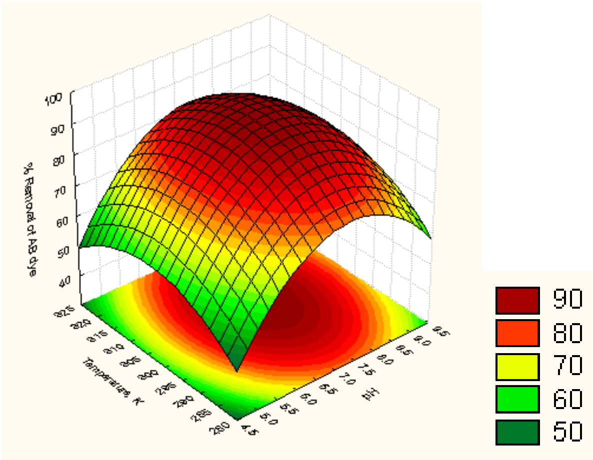

Fig. 1.8 (f) Surface contour plot for the effects of temperature and pH on % removal

6. Characterization of Chaetomorpha Antennina powder

A). FTIR spectrum of untreated Chaetomorpha Antennina powder

Infrared spectroscopy has a place with the gathering of atomic vibrational spectro scopies which are particle explicit and give direct data about the utilitarian gatherings, their sort, associations and directions. Its inspecting prerequisites permit the addition of data from fluids and gases and specifically from strong surfaces. Regardless of whether generally IR has been for the most part utilized for subjective investigation, to acquire underlying data, these days instrumental development makes non-dangerous and quantitative examination conceivable with critical exactness and accuracy. The shift of the groups and the progressions in signal force permit the recognizable proof of the utilitarian gatherings associated with metal biosorption

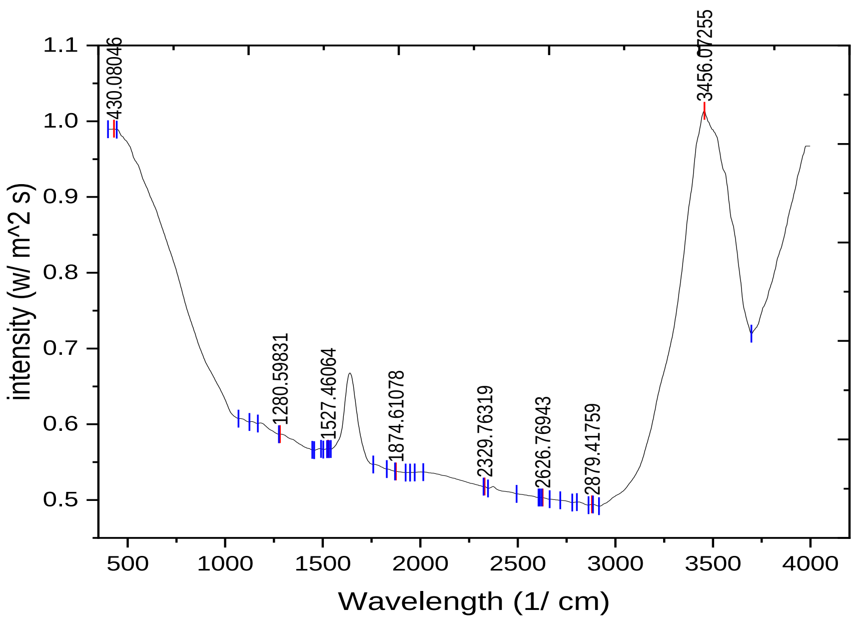

Fig. 1.9 (a) FTIR spectrum of untreated Chaetomorpha Antennina powder

b) FTIR spectrum of treated Chaetomorpha Antennina powder

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FTIR measurements presented in below figure 1.10 (b) shows the presence of C-I stretch at band of 430.08046 Cm ($^{-1}$). The band at 1766.60851 is due to C=O stretch and asymmetric stretch. The band at 1944.04081, 2065.54336, and 2377.97849 is due to Alkyl C-H stretch. The band at 2543.83912, 2653.77, 2813.84478, 346.57284, and 3479.2159 is due to O-H stretch.

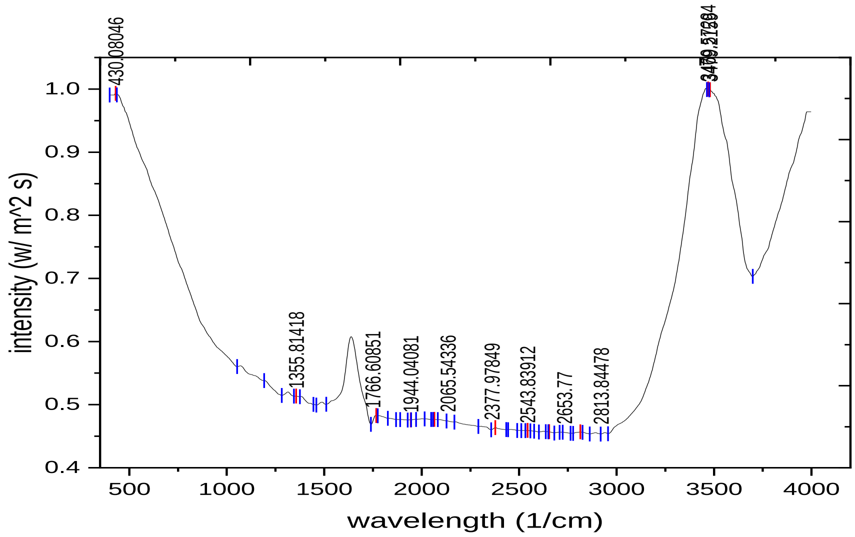

Fig. 1.10 (b) FTIR spectrum of treated Chaetomorpha Antennina powder

Table-1.8

Shift of FTIR peaks between untreated and Amido Black treated Chaetomorpha Antennina powder

S.No	Peaks in 5(a), cm($^{-1}$)	Peaks in 5(b),cm($^{-1}$)	Description
1	430.08046	430.08046	C-I stretch
2	1280.59831	---	C-F stretch
3	---	1355.81418	
4	1527.46064	---	Aromatic ring C=C stretch
5	---	1766.60851	C=O stretch & asymmetric stretch
6	1874.61078	---	Alkyl C-H stretch
7	---	1944.04081	Alkyl C-H stretch
8	---	2065.54336	Alkyl C-H stretch
9	2329.76319	---	Alkyl C-H stretch
10	---	2377.97849	Alkyl C-H stretch
11	2626.76943	---	O-H stretch
12	---	2543.83912	O-H stretch
13	2879.41759	---	Alkyl C-H stretch
14	---	2653.77	O-H stretch
15	3456.07255	---	O-H stretch
16	---	2813.84478	O-H stretch
17	---	3466.57284	O-H stretch
18	---	3479.2159	O-H stretch

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X–Ray Diffraction

A). Untreated Chaetomorpha Antennina powder

The X-Ray Diffractograms (XRD) of the powder tests are taken utilizing a Rigaku Ultima model IV. The diffracted X-beam forces are recorded as an element of 2q by utilizing copper target (Cu-Ka radiation with frequency, l = 1.5492 A^0) at an output speed of 20/min. XRD designs are recorded from 3 to 900. Various periods of the examples in figs.1.16 (a) and 1.16 (b) are recognized by contrasting a bunch of 'd' values and the comparing powers with the principles from the ICDD (International Center for Diffraction Data) records. XRD design doesn't show exceptionally sharp and unmistakable pinnacles and displays pretty much undefined nature. The peaks at 2theta values of 10.59, 10.79 and 88.29 corroborate the presence of Esperite, ((Fe (C $O)_3)_2$ (C O)) (Fe (C $O)_4)_4$ Sn2, with corresponding d values are 8.34708 (the miller indices (h k l) values are 1 0 0), 8.19280 and 1.10599 (the miller indices (h k l) values are 7 2 2 respectively

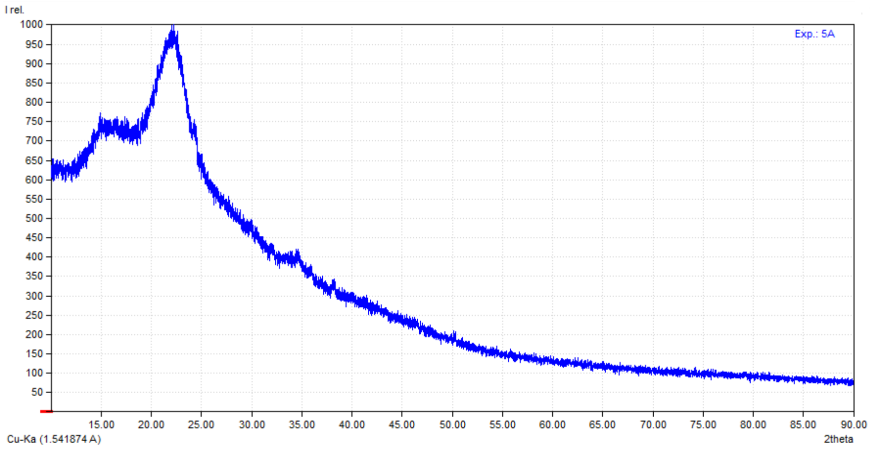

Fig.1.11 (a) XRD pattern of untreated Chaetomorpha Antennina powder

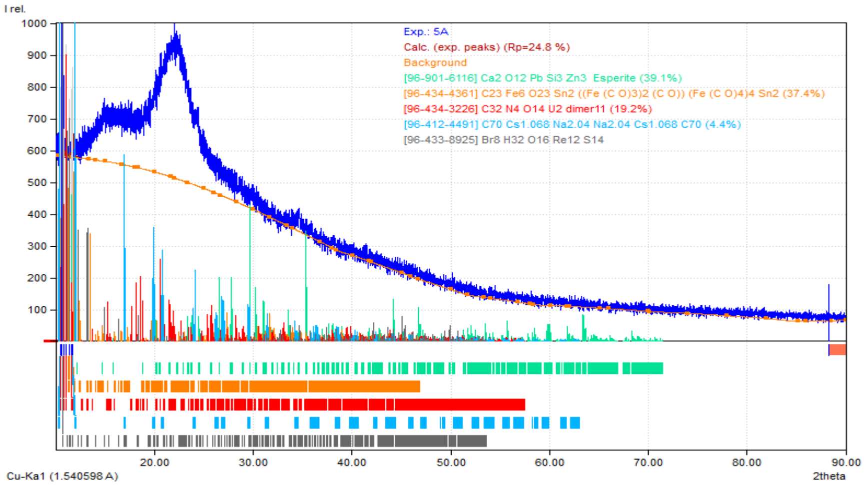

Fig.1.12 (b) XRD pattern of untreated Chaetomorpha Antennina powder –matching compounds

B). Chaetomorpha Antennina powder treated with Amido Black

XRD designs for treated powder [Figs.1.16(c) and 1.16(d)] display more formless nature and expansion in surface region and porosity. The peaks at 2theta values of 10.73, 18.76, 20.40, 20.81, 21.01, 22.07, 22.81 corroborate the presence of Tridymite, I2@SL-1, Acetone, Porphyrazinegalliumchloride chloride, Cannizzarite, Nb8 W9 O47, Hf (P_2 O_7) with corresponding d values are 8.2385, 4.7263, 4.3499, 4.2651, 4.2250, 4.0244 and 3.8955 respectively.

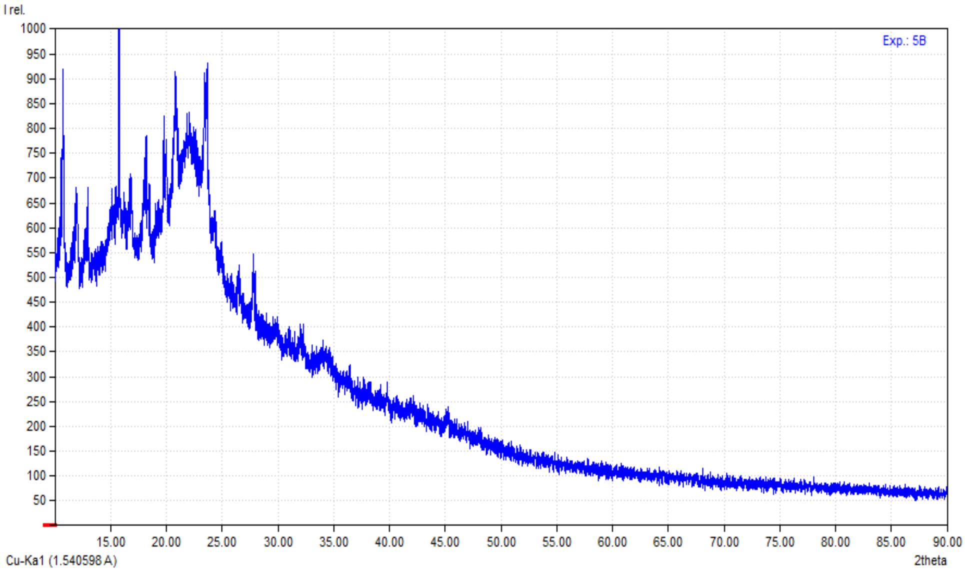

Fig.1.13 (c) XRD of Amido Black treated Chaetomorpha Antennina powder

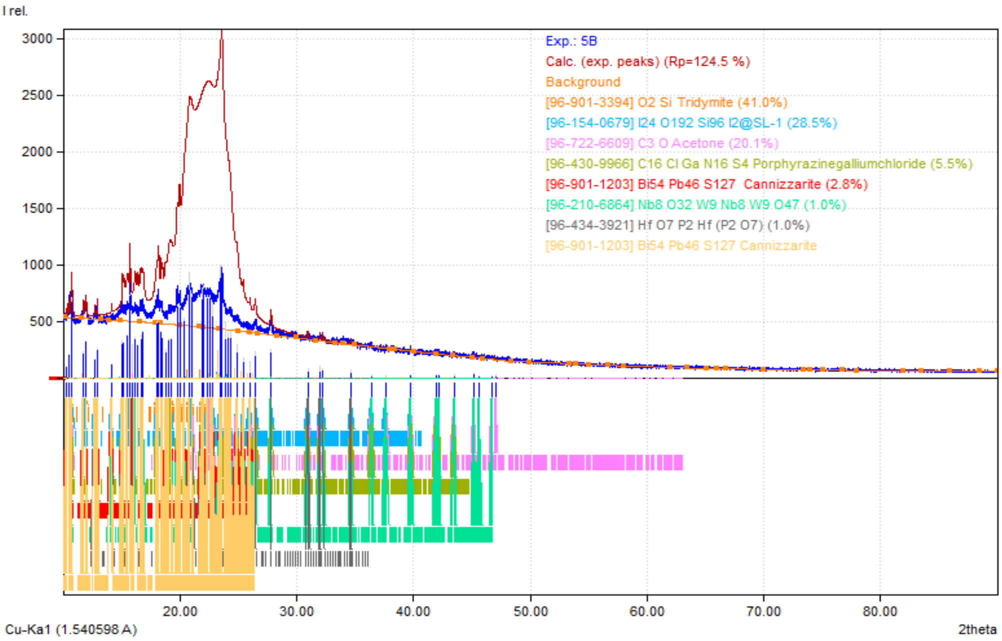

Fig.1.14 (d) XRD of Amido Black treated Chaetomorpha Antennina powder–matching compounds

IV. CONCLUSIONS

1. From the predicted values of RSM results, maximum sorption of Amido Black dye (92.63301 %) is observed when the processing parameters are set as pH = 7.3595, w = 35.7794 g/L, Co = 20.0617 mg/L and T = 304.6548 K.

2. The investigation also reveals the: endothermic nature of sorption as ΔH is positive (13.6845 KJ/mole K) spontaneity of the sorption as ΔG is negative (10.319.6 kJ/mole K) irreversible nature of sorption as ΔS is positive (34.0129 J/mol k)

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