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Removal of Coomassie brilliant blue dye from waste waters using active carbon derived from barks of *Ficus racemosa* plant

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ABSTRACT

An active carbon derived from the barks of *Ficus racemosa* plant has been developed as bio-adsorbent in the removal of Coomassie brilliant blue R-250 dye from the effluents of Industries based on the dyes. The sorption properties have been explored towards the said dye with respect to various physicochemical parameters such as pH, time of contact, adsorbent dosage, temperature and initial concentration of the dye and the conditions have been optimized for the maximum removal of the dye. The adsorption process is found to be maximum at low pH values: 2-4. The co-ions generally found in natural waters have not interfered. The adsorption process has been analyzed using Langmuir and the Freundlich isotherms and the latter model has been found to be better fit for the observed data indicating the heterogeneous and multilayer nature of the adsorption process. The Kinetics of adsorption is found to follow pseudo second-order model. The FTIR data before and after the adsorption of the dye indicate the binding of the dye to the surface of the adsorbent. The thermodynamic parameters indicate the spontaneous and endothermic nature of the adsorption. The maximum dye adsorbed on to ACFR is found to be 65.0 mg/g. The methodologies developed are successfully applied to real industrial water samples collected from the effluents of dyeing industries.

Key words: Coomassie brilliant blue, active carbon, *Ficus racemosa* plant, adsorption, applications.

INTRODUCTION

Most of the synthetic dyes are non-degradable and their presence in the natural bodies is posing grave problems to the aquatic life [1]. Some of these organic dye stuffs are carcinogenic in nature and are potential toxins. Further, the presence of color in water bodies curtails the passage of light to the aquatic organisms living in deep waters of the water bodies and thereby, disturbs the ecology of the water bodies [2-4]. In view of the potential environmental pollutive nature of the synthetic dyes, researchers are investigating simple and effective methods for extracting dyes from waste waters [5-39].

Of the various methods being probed to address the problems caused by the presence of dyes in natural water bodies, the methods based on using bio-wastes and agricultural waste materials either in their native form or activated form are proving to be effective, simple and economical especially for agricultural country like India. Our research group is probing these bio-methods of pollution control in controlling various potential pollutants and obtained some interesting results and they have been reported to the literature [40-54].

The literature survey indicates that investigations have been made for the removal of dyes using the bio-sorbents obtained from fruit peels [5,6], sugarcane bagasse [7,8], husk [9], sawdust [10], sludge [11], leaves and barks of some plants [12,13], palm shells [14, 15], straws [16], seeds [17], Peels of *Citrus documana*, *Citrus medica* and *Citrus aurantifolia* [18], tapioca [19], Jackfruit [20], orange [21] durian [22] and Grape juice waste [23] in the removal of dyes from waste waters. Dyes like methyl red [24], ethylene blue [25] some reactive dyes [26], n-ethylene blue [27], congo red [28] and malachite green [29, 30] have been extracted from polluted waters.

Basic violet 10, Basic violet 1 and Basic green 4 have been extracted using sorbents derived from Sugarcane Bagasse. [31]. D. S. Kharat [32] reviewed and amply discussed the removal of various dyes using adsorbents derived from agricultural residues.

Coomassie Brilliant Blue R-250 (Fig No. 1) is widely used as dye in textile and wool industry. The effluents from these industries possess residual amounts of this dye. The effluents are to be treated for removing this dye before disposing the effluents into the water bodies. Otherwise, as the dye is non-degradable in nature, it gets accumulated in the water bodies and turns to be a potential pollutant as it is toxic in nature. It is reported in literature that the consumption of waters containing this dye causes severe eye problems, toxic to aquatic life, and causes irritation to the mucous membranes and upper respiratory tract of the living organisms [35]. Further, it is likely to cause problems with regard to inhalation, skin and ingestion.

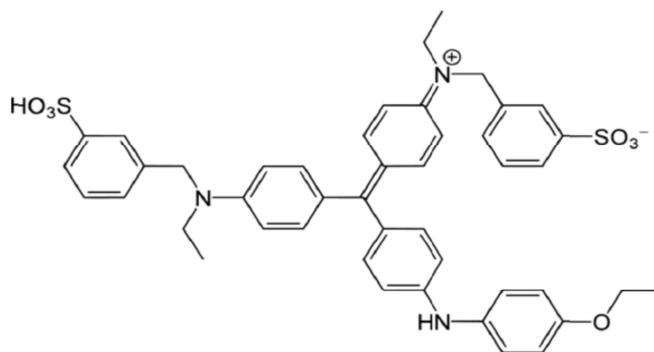


Figure 1. Chemical structure of Coomassie Brilliant Blue (R-250)

The reports available in removing this potential toxic dye from waste waters are few and far between. Ganga Ram Chaudhary *et al* [33] studied the removal of Coomassie Brilliant Blue R-250 dye from water using γ -Fe₂O₃ nanoparticles. Sadia Ata *et al* [34] studied the equilibrium, thermodynamics, and kinetic sorption studies for the removal of Coomassie Brilliant blue on wheat bran as a low-cost adsorbent. Abbas Moussa *et al* [35, 36] studied the kinetic and equilibrium of Coomassie Blue G-250 adsorption on apricot stone activated carbon. R. Naveen Prasad *et al* [37] studied the biosorption of Coomassie Brilliant Blue using acid treated coir-pith using batch modes of extraction.

While we are probing the plant materials for their sorption nature, we noticed that the active carbons derived from barks of *Ficus racemosa* have affinity towards the Coomassie Brilliant Blue R-250 Dye. In this work, the sorption nature of the active carbons derived from the said plant has been thoroughly investigated in removing the Coomassie Brilliant Blue R-250 Dye from polluted waters.

MATERIALS AND METHODS

Plant description:

Ficus racemosa (Fig No. 2) belongs to the family of Moraceae and is grown along rivers and streams to a height of nearly 16 m. It is reported that this plant materials have medicinal values; leaves are used in curing bronchitis; barks in asthma curing; fruits in curing diseases of blood.

Preparation of active carbon:

Bark of *F. racemosa* plant were scrapped, cut into pieces, washed with distilled water and dried under sunlight for 3 days. The dried bark pieces were carbonized in muffle furnace at 500⁰C for 4 h. After carbonization, the carbon was washed with distilled water several times and then filtered. The filtered carbon was dried in an oven at 110⁰C and was sieved by 75 μ m ASTM mesh. The screened carbon was activated by boiling the carbon powder in 1N HNO₃ for 2-3 hours on Bunsen burner flame. After treatment with acid, the treated material was washed with distilled water repeatedly and brought the material to neutral pH and was dried in oven at 110⁰C for 5h. This activated carbon was named as ACFR (Active carbon of *F. racemosa*). The material was stored in an airtight container for further use.



Figure 2: *Ficus racemosa* plant identified to have affinity towards Coomassie brilliant blue dye R-250

Reagents and Chemicals:

All the chemicals used were of analytical reagent grade purchased from Merck. India Pvt. Ltd and Sd. Fine Chemicals and all the solutions were prepared using double distilled water throughout this study.

Preparation of dye solution: Stock solution of 100 ppm of Coomassie brilliant blue R-250 Dye was prepared and stored in brown reagent bottles to prevent any oxidation of the dye solution. The solution was suitably diluted as per need.

Adsorption Experiments: Batch adsorption experiments [55-57] were used to determine the adsorption capacity of the adsorbent, active carbon, ACFR. Simulated dye solution of 20 ppm of Coomassie brilliant blue dye R-250 Dye was prepared from the stock solution (100 ppm) using distilled water. 100 ml of 20 ppm dye solution was taken into 250 ml conical flask and to which weighed quantities of the adsorbent was added and pH was maintained. Then the conical flask with test solution and adsorbent was shaken in horizontal shaker at 120rpm. After the desired contact time, the conical flask was removed from shaker and the solution was filtered using Whatman No.1 filter paper and the filtrate was analyzed for the residual dye concentration using Spectrophotometric method.

Method: The dye has λ_{\max} at 547 nm and obeys Beers-Lamberts law at low concentrations. The O.D. measurements were made at the said λ_{\max} using UV-Visible Spectrophotometer against blank. The obtained O.D value for un-known solution was referred to standard graphs (drawn between O.D and concentration) prepared with known concentrations of the dye by adopting method of Least Squares to know the concentration of the dye.

By this way the effect of parameters like pH, adsorbent dosage, agitation time, initial concentration of adsorbate (dye) and temperature on the sorption nature of the active carbon towards the dye were studied and conditions were optimized.

The dye uptake capacity for each sample was calculated according to a mass balance using equation (1).

$$\text{Amount adsorbed } (q_e) = \frac{(C_0 - C_e)}{m} \times V \quad (1)$$

where m is the mass of adsorbent (g), V is the volume of the solution (L), C_0 is the initial Concentration of dye (mg L^{-1}), C_e is the equilibrium dye concentration (mg L^{-1}) and q_e is the amount of dye quantity adsorbed at equilibrium (mg/g). The percent removal of dye from the solution was calculated by the following equation (2).

$$\% \text{ removal} = \frac{(C_0 - C_i)}{C_0} \times 100 \quad (2)$$

where C_0 (mg/L) is the initial dye concentration and C_i (mg/L) is the final concentration of dye in the solution.

The sorption characteristics of the active were studied with respect to the time of equilibration, pH, sorbent dosage, temperature and initial concentration of adsorbate. The obtained results were presented in the Graph No. 1-6; Table No.1-3.

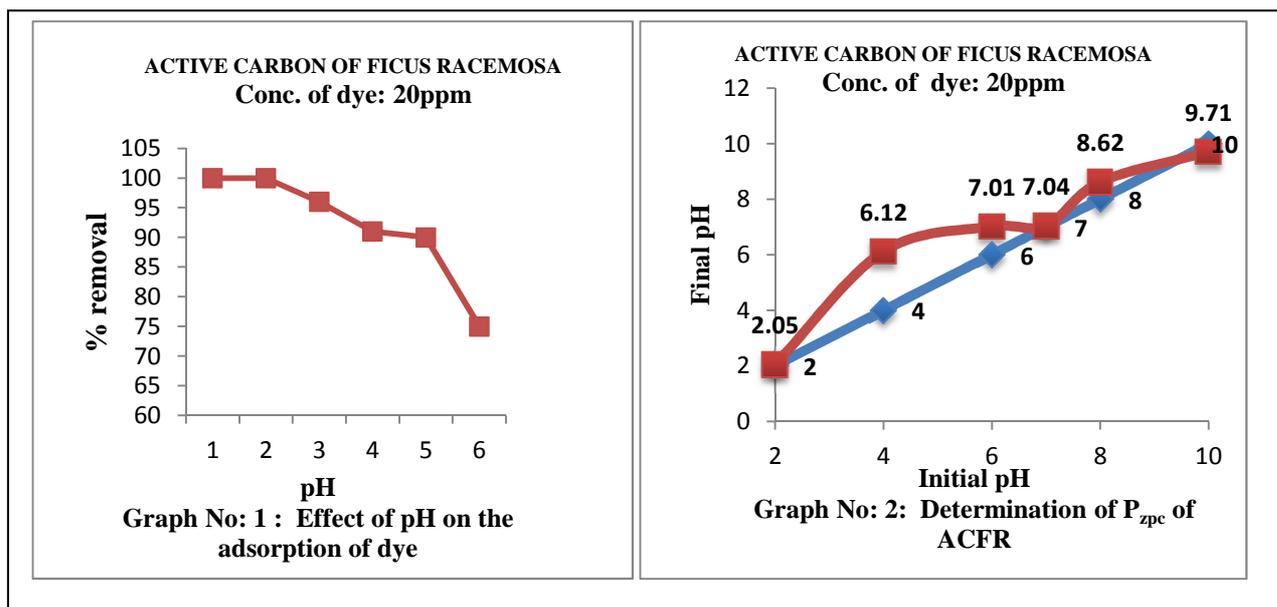
The interference of common co-ions present in natural waters on the % of extraction of the dye was studied and was presented in the Table No.3. The observed nature of sorption was analyzed using adsorption isotherms and kinetic studies and the obtained data was presented in the Graph No: 1 to 8.

RESULTS AND DISCUSSION

The effect of various parameters such as pH, adsorbent dosage, agitation time, temperature, initial concentration of dye and interfering ions on the extraction of Coomassie brilliant blue R-250 dye from polluted waters was studied. The following observations are significant.

1: Effect of pH:

The pH of the dye solution plays an important role in the whole adsorption process. The effect of pH was studied in a range 2–10 at a temperature of 30⁰ C and the results were presented in Graph No.1. It was observed from the Graph that 100% removal is possible in the pH range: 2 to 4 at other optimum conditions of extraction and % of extraction was decreased as the pH increased further from 4 to 10.

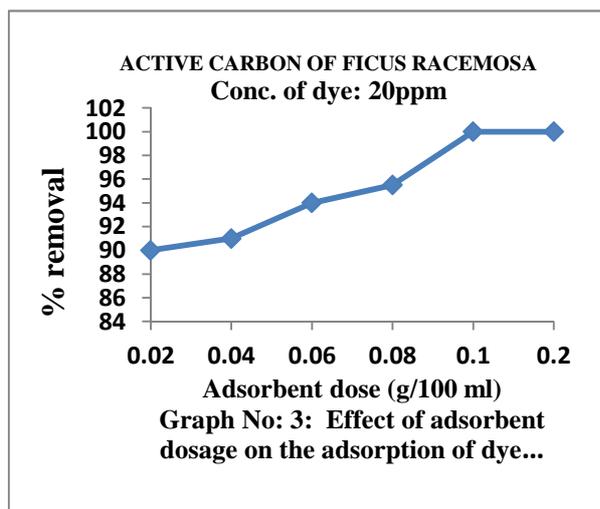


The observations may be accounted from the zero point surface charge (pH_{zpc}) of adsorbent (pH_{zpc}) which plays a significant role in mode of adsorption at different pHs. As from the Graph No. 2, the pH_{zpc} for the active carbon is 7.04; hence, the surface charge is positive at pH values lower than pH_{zpc} i.e. 7.04, neutral at $pH_{zpc} = 7.04$, and negative at pH values higher than it.

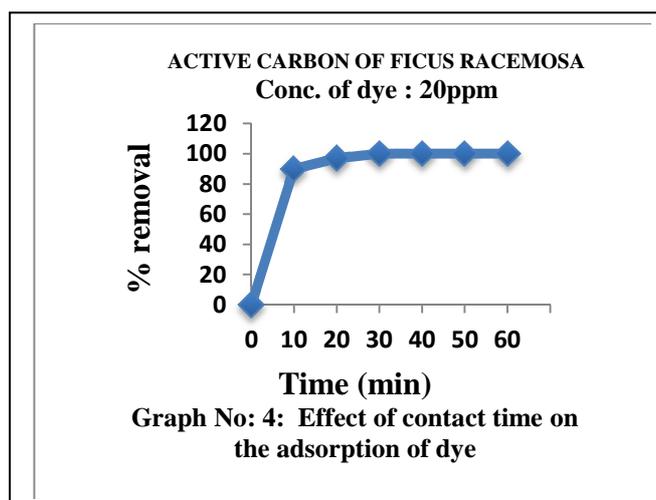
At low pH values i.e. 2-4, the surface of the adsorbents is endowed with positive charge and so, it has affinity towards the negatively charged dye species and hence the removal of the dye is 100%. But as the pH is progressively increased beyond 7.04, the surface of the adsorbent acquires negative charge and this negatively charges less favours the binding of dye to the surface of the adsorbent and hence, the % extraction is low. However, even at higher pH values, substantial removal of the dye was observed. This indicates that the binding forces are not simple electrostatic interaction but also through other mechanism especially via hydrogen bond formation.

2: Effect of adsorbent dosage:

The extraction studies were made by varying the sorbent concentrations from 0.02 to 0.2 g/100 ml at other optimum extraction conditions: pH: 4, time of equilibration: 30 min, rpm: 120; initial conc. of dye: 20 ppm; temp. : 30⁰ C. The results are presented in the Graph No. 3. As the sorbents concentration is increased, the % removal of the dye also increased but after certain dose, it remains constant. It is observed from the Graph that the % removal of the dye is increased from 90% to 100% removal as the sorbent concentration is increased from 0.02 to 0.1 g/100 ml. The increase of % of extraction with the increase in the sorbent concentration may be attributed to the increase in the availability of active sites for binding the dye on the surface of the adsorbent with the increase in the concentration of the adsorbent.

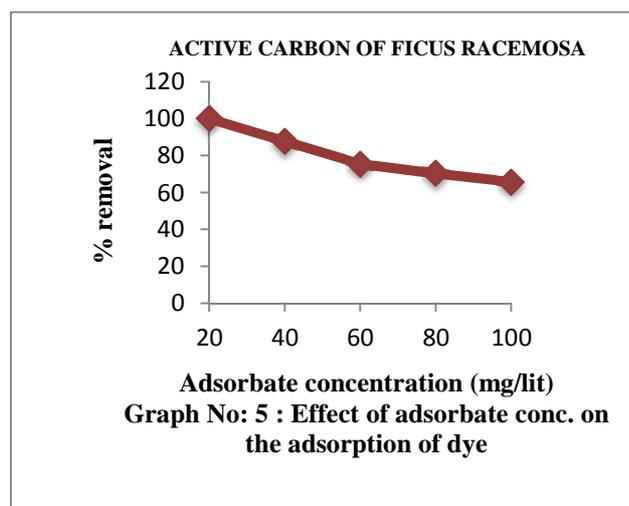


3: Effect of Contact time: To determine the optimum equilibrium time, % removal is studied as the time is varied from 10 to 60 minutes at pH: 4, dosage: 0.1 g/100ml, rpm: 120 and temp. 30°C. It is evident from the Graph No: 4, that the removal of the dyes is increased with increase in the agitation time. At 10 minutes itself 90.0% dye is removed when the concentration of the dye is 20ppm and then onwards, the removal is slow down and the 100% removal is attained only after 30 minutes of agitation with 20ppm dye solution. The substantial removal of the dye within 10 min is due to the large availability of active site on the adsorbent initially and as the active sites are used up with time, the adsorption process becomes slow and it has taken 30 minute to remove the dye completely.



4: Effect of initial concentration:

The effect of initial concentration of the dye on the % of extraction when all the other conditions are optimized has been studied and the results have been depicted in the Graph No. 5. It is observed from the graph that the % removal decreases with the increase in the initial concentration. Changing of the initial concentration of acid dye from 20 to 100 mg/L, % removal of the dye is decreased from 100 to 65.8%. This may be attributed to the fact that at higher concentration of dye, the active sites of fixed adsorbent are less available and are not sufficient for the complete removal of dye.



5: Effect of temperature:

The effect of temperature on the % removal of dye was studied using synthetic solutions of the dye of concentration: 50 ppm and varying the temperatures from 303 to 333 k at optimum conditions of extraction i.e., pH: 4, contact time: 30 mints, rpm: 120 and dosage: 0.1gm/100ml. The results obtained were plotted in the Graph No: 6(a); 6(b) as temperature vs. % removal: (1/T) vs. $\ln(K_d)$.

The thermodynamic parameters such as change in free energy (ΔG) (KJ/mole), change in enthalpy (ΔH) (KJ/mole) and change in entropy (ΔS) (KJ/mole) were determined using the equations; $\Delta G = -RT \ln K_d$; $\ln K_d = \Delta S / R - \Delta H / RT$; $K_d = q_e / C_e$; $\Delta G = \Delta H - T\Delta S$,

where K_d is the distribution co-efficient for the adsorption, q_e is the amount of dye adsorbed on to the ACFR at equilibrium, C_e is the equilibrium conc. of dye, T is the absolute temperature in Kelvin, R is the gas constant. ΔG is the change in free energy, ΔH is the change in enthalpy and ΔS is the change in entropy.

The values of ΔH and ΔS were obtained from the slope and intercept of the plot between $\ln K_d$ and $1/T$ and ΔG values were obtained from the equation $\Delta G = \Delta H - T\Delta S$ [58-62] and were presented in the Table No: 1. It is inferred from the graph that with increase in the temperature, the % removal of dye is also increased from 36% to 80% (initial conc. of the dye: 50 ppm). This is due to the fact that with increasing the temperature, the kinetic energy of the dye molecules is increased across the internal pores of the adsorbent and hence, more removal.

It is inferred from the Table No: 1 that ΔH value is positive indicating that the adsorption process is endothermic in nature and ΔG value is negative indicating that the adsorption process is spontaneous.

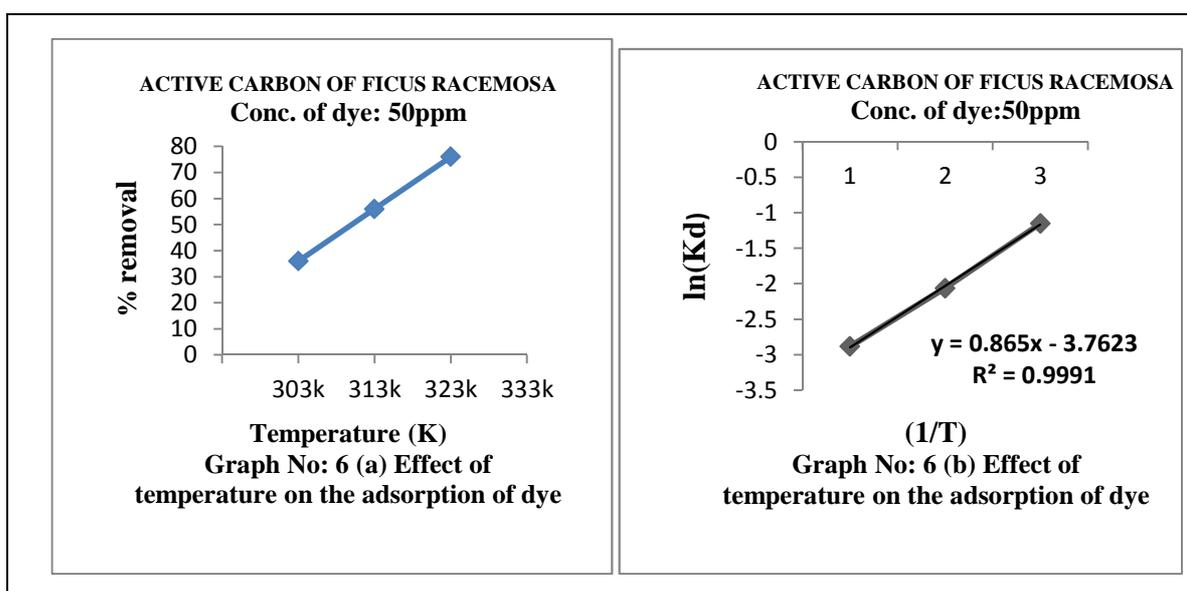


Table No: 1 Thermodynamic Parameters of Adsorption of dye on ACFR

ΔH (KJ/mole)	ΔS (KJ/mole)	ΔG (KJ/mole)				R^2
		-9.167 (303K)	-10.146 (313K)	-11.138 (323k)	-12.124 (333k)	
7.19	31.27					0.9991

6: Adsorption Isotherms:

The nature of interaction between the adsorbate and adsorbent is described by the adsorption isotherm. The Freundlich [63] and Langmuir [64] isotherms are often used models. Linear form of Freundlich isotherm equation is: $\log(q_e) = \log k_F + (\frac{1}{n}) \log C_e$ and Langmuir isotherm equation is: $(C_e/q_e) = (a_L/k_L) C_e + 1/k_L$

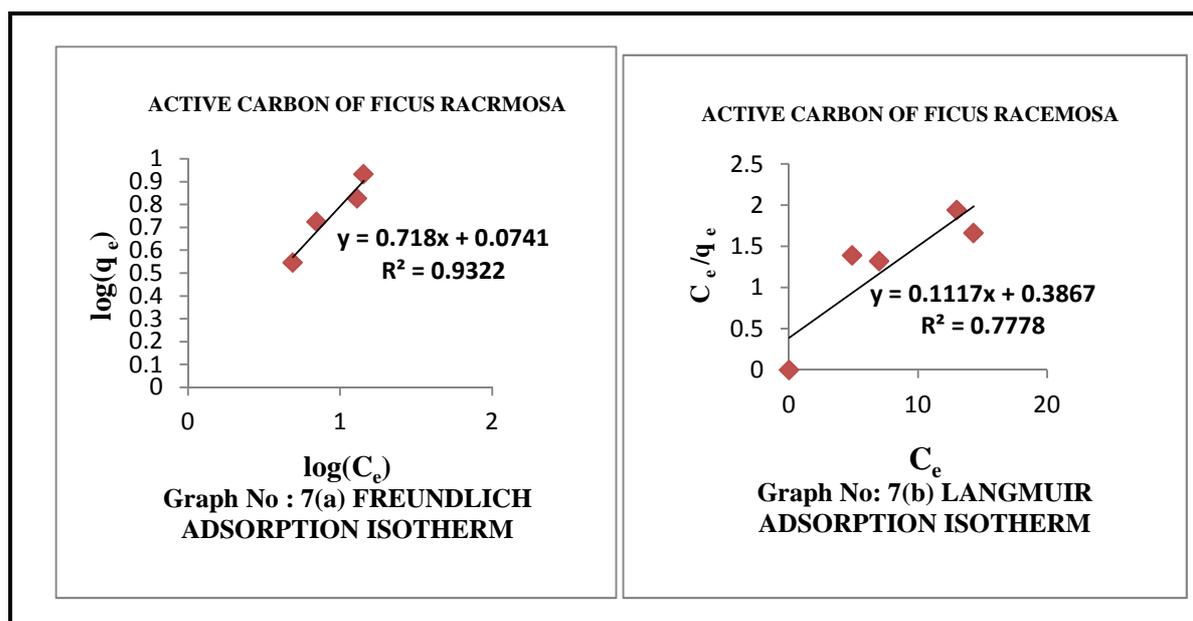
where k_F and $1/n$ are the Freundlich constants. k_F is an approximate indicator of adsorption capacity, while $1/n$ is a function of the strength of adsorption in the adsorption process, C_i and C_e are the initial and final concentration of the dye solution (mg L^{-1}) and q_e is the amount of dye quantity adsorbed at equilibrium (mg/g) and K_L and a_L are the Langmuir constants related to capacity and energy of adsorption respectively.

The significant features of Langmuir isotherm model can be defined by dimensionless constant separation factor R_L which can be expressed as:

$$R_L = \frac{1}{(1 + a_L C_i)}$$

R_L value indicates the adsorption nature to be either unfavourable if $R_L > 1$, linear if $R_L = 1$, favourable if $0 < R_L < 1$. Freundlich adsorption isotherm of $\log(q_e)$ vs $\log(C_e)$ at different dye concentrations were plotted and depicted in Graph 7(a). Freundlich constants k_F and $1/n$ were calculated from the intercept and slope of the straight line. $1/n$ is a heterogeneity parameter and if its value is smaller, greater is the heterogeneity of the surface of the adsorbent. If n lies between one and ten, this indicates a favorable sorption process. From the data in Table 2, the value of $1/n$ is 0.7183 while n is 1.392. This indicates that adsorption on the heterogeneous surface is favourable and moreover R^2 value is 0.9322. This confirms the applicability of Freundlich adsorption isotherm indicating the multilayer adsorption and heterogeneous surface of the adsorbent.

Further, Langmuir isotherms of C_e/q_e Vs C_e were plotted for the observed data and were presented in Graph No. 7(b). The slope (a_L/k_L), intercept ($1/k_L$), R_L and R^2 values were calculated and presented in the Table 2. It can be inferred from the Table that these two models adequately explain the sorption nature but however, as the R^2 value of Freundlich model is greater than the R^2 value of Langmuir model; the former model is more suited. In summary, the surface of the adsorbent is heterogeneous in nature and adsorption is multilayered.



7: Adsorption kinetics:

The kinetic study is important for the adsorption process, it describes the uptake rate of adsorbate and controls the residual time of the whole adsorption process. Pseudo first order and pseudo second-order equations are more frequently used to describe the adsorption process. The pseudo first order equation is: $\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} \cdot t$ while the pseudo second order equation is: $\frac{t}{q_t} = \frac{1}{K_2 \cdot q_e^2} + \frac{1}{q_e} \cdot t$

Where q_t (mg/g) is the amount of metal adsorbed on the adsorbent at various times (min), K_1 the rate constant of the pseudo first order kinetic (min^{-1}), K_2 the rate constant of the pseudo-second order kinetic. (min^{-1}). [65-67].

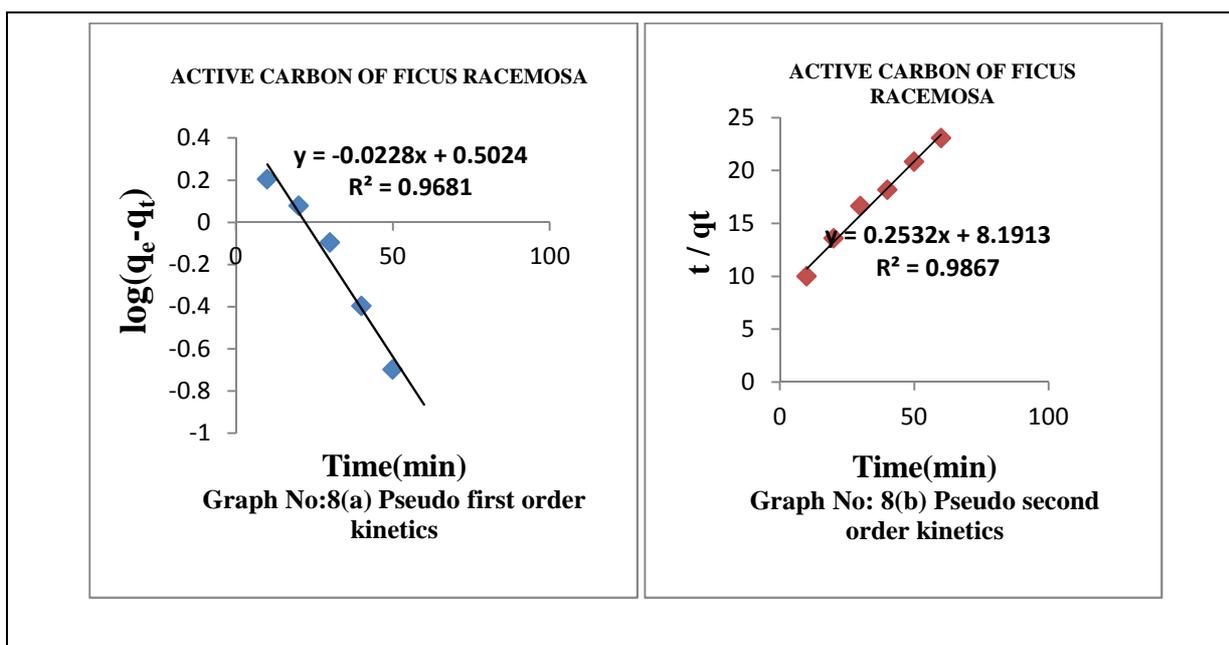


Table 2: Adsorption and Kinetic parameters

S.No.	Adsorption isotherms and Kinetic models		Slope	Intercept	R ²
1.	Freundlich isotherm	n= 1.392	0.718	0.0741	0.9322
2.	Langmuir isotherm	R _L = 0.047	0.1117	0.3867	0.7778
3.	Pseudo first order model		-0.022	0.502	0.9681
4.	Pseudo second order model		0.253	8.191	0.9867

The plots and values of pseudo first-order and pseudo second-order model constants together with the correlation coefficient values were presented in Graph No. 8(a) & 8(b) and Table 2 respectively.

The correlation coefficient (R^2) values are used to judge the best model. In this work, the R^2 value is 0.9681 for pseudo first order and 0.9867 for pseudo second order. As the R^2 values are near to unity, they indicate their applicability and further it indicates that the pseudo second order model is more probable than the pseudo first order model.

8: Effect of Co-ions:

% removal of the dye in presence of fivefold excess of the co-ions generally present in natural waters was studied at optimum conditions of extraction keeping the initial concentration of the at 20 ppm. The results are presented in the Table No.3. It is inferred that the effect is marginal.

Adsorbent	Maximum Extractability (%)
Active carbon of <i>Ficus racemosa</i>	100.0 without Co-ions (Dye conc: 20 ppm, pH: 4, 30 mints, 1.0 g/lit)
Interfering ions	Extractability of dye in presence of fivefold excess of (100 ppm) interfering ions at optimum conditions (Dye conc: 20 ppm, pH: 4, 30mints, 1.0 g/lit).
SO ₄ ²⁻	97.4
PO ₄ ³⁻	96.2
Cl ⁻	98.8
CO ₃ ²⁻	99.5
NO ₃ ⁻	96.7
Ca ²⁺	97.7
Cu ²⁺	96.0
Zn ²⁺	98.5

9: I R Spectral Data:

Surface morphological nature of the adsorbent with respect to functional groups present on the sorbent and their interaction with the functional groups of the dye can be understood by FTIR data of the adsorbent before and after adsorption of the dye. The FTIR data of the adsorbent before and after adsorption of the dye and also the FTIR spectrum of the individual dye are present in the Figure No. 3 a to c. The adsorption spectra showed a number of absorption peaks, indicating the complex nature of adsorption phenomenon. On analyzing the spectra before and after adsorption of the dye, it may be inferred that there are many peaks either shifted or broadened and further, some peaks characteristics of the dye have appeared in the FTIR spectrum of the adsorbent after adsorption.

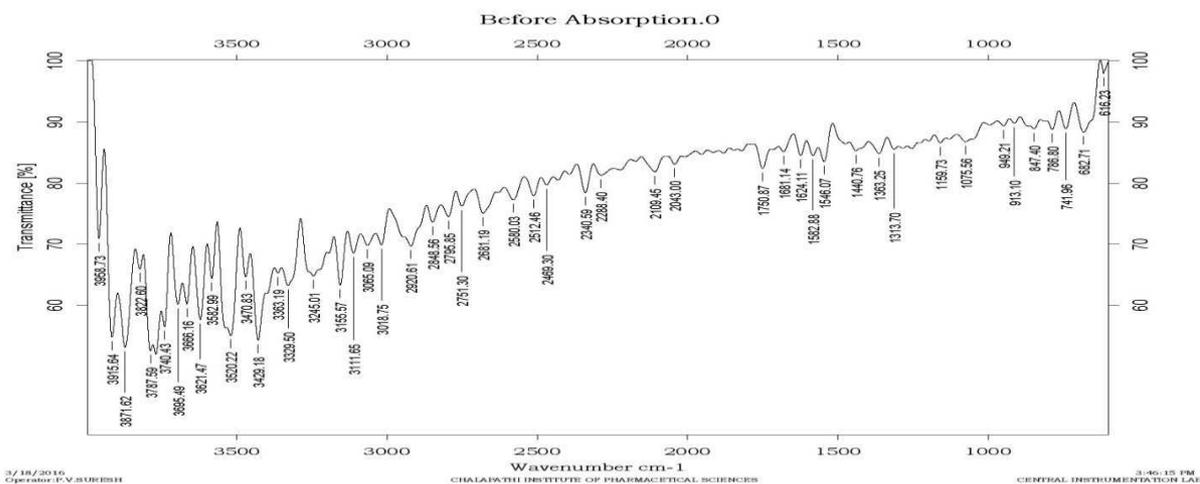


Figure No. 3a : IR Spectrum of the Adsorbent : Before adsorption of the Dye

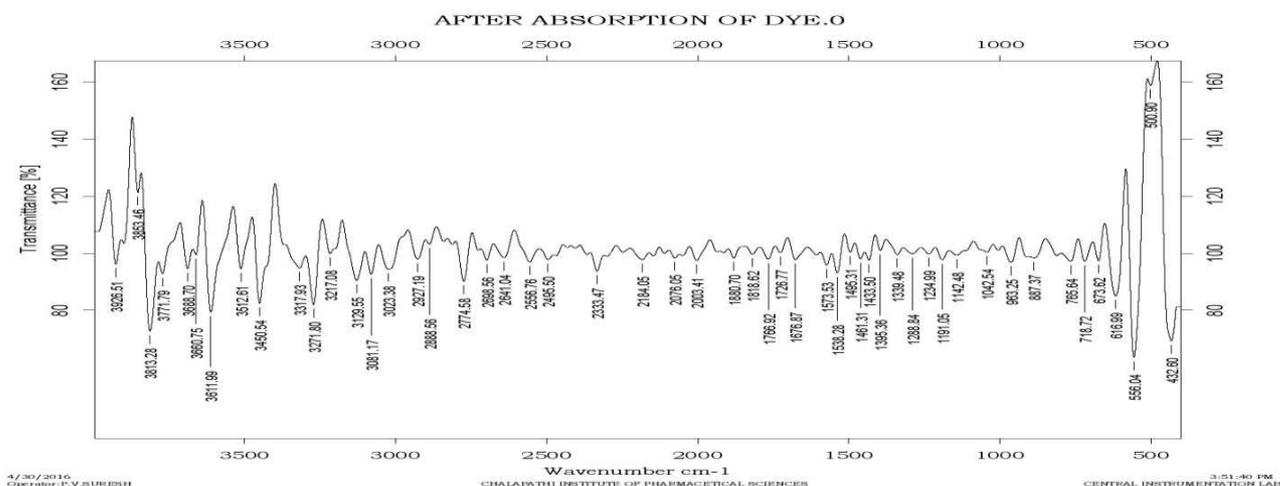


Figure No. 3: b: IR Spectrum of the Adsorbent after the adsorption of the Dye

Even fivefold excess of co-ions such as Sulphate, Phosphate, Nitrate, Chloride, Carbonate, Cu^{2+} , Ca^{2+} , Zn^{2+} , Mg^{2+} , Fe^{2+} that are generally present in natural waters have not effected the % removal of the dye.

Adsorption process has been analyzed using Langmuir and the Freundlich isotherms and it is found that the process can well be defined by Freundlich isotherms indicating the heterogeneity of the surface of the adsorbent and also the multilayer nature of the adsorption process. The kinetics of adsorption has been analyzed using pseudo first-order and pseudo second-order models and it is found the process follows the pseudo second-order.

The thermodynamics parameters namely, ΔH , ΔG and ΔS are calculated and found that the adsorption process is spontaneous and endothermic in nature. The observed increase in the % removal with the increase in temperature may be attributed to the enhancement in the kinetic energy of the dye molecules resulting the deep penetration of the dye across the internal pores of the adsorbent and hence, more removal.

The developed methodologies have been applied to the samples collected from the effluents from dyeing industries and found that they are remarkably successful.

The maximum dye adsorbed on to ACFR is found to be 65.0 mg/g and this active carbon derived from a common and widely grown plant, may turn to be an effective adsorbent in the removal of dyes from the effluents of textile industries especially an agricultural country like India.

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