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Removal of Red RB Dye from Aqueous Solution by Belpatra Bark Charcoal (BBC) Adsorbent

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- ✓ Adsorption;
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Abstract

Activated carbon prepared from Belpatra bark has been used as an adsorbent for the removal of Red RB dye from water solution. In order to study the process of adsorption, number of parameters such as contact time, initial adsorbate concentration, temperature, pH, adsorbent dosage and size were varied. Langmuir, Freundlich and Temkin adsorption isotherm models were tested for adsorption process. Adsorption kinetics fitted well with the pseudo second order kinetic model. FTIR and SEM studies were carried out to characterize BBC before and after adsorption of the dye. Results have been discussed.

1 Introduction

Intensive technological and industrial development, along with the growth of other human activity, has led to excessive use of water resources and increasing water pollution. Textile industry is the key user of dyes and hence the prime source of water pollution, which risks aquatic as well as human life. Removing synthetic dyes from waste effluents has become a challenge preventing their environmental hazard in particular for aquatic bio-systems [1]. Synthetic dyes are biologically non-degradable due to their aromatic structure and their synthetic origin. Searching for ecofriendly, sustainable and cost effective technologies for dye removal have gained considerable interest. In the last few years, various physical, chemical and biological methods were used for the degradation of different textile reactive dyes in order to control water pollution. The crucial goal of these processes was to change the toxic dyes into harmless end products, but many of the methods were unsuccessful as the end products were persistent and more toxic than original dyes [2]. Among these technologies, adsorption has emerged as an effective and economical technique for decontamination of organic pollutants from wastewater [1]. Recently, there has been a significant interest in converting agricultural and forest biomass into value-added products, especially adsorbents for mitigation of environmental pollutants. As a result, several researches proposed various agricultural wastes as precursors for the synthesis of activated carbons capable of removal of ammonium from water [3].

Recently, it is reported that degradation of reactive dyes, especially RB19 is possible by the combination of advanced oxidation processes in the presence of S-TiO_2, visible light and sonolysis [3]. However, the method was not much effective. In this paper, activated carbon was synthesized from Belpatra bark (Aegel marmelos) and used as an adsorbent for the removal of Red RB dye from aqueous solution varying different parameters.

2. Materials and Methods

2.1 Materials

The raw material used for the preparation of activated charcoal is Belpatra bark, collected from local garden of Ghaziabad. It was used as adsorbent (Figure 1).

Figure 1: Belpatra Bark Charcoal (BBC)

Reactive Red RB dye was supplied by Rituraj Textile Industry, Ghaziabad. It is anionic in nature. Red RB is a vinyl sulphone based reactive dye containing azo bond (Figure 2).

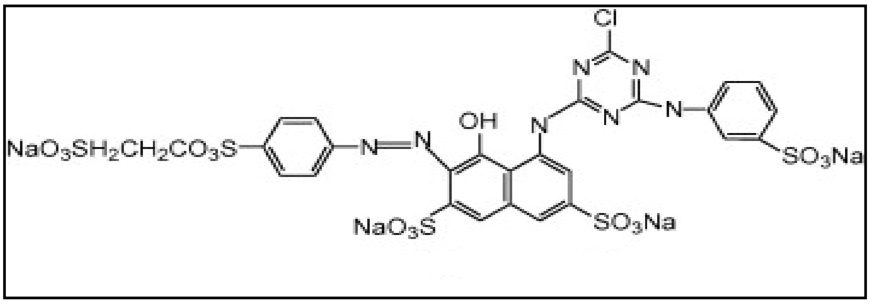

Figure 2: Structure of Red RB dye

2.2 Methods

2.2.1 Preparation of Activated Carbon (Adsorbent)

Belpatra bark was washed several times with distilled water to remove surface adhered as well as foreign particle impurities. After that it was soaked for two days in distilled water for removing the colored material present in Belpatra bark and dried firstly in sunlight for 5-6 days, secondly in the oven for 15 hours at the temperature of 120°C. This bark was crushed manually and then subjected to carbonization in a muffle furnace. In carbonization process, the sample was taken in a container and kept at the 400°C in a muffle furnace for 30 minutes. The activated charcoal obtained from a muffle furnace was washed, dried and passed through different sieve plates for getting particle sizes in the range of 75-149 µm, 150-299 µm, 300-599µm and 600-899 µm. The activated carbon then stored in airtight closed containers for further use as an adsorbent.

2.2.2 Preparation of Dye Solution (Adsorbate)

A stock solution of Red RB dye was prepared by dissolving 50 mg of dye in 100 ml of double distilled water. By diluting the stock solution in definite proportions, experimental solutions of desired concentration were obtained.

2.2.3 Adsorption Experiment

Adsorption experiments were performed by taking 100ml dye solutions of known concentration (10-30 mg/L) and known quantity of adsorbent (0.3-1.0gm) having particle sizes in the range of 75-899 µm, by adjusting the pH of the solution in the range of 1.5-7.5 at different operating temperatures (300-330K). The pH of sample solution was adjusted by adding appropriate amount of 0.1 N HCl or NaOH. After a predetermined time interval of 15 minutes, samples were taken out of the solution and filtered. The filtrates were stored in sample bottles and analyzed spectrophotometrically for the residual concentration by measuring absorbance through Shimadzu UV-1800 spectrophotometer. All experiments were done at a wavelength corresponding to the maximum absorbance (λ_{max}= 527 nm).

3. Characterization of adsorbent

3.1 SEM / External Chemistry of surface:

The surface morphology was analyzed using Scanning Electronic Microscopy, which was associated with energy dispersive X- ray spectrometer (SEM-EDX), model no. Zeiss 18 EVO.

3.2 FTIR / Internal Chemistry of Surface:

The presence of functional groups on the surface of adsorbent before and after adsorption was determined by Fourier Transform Infrared Spectroscopy. It was taken in KBr phase between 4000 to 650 cm^{-1} frequency.

4. Result and Discussion

4.1 SEM Analysis

Scanning electron micrographs of Belpatra bark before and after carbonization are shown in figure 3a and 3b respectively. From figure 3a, it was seen that Belpatra bark has a rough, porous and uneven surface. It was also reported that it is an amorphous substance containing many cavities. After carbonization, the surface characteristics of Belpatra bark get enhanced (figure 3b) which provided more surface area so that the process of adsorption of dye molecules is accelerated.

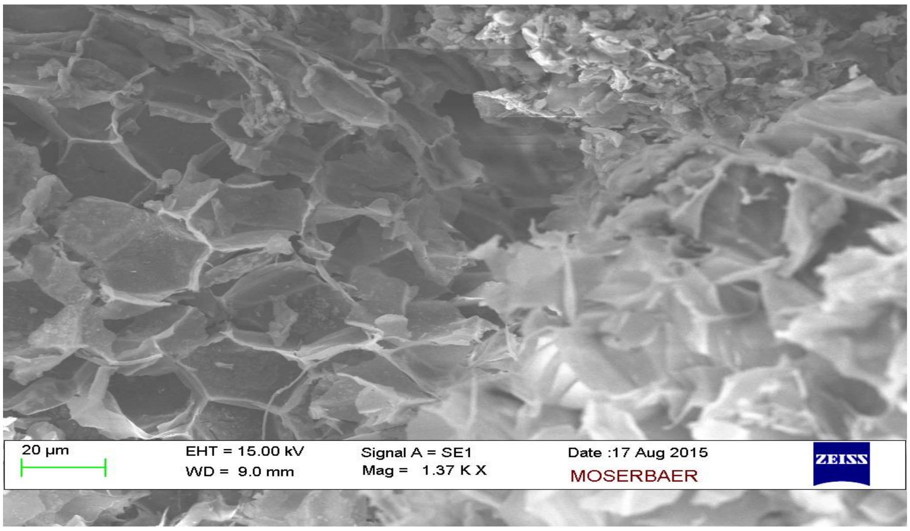

Figure 3a

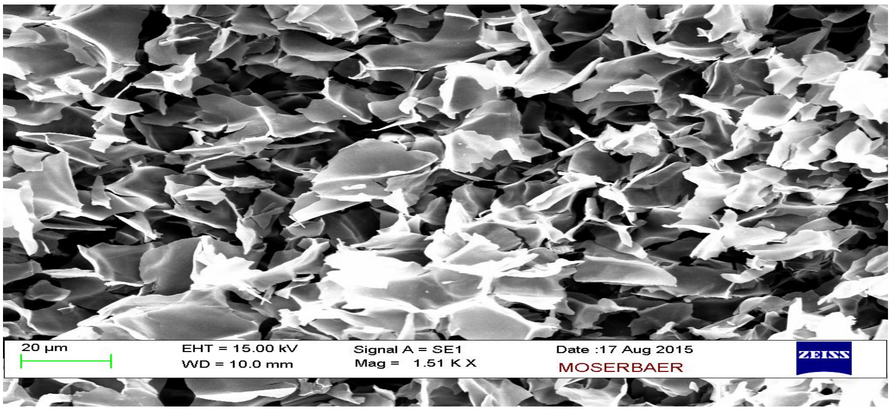

Figure 3b

Figure 3: SEM analysis of Belpatra bark before (**3a)** and after (**3b)** carbonization

4.2 FTIR Analysis

The FTIR spectra of BBC before and after adsorption of Red RB dye are shown in figure 4a and 4b respectively. Figure 4a shows various characteristic bands in raw wood sample related to different functional groups, such as a broad and weak band was observed at 3300 cm^{-1} associated with the stretching vibration of –OH group. The band at 2900 cm^{-1} ascribed to the stretching vibration of –CH group. Furthermore, there was one characteristic band observed at 1040 cm^{-1} which was attributed to C-O-C stretching vibration.

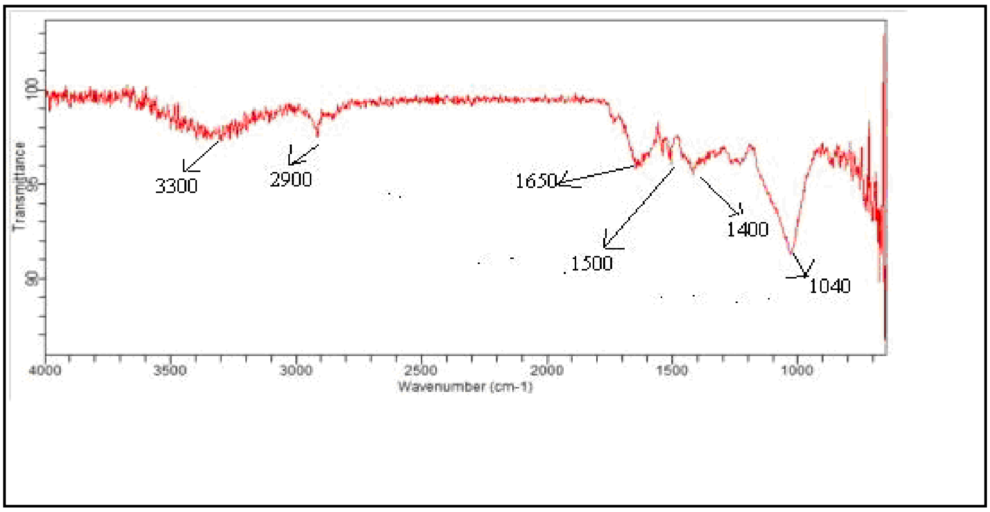

Figure 4a: FTIR spectrum of BBC before adsorption

From figure 4b, it could be seen that the FTIR spectrum of activated carbon after adsorption is almost same, but a slight reduction in intensity of characteristic bands has been observed. It is clearly suggested that adsorption of Red RB dye onto the BBC takes place through physical forces. Some new interactions have also been noticed between the functional groups of adsorbent and adsorbate molecules, which proved the adsorption of dye onto the surface of adsorbent.

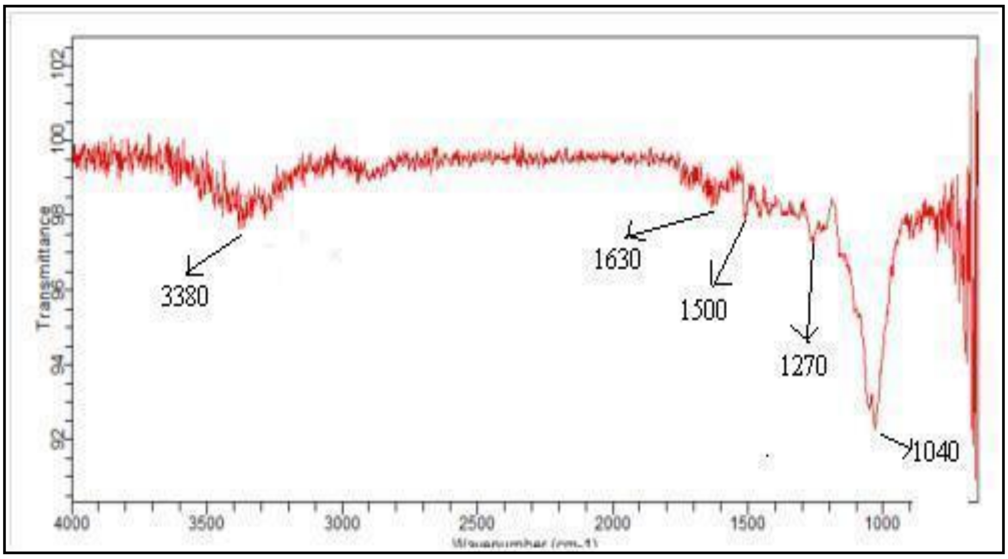

Figure 4b: FTIR spectrum of BBC after adsorption

4.3 Batch Experiments for Dye adsorption

The amount of dye adsorbed onto BBC and percentage removal of dye were calculated by using equation 1 and 2 respectively.

$$q_e = \left(\frac{C_o - C_e}{M}\right) \text{x V} \qquad (1)$$

$$\% \text{ Dye removal} = \left(\frac{C_o - C_e}{C_o}\right) \text{x } 100 \qquad (2)$$

Where q_e is the amount of dye adsorbed at equilibrium, C_o is the initial concentration of dye in the solution, C_e is the final concentration of dye at equilibrium, M is the mass of adsorbent (g) and V is the volume of solution (l). Concentration of dye was calculated by plotting a calibration curve at wavelength 527 nm (Figure 5).

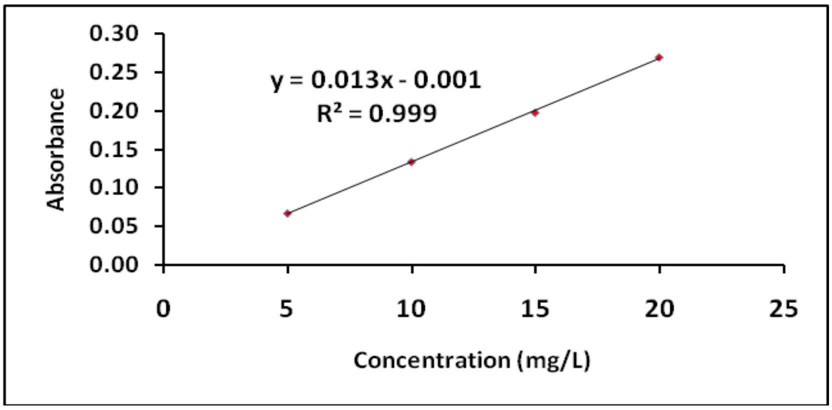

Figure 5: Calibration curve of Red RB Dye at wavelength of 527 nm

The influence of variable parameters for color removal of Red RB dye was studied as follows.

4.3.1 Influence of contact time and initial dye concentration

The influence of initial dye concentration for the color removal of Red RB dye onto BBC is shown in figure 6. For this purpose five initial dye concentrations 10, 15, 20, 25 and 30 mg/L were used with adsorbent dosage 0.5gm/100 ml, particle sizes 150-299 µm, temperature 320 K and pH 3.0 for contact time of 90 minutes. It was seen clearly that maximum adsorption occurred in 15-20 minutes and after 35- 40 minutes; an equilibrium state was obtained in all cases. This is because, each adsorbent has limited adsorbent sites and after a certain time, these were exhausted and adsorption process attained an equilibrium state [4].

It was also found that as the initial dye concentration increased from 10 to 30 mg/L, the percentage of dye removal decreased from 92.8 to 70.1 % because at lower concentration, there were sufficient active adsorbent sites for adsorption of presently available dye molecules, but at higher concentration, due to the inadequate ratio of active sites with dye molecules, adsorption is less [5].

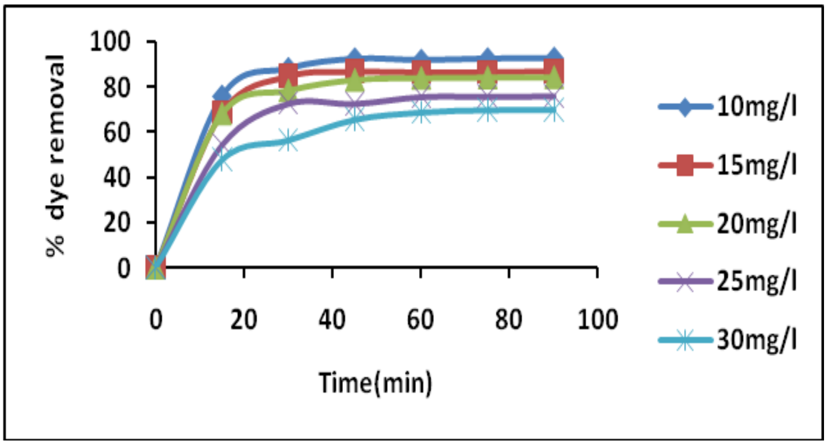

Figure 6: Effect of contact time and initial dye concentration on adsorption of Red RB dye onto BBC (Temp.=310 K, pH=3.0, adsorbent dosage=0.5 gm/100ml, particle sizes = 150-299 µm)

4.3.2 Influence of Particle size

The influence of particle size of adsorbent was studied by taking four ranges of particle sizes; 75-149 µm, 150-299 µm, 300-599 µm and 600-899 µm in conjunction with 20 mg/L dye concentration, 0.5gm/100 ml adsorbent dosage, 320 K temperature and 3.0 pH (Figure 7). It was observed as the size of the particles was decreased, exposed surface area was increased many folds [6, 7] and percentage of dye removal increased accordingly (63.9 to 94.0%).

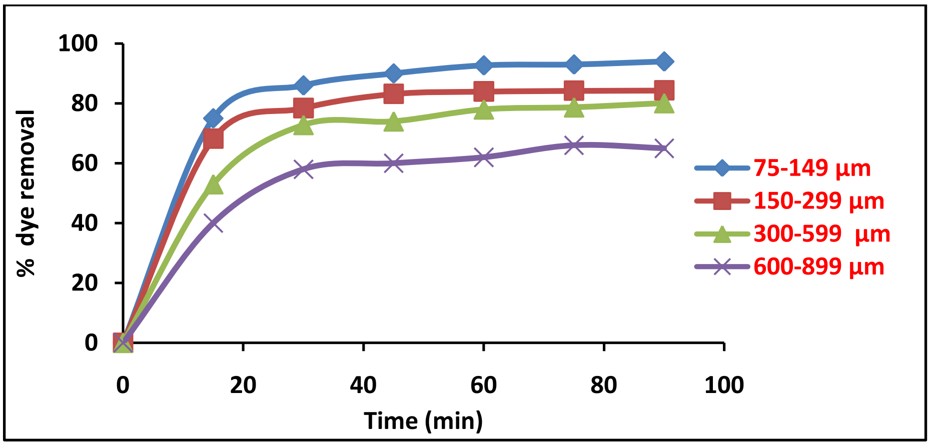

Figure 7: Effect of particle size on adsorption of Red RB dye onto BBC (Temp. = 310K, pH=3.0, adsorbent dosage = 0.5 gm/100ml, dye conc. =20mg/L)

4.3.3 Influence of Adsorbent dosage

The influence of four initial adsorbent dosage 0.3, 0.5, 0.7 and 1.0 gm on adsorption of Red RB dye was examined with 20 mg/L dye concentration, 150-299 µm particle size, 320 K temperature and 3.0 pH . It was seen from figure 8 that removal of dye increased from 76.0 to 91.7%, when the amount of adsorbent is increased. This is because active adsorption sites (surface area) were increased with increasing dosage of adsorbent and thereby leading to more adsorption [8, 9], since adsorption is a surface phenomenon.

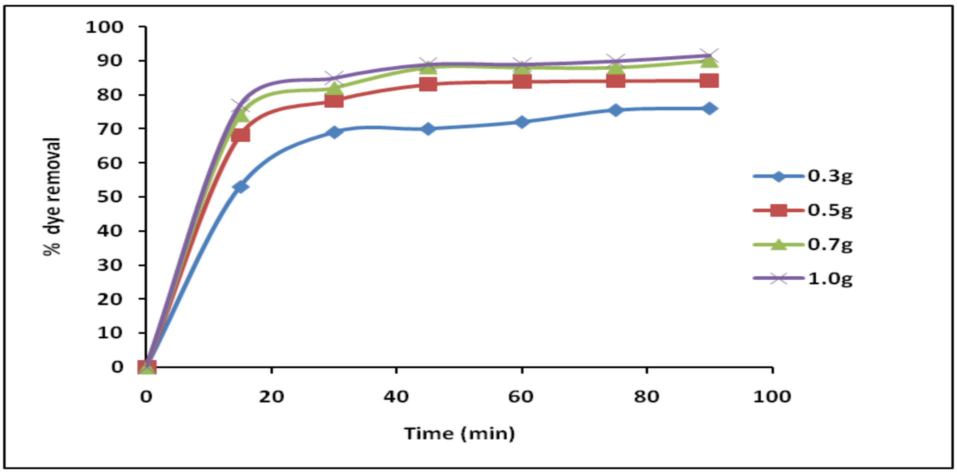

Figure 8: Effect of adsorbent dosage on adsorption of Red RB dye onto BBC (Temp. = 310 K, pH = 3.0, dye conc. = 20mg/L, particle size = 150-299µm)

4.3.4 Influence of pH

The influence of pH of the system on adsorption capacity was studied by adjusting five initial pH viz. 1.5, 3.0, 4.5, 6.0 and 7.5 of the solution and keeping other parameters of the system constant. It was studied from figure 9 that dye removal was maximum (93.7%) at pH 1.5. This was due to fact that at low pH, surface of the adsorbent was positively charged by absorbing H^+ ions. This condition increased the adsorption of anionic dye (Red RB) on positively charged activated carbon surface due to electrostatic attraction. As the pH of the solution increased, the surface of adsorbent gradually acquired excess of OH^- ions, this repelled the dye molecules and retarded the adsorption (93.0 to 58.5%). Similar results were reported earlier [10, 11].

4.3.5 Influence of temperature

The influence of operating temperatures viz. 300, 310, 320 and 330K while keeping other variables constant, for the color removal of Red RB dye is shown in figure 10. It is clear from the figure 10 that the dye removal decreased from 90.0 to 78.1 % with increasing temperature from 300 to 330 K. It was due to the fact that the adsorptive forces between adsorbate and the active sites on the adsorbent became weak by increasing the temperature so that dye removal was decreased [12,13].

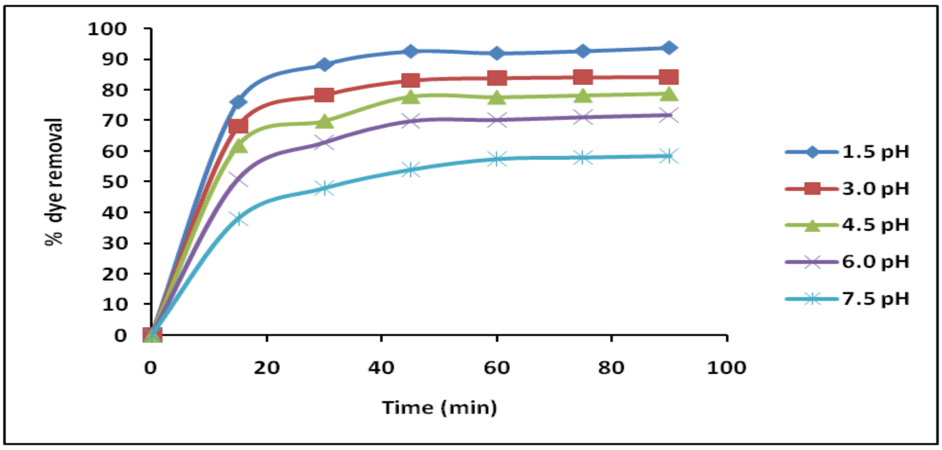

Figure 9: Effect of pH on adsorption of Red RB dye onto BBC (Temp. = 310 K, adsorbent dosage = 0.5 gm/100ml, dye conc.= 20mg/L, particle size = 150-299 µm)

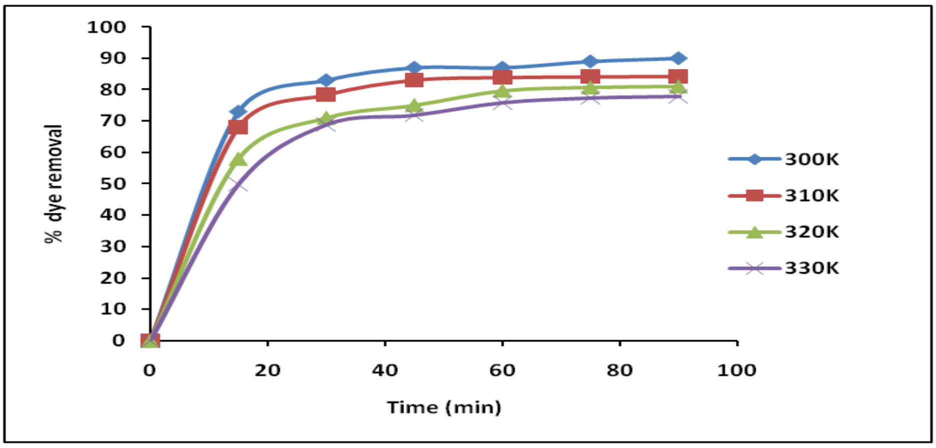

Figure10: Effect of temperature on adsorption of Red RB dye onto BBC (pH = 3.0, adsorbent dosage=0.5 gm/100ml, dye conc. = 20mg/L, particle size = 150-299 µm)

4.4 Estimation of thermodynamic parameters

The equilibrium constants ($K_{1,}$ $K_{2,}$ K_3 and K_4) at temperature 300, 310, 320 and 330 K were calculated according to the relation:

$$K_{equ} = \frac{C_{ads}}{C_{sol}} \quad (3)$$

Where C_{ads} and C_{sol} are the concentrations of dye on the adsorbent surface and in solution respectively.

The various thermodynamic parameters like Gibb's free energy change (ΔG°), enthalpy change (ΔH°) and entropy change (ΔS°), helpful in determining the spontaneity of adsorption process can be calculated by using the equilibrium constants through following equations:

$$\Delta G^\circ = - RT \ln K_{equ} \quad (4)$$

$$\Delta H^\circ = R\left(\frac{T_2 T_1}{T_2 - T_1}\right) \ln\left(\frac{K_2}{K_1}\right) \quad (5)$$

$$\Delta S^\circ = \left(\frac{\Delta H^\circ - \Delta G^\circ}{T}\right) \quad (6)$$

The values of thermodynamic parameters are given in table 1. From the results, it was concluded that Gibb's free energy values were found to be negative, suggesting feasibility and spontaneous nature [14] of adsorption of dye onto BBC in the temperature range of 300 to 330 K. The negative values of entropy change indicate that decreased disorder and randomness. Same results have been reported previously [15, 16]. The low negative values of enthalpy change imply physical adsorption which is exothermic in nature [17].

Table 1: Thermodynamic parameters of Red RB dye at different temperatures

Temp. (K)	**Thermodynamic Parameters**		
	$-\Delta G^o$ [KJ/mol]	**$-\Delta H^o$ [KJ/mol]**	**$-\Delta S^o$ [J/(Kmol)]**
300	5.479	39.936	113.000
310	4.331	19.107	47.666
320	3.855	16.263	38.775
330	3.468	_	_

4.5 Adsorption Kinetics

To evaluate the dynamics of adsorption process, the kinetic data was tested with pseudo first order and pseudo second order kinetic models. Integrated pseudo first order and pseudo second order rate expressions are given by equation 7 and 8 respectively.

$$\log(q_e - q) = \log\ q_e - \frac{k_1}{2.303}t \qquad (7)$$

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \qquad (8)$$

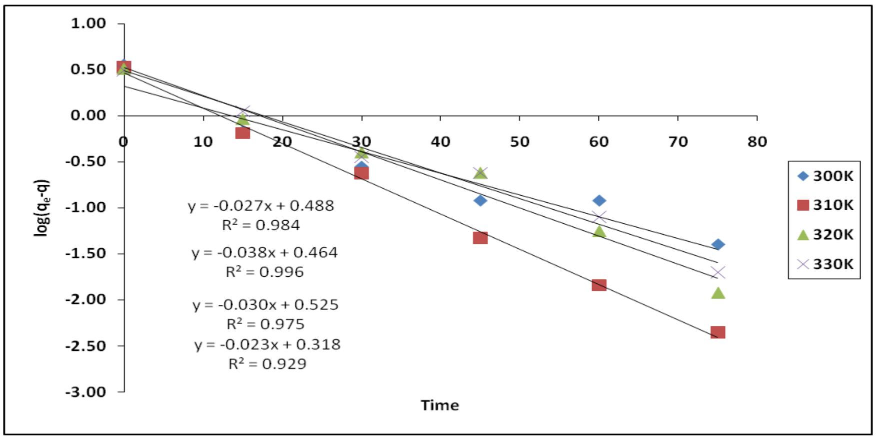

Figure 11: Pseudo first order plot for the adsorption of Red RB dye onto BBC at different temperatures.

Where q_e is the amount of dye adsorbed at equilibrium and q is the amount of dye adsorbed at various time t, k_1 and k_2 are the pseudo first order and pseudo second order rate constants respectively.

The plots of log (q_e-q) versus t for pseudo first order and t/q versus t for pseudo second order at different temperatures are depicted in figure 11 and 12 respectively. The values of k_1, k_2, R_1^2 and R_2^2 and q_e under different temperatures were calculated from these plots for the adsorption of Red RB dye onto the BBC and are given in table 2. Although both plots show linear relationship, but the correlation coefficients R_1^2 for pseudo first order reaction are found between 0.929-0.996 while R_2^2 for pseudo second order reaction are found between 0.992-0.998. Experimental q_e values were also found close to the pseudo second order reaction. Excellent linearity with high correlation coefficients and good agreement between calculated and experimental results for pseudo second order reaction proved that pseudo second order reaction got fitted better than pseudo first order reaction. Same results have also been reported in literature [18, 19].

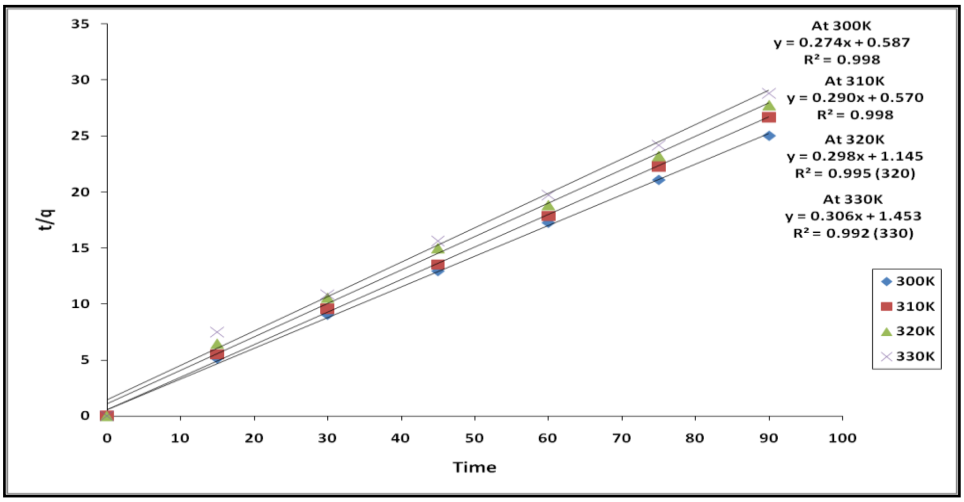

Figure12: Pseudo second order plot for the adsorption of Red RB dye onto BBC at different temperatures.

Table 2: Kinetic Parameters For Adsorption of Red RB Dye onto BBC

Temperature (K)	**Pseudo First Order**			**Pseudo Second Order**			**Experimental q_e(mg/g)**
	Calculated q_e (mg/g)	k_1 (min^{-1})	R_1^2	Calculated q_e(mg/g)	k_2 (g/mg/min)	R_2^2	
300	2.080	0.053	0.929	3.649	0.128	0.998	3.60
310	2.911	0.088	0.996	3.448	0.148	0.998	3.37
320	3.350	0.069	0.975	3.355	0.078	0.995	3.24
330	3.076	0.062	0.984	3.267	0.064	0.992	3.12

4.6 *Activation Energy*

The pseudo second order rate constants (k_2) at different temperatures were used to calculate the activation energy of the system by the following Arrhenius equation:

$$\ln k_2 = \ln A - \frac{Ea}{RT} \tag{9}$$

Where Ea is the Arrhenius activation energy, A is the Arrhenius factor, R is the gas constant and T is the absolute temperature. The plot of $\ln k_2$ versus 1000/T is shown in figure 13. The activation energy can be determined from the slope of the plot which is equal to -Ea/ R. Activation Energy is very helpful to decide the type of adsorption i.e., physical or chemical. Lower value of activation energy between 5-40 KJ/mol indicates physical adsorption whereas higher values of the activation energy between 40- 800 KJ/mol suggests chemical adsorption [18, 20]. The obtained value of the activation energy from the slope of the plot is **5.863** KJ/mol, which reveals that adsorption of tested dye onto BBC is through the physical process involving weak forces of attraction.

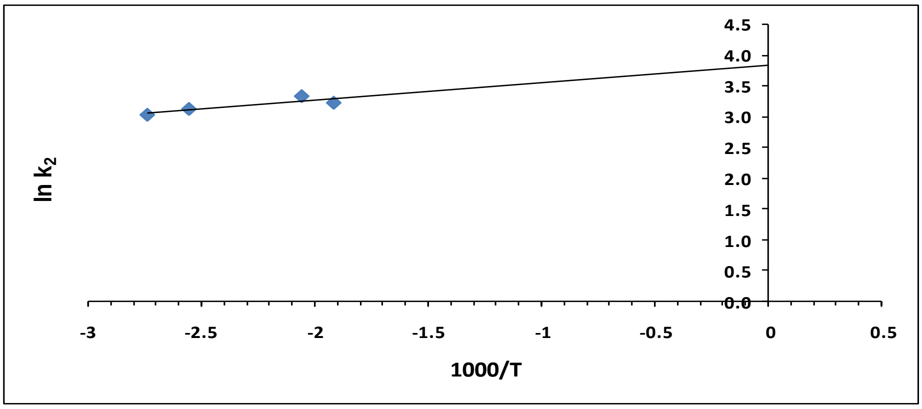

Figure 13: Arrhenius Plot for the adsorption of Red RB Dye onto BBC

4.7 Adsorption Isotherms

Adsorption Isotherms or equilibrium data are helpful to describe the sorption interaction and adsorption capacity of adsorbent. The suitability of appropriate model to design the adsorption process was done by comparing the correlation coefficients for the following three models.

4.7.1 Langmuir Adsorption Isotherm

The Langmuir adsorption isotherm predicts the monolayer coverage of adsorbate at the surface of adsorbent. It is applicable only for homogeneous sites within the adsorbent. The chemical equation for Langmuir adsorption isotherm is given by following expression.

$$\frac{C_e}{q_e} = \frac{1}{Q^{\circ}b} + \frac{C_e}{Q^{\circ}} \qquad (10)$$

Where C_e is the equilibrium concentration of dye (mg/L); q_e is the amount of dye adsorbed at equilibrium (mg/g); Q^0 (mg/g) and b (L/mg) are the Langmuir constants. The plot of C_e/q_e versus C_e (Figure 14) gives straight line (R^2=0.995). The slope and intercept of the plot were used to calculate the values of Q^o (mg/g) and b respectively, which are given in table 3.

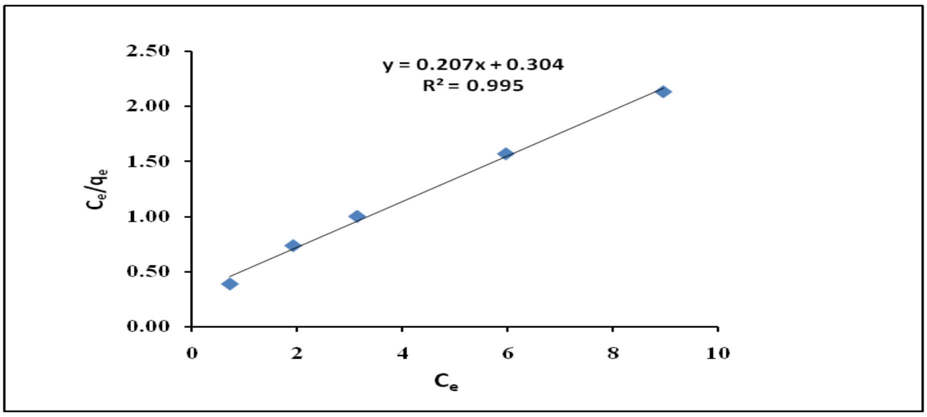

Figure 14: Langmuir adsorption isotherm for Red RB dye

4.7.2 Freundlich Adsorption Isotherm

Freundlich adsorption isotherm explains both the monolayer as well as multilayer coverage of adsorbate at the surface of the adsorbent. It is applicable only for heterogeneous surface of adsorbent.

The logarithmic equation for Freundlich adsorption isotherm is given by following expression.

$$\log q_e = \log K_f + \frac{1}{n}\log C_e \qquad (11)$$

Where K_f and 1/n are Freundlich constants related to adsorption capacity (L/mg) and adsorption intensity respectively. A plot of log q_e versus log C_e (Figure 15) was used to calculate the value of K_f and 1/n from intercept and slope respectively. These values are given in table 3.

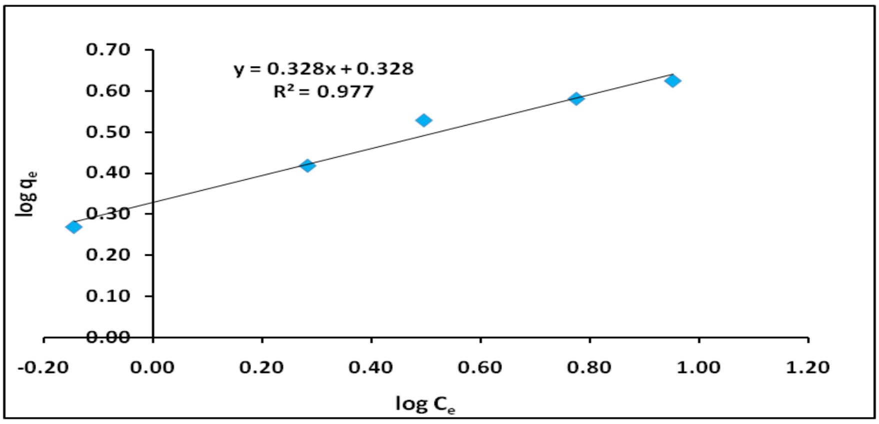

Figure 15: Freundlich adsorption isotherm for Red RB dye

4.7.3 Temkin Adsorption Isotherm

Temkin adsorption isotherm predicts that the adsorption energy decreases linearly with the surface coverage [21]. It is expressed by following linear equation.

$$q_e = B \ln A + B \ln C_e \tag{12}$$

Where, $B = \frac{RT}{b}$, A (L/g) is Temkin isotherm constants or equilibrium binding constant and b is also Temkin constant related to the heat of sorption. R is the gas constant (8.314 J/mol) and T is the absolute temperature (K). The plot of q_e versus ln C_e is shown in figure 16.

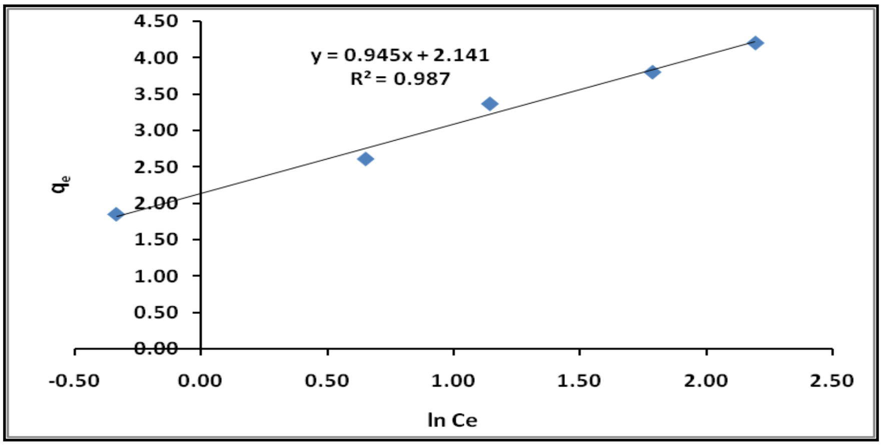

Figure 16: Temkin adsorption isotherm for Red RB dye

Although all the above three plots are almost linear, but the value of Regression coefficient is the highest for Langmuir adsorption isotherm (R^2 = 0.995), which shows that Langmuir adsorption isotherm suites with the experimental data very well. All the parameters of Langmuir, Freundlich and Temkin adsorption isotherm are summarized in table 3.

Table 3: Langmuir, Freundlich and Temkin adsorption constants of Red RB dye onto BBC

Langmuir Adsorption Isotherm		
Q^o	b	R^2
4.831	0.681	0.995
Freundlich Adsorption Isotherm		
n	K_f	R^2
3.049	2.128	0.977
Temkin Adsorption Isotherm		
B	A	R^2
0.945	9.637	0.987

Conclusions

It was concluded that the percent color removal of Red RB dye from aqueous solution by using activated carbon (BBC) prepared from plant material (Belpatra Bark) increased with the decrease in initial dye concentration, particle size, temperature and pH of the solution. However, it was also increased with increasing dosage of adsorbent. From the thermodynamic analysis, it was proved that adsorption process was exothermic, feasible and spontaneous. It was found that it can remove maximum 94.0% dye in 20 mg/L dye concentration having 75-299 µm particle sizes of adsorbent, 0.5 gm adsorbent dosage at 320 K temperature and 3.0 pH in only 40-45 minutes.

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