



## Research Article

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### Adsorption of Bismarck Brown R Dye from Aqueous Solution onto Activated Carbon Prepared from *Delonix Regia Pods shell* (Flame Tree)

A.Rajappa<sup>1</sup>, K.Ramesh<sup>2</sup> and V. Nandhakumar<sup>3\*</sup><sup>1</sup> Department of chemistry, Sri Manakula Vinayagar Engineering College, Madagadipet, Puducherry, India.<sup>2</sup> Department of chemistry, Arasu Engineering College, Kumbakonam, Thanjavur, India.<sup>3\*</sup> Department of chemistry, A.V.V.M Sri Pushpam college, Poondi, Thanjavur, India.

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#### Abstract

Commercial Activated Carbon has been widely used as a good adsorbent. Activated carbon prepared from *Delonix regia pods* shell (Flame tree) may be alternative an adsorbent. This work deals with the study of adsorption Bismarck Brown R dye from aqueous solution onto Carbon prepared from *Delonix regia pods* (Flame tree) activated with Zinc chloride (DRZAC) under various experimental conditions. Batch mode adsorption experiments were conducted. The characteristics of the DRZAC were determined by BET analysis and  $\text{pH}_{\text{ZPC}}$ . Experimental data obtained were fitted with linearised forms of Ho and Lagergren kinetic models. The Sum of Error Squares Percentage (SSE %) for second order and first order kinetics were 2.73 & 5.56 respectively and hence this adsorption followed second order kinetics. Equilibrium isotherms data were fitted with Langmuir, Freundlich, Tempkin and Dubinin-Raduskevich isotherm equations. The equilibrium data was best described by Freundlich isotherm model. Significance of these results were discussed in detail. The results in this study indicated that DRZAC could be employed as an adsorbent for the removal of Bismarck Brown R dye from an aqueous solution.

**Keywords:** Adsorption, *Delonix Regia ZnCl<sub>2</sub> Activated Carbon*, Bismarck Brown R Dye, Kinetic Models, Isotherms.

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#### \*Corresponding author

V. Nandhakumar

Department of Chemistry,  
A.V.V.M Sri Pushpam College,  
Poondi, Thanjavur, India.  
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#### 1. Introduction

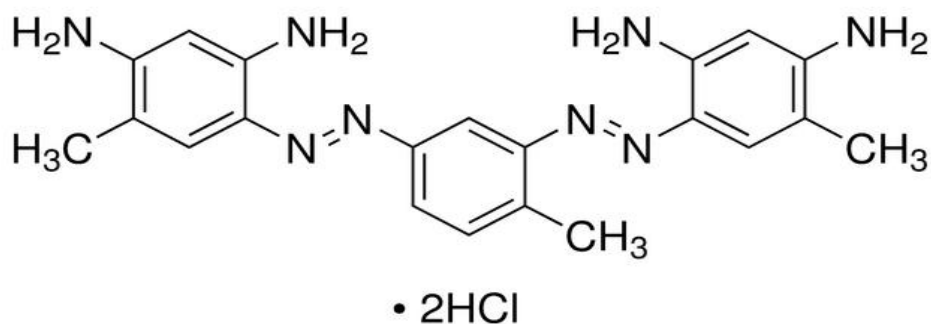
Colored compounds comprising pigments and dyes are used widely in textile and leather dyeing, paper, printing, pharmaceutical and cosmetic industries. 10,000 different dyes weighing about 0.7 million tons are produced annually

for various industrial processes [1]. A percentage of these dyes are lost in the effluent during the dyeing process. Many of these are toxic or even carcinogenic [2]. Discharge of these into water bodies would pollute the water and make it unfit for aquatic life by their toxicity. Further, the dyes would color the water and make penetration of sunlight to the lower layers impossible. So, it is not possible for aquatic plants to perform photosynthesis [3]. Polluted water not only damages plants and animals but also harms the environment. Majority of these dyes are stable to light and oxidation. They are immune to aerobic digestion. Biological treatment may remove BOD, COD and suspended solids but remains ineffective in removing the color of dyes. Many physical methods such as adsorption, coagulation, precipitation and filtration have been attempted for treatment of dye containing effluent. Chemical methods such as oxidation have also been used. Biological methods would be economically simple but most dyes are resistant to bacterial degradation [3].

The potential of various methods have been explored and adsorption of the chemical dyes has been found to be the most effective. Commercially available activated carbon as an adsorbent has yielded excellent results [4]. However, taking into account the high costs involved in such a process, feasibility of alternate adsorbents have been studied. These include industrial as well as agricultural byproducts. Industrial waste products include slurry from fertilizer plant and blast furnace slag. A variety of agricultural products have been used namely rice straw, coconut husk, rice husk, bagasse and tree bark [5]. In a country like India *Delonix regia pods* (Flame tree) is abundantly available.

Azo dyes are divided according to the presence of azo bonds ( $-N=N-$ ) in the molecule; these include mono azo, diazo, triazo etc [6]. Azo dyes resist the effect of oxidation agents and light, thus they cannot be completely treated by conventional methods of anaerobic digestion [7]. It is necessary to find an effective method for the treatment of Bismarck Brown R. The degradation of Bismarck brown R dye in the presence of aqueous zinc oxide suspension has been reported before [8]. The adsorption technique proved to be an effective and attractive process for removing dyes from aqueous solutions in term of initial cost, ease of operation, insensitivity to toxic substance, high efficiency, easy recovery and simplicity of design [9].

#### Structure of Bismarck Brown R:



*Delonix Regia pods shell*



*ZnCl<sub>2</sub> Activated carbon*

In recent years, Carbon prepared from *Delonix Regia pods* have been accepted as one of the most appropriate low cost adsorbent. Investigations have been carried-out to evaluate the adsorption of dyes onto a wide range of *Delonix Regia pods*.

## 2. Materials and Methods

### 2.1 Materials:

All the chemicals used for this experiment are of analytical grade. Activated Carbon was prepared from *Delonix Regia pods* shell. Bismarck brown R dye purchased from Merck Company.

### 2.2 Preparation of Dye Solution:

Bismarck brown R dye was used without further purification. The dye stock solution was prepared by dissolving appropriate amount of accurately weighed dye in double distilled water to a concentration of 500 mg/L. The experimental solutions were prepared by proper dilution.

### 2.3 Preparation of Adsorbent:

*Delonix Regia pods* were collected the region in Kumbakonam, Thanjavur District. *Delonix Regia pods* repeatedly washed with distilled water to remove dirt, dust and impurities. The washed shells materials were then dried in sunlight for a month. The dried shells were chopped into small chips. Chemical activation of the *Delonix Regia pods* was carried out with  $ZnCl_2$  solution. 20g of dried *Delonix Regia pods* were well mixed with 20ml of  $ZnCl_2$  1:1 w/v aqueous solution. The slurry was kept in an air oven at  $100^\circ C$  for 24 hours. The resulting product was then subjected to carbonization and activation process in a muffle furnace at  $400^\circ C$  for 1 hour. Then the sample was cooled and subsequently washed with 0.5M HCl and then with deionized water for several times. It was ground and sieved. The particle size ranged between 110  $\mu m$  & 90  $\mu m$  were taken and kept in a desiccator for further use.

### 2.4 Physical Characterization of DRZAC

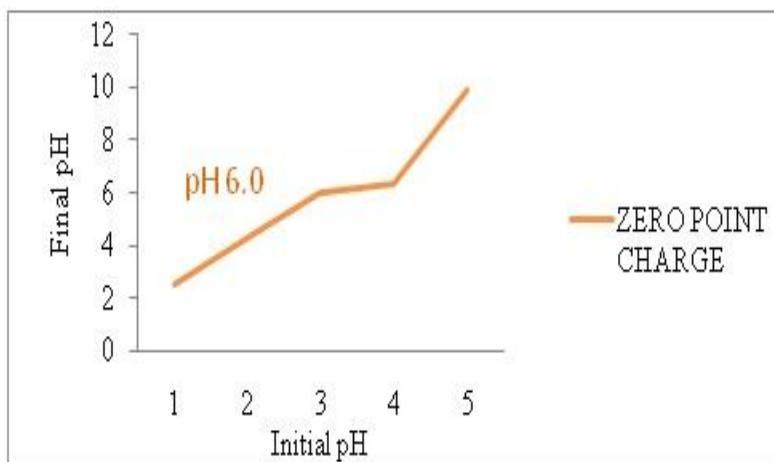
The properties of DRZAC were given in Table 1. The total surface areas were calculated using the BET equation [10].

**Table 1:** Physico-chemical characteristics of DRZAC

S.No	Properties	DRZAC Values
1.	pH <sub>zpc</sub>	6.0
2.	Particle size, $\mu m$	90-110
3.	Surface area (BET), $m^2/g$	916.1234
4.	Pore volume, $cm^3/g$	0.3986
5.	Pore size (Pore width), nm	2.7174
6.	Bulk density, g/mL	0.52
7.	Fixed Carbon, %	71.11
8.	Moisture content, %	4.36

### 2.5 Determination of point of zero charge:

20 mg of the DRZAC was added to five such mixture were prepared 50ml of 50 ppm Bismarck brown R dye solution taken in 250 ml of Iodine flask. The initial pH values of the solutions were adjusted to 2.0 to 10.0 by adding either 0.1M HCl or 0.1M NaOH. The mixtures were allowed to equilibrate for 24 hrs with periodic shaking. The pH values of the supernatant liquid were determined. A plot of the initial pH versus final pH was drawn. From the horizontal portion, the pH<sub>zpc</sub> was evaluated which is shown in figure 1 [11].



**Figure 1:** point of zero charge

## 2.6 Adsorption Experiments:

20mg of DRZAC was interacted with 50ml known concentration of Bismarck brown R dye solution in a Iodine flask at predetermined temperatures. The mixtures were agitated in a Mechanical shaker at 180 rpm continuously for predetermined time intervals. It was then centrifuged. The concentration of dye in the centrifugate was determined using Systronics Double Beam UV-visible Spectrophotometer: 2202 at 470 nm. The amount adsorbed at equilibrium,  $q_e$  (mg/g), was calculated as follows:

$$q_e = C_0 - C_e / W \times V$$

Where,  $C_0$  and  $C_e$  (mg/L) are the liquid-phase concentrations of BBR dye at the initial stage and the equilibrium respectively.  $V$ (L) is the volume of the Bismarck brown R dye solution and  $W$ (g) is the weight of the adsorbent used. The percentage dye removal was calculated as:

$$\text{Percentage of removal} = C_0 - C_e / C_0 \times 100$$

## 2.7 Effect of temperature:

To observe the effect of temperature on the adsorption capacity, experiments are carried out at three different temperature (30,40 and 50°C) using 20mg of DRZAC per 50 ml 50 ppm of the solution. The experiment was carried out in a constant temperature water bath. It was seen that as the temperature was increased, the dye removal was also increased [12].

## 3. Result and Discussion

### 3.1 Effect of Contact time and initial concentration:

The effect of contact time on percentage removal of dye for different initial concentration have been shown in figure 2. Adsorption of dye from the solution increases with the time and finally attains equilibrium in 80,100 and 120 Minutes for the initial concentrations of adsorbates 50,100 and 150  $\text{MgL}^{-1}$  respectively. The percentage of removal increased with the increase in contact time and decreased with the increase of initial concentration of the dye. However the amount of dye adsorbed on the adsorbent increased with the increase of initial concentration of the dye solution [13] which is depicted in figure 3 and given in table 2.

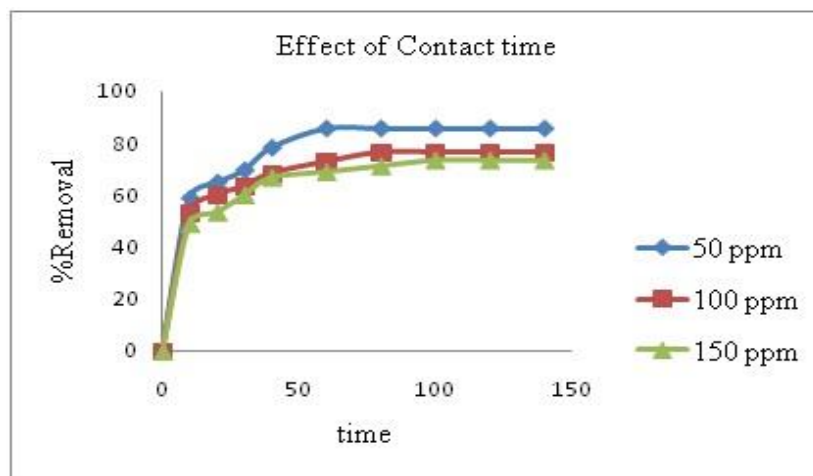


Figure 2: Effect of Contact time and initial concentration

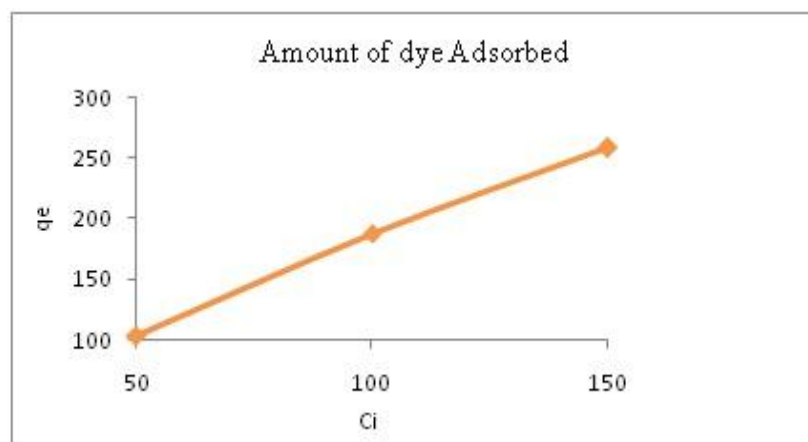


Figure 3: Amount of dye adsorbed at equilibrium

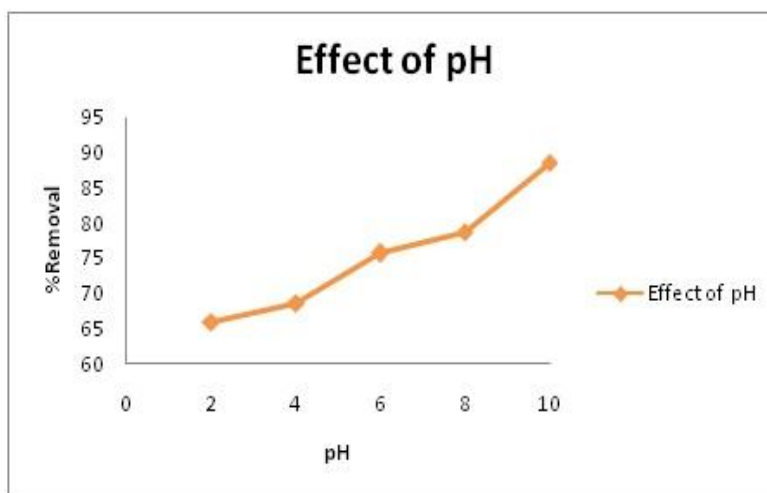
**Table 2:** Percentage of removal of dye and amount of dye adsorbed

Ci (mg/L)	% of Removal of dye At equilibrium	Adsorption capacity at equilibrium (mg/g)
50	85.34	104.17
100	76.33	187.50
150	73.33	258.33

### 3.2 Effect of pH:

Figure 4 shows the effect of pH on the adsorption of BBR dye onto DRZAC. It is found that % of removal of dye from aqueous solution increases with the increase of pH of the solution. This may be due to two reasons, one reason is accumulation of  $H^+$  ion on the surface of the adsorbent at lower pH may exert a repulsive force towards the cationic dye approaching the adsorbent. As the pH of the solution increases up to 7, the concentration of the accumulated ions on the surface decreases and hence the repulsive force exerted on the approaching cationic dye decreases which facilitates the increase of percentage removal of the dye solution.

Increase of pH from 7 to 14 may increase the accumulation of  $OH^-$  ion on the surface of the adsorbent which attracts the cationic dye, thus the smaller  $OH^-$  ions are gradually replaced by larger dye ions. This effect becomes more pronounced as the increase of pH from 7 to 14. The second reason is increase of pH beyond 7 may precipitate the cationic dye. In order to dye at alkaline medium, experiments were carried out with and without adsorbent at alkaline pH. It is found the concentration of dye solution remains constant in the alkaline solutions having no adsorbent, but the concentrations of dye were found to decrease in alkaline solutions having the adsorbents. Thus the possibility of dye removal due to precipitation was not happened. However, the other adsorption experiments were carried out at neutral pH only.

**Figure 4:** Effect of pH

## 4. Adsorption kinetics

Experimental data were fitted into two kinetics models such as pseudo-first-order and pseudo-second-order kinetics models to examine the adsorption kinetics.

### 4.1 Pseudo-First-Order Model:

The adsorption kinetics can be described by a pseudo first order equation as suggested by Lagergren [14].

$$\text{Log} (q_e - q_t) = \text{Log} q_e - \frac{k_1}{2.303} t$$

Where  $q_e$  and  $q_t$  are the amount of dye adsorbed (mg/g) at equilibrium and at time  $t$  (min) respectively and  $k_1$  is the rate constant of adsorption (l/min). The values of  $k_1$  and theoretical  $q_e$  for different initial concentrations calculated from the slope and intercepts of these curves respectively were presented in Table 3.

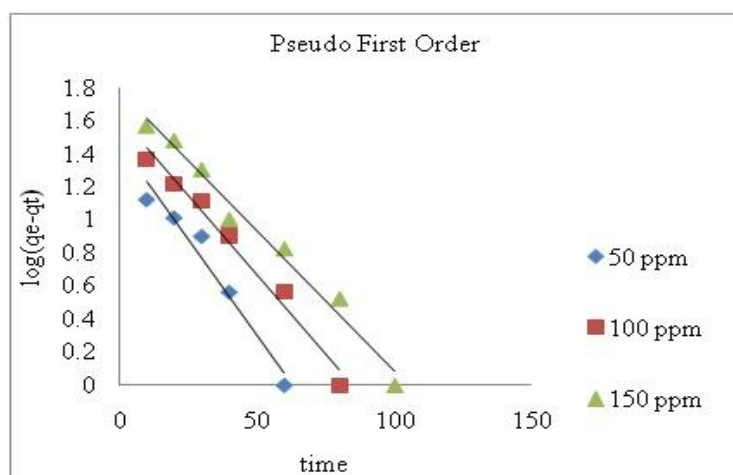


Figure 5: Pseudo-First-Order Model

#### 4.2. Pseudo-Second-Order Model:

The adsorption kinetics can be described by a pseudo second order equation as suggested by Ho [15].

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$

Theoretical equilibrium adsorption capacity ( $q_e$ ), and the second-order rate constant  $k_2$  (g/(mg min)) can be determined experimentally from the slope and intercept of plot of  $t/q_t$  versus  $t$  (Figure 6). The  $k_2$  (g/ (mg min)) and correlation coefficients values calculated were listed in Table 3.

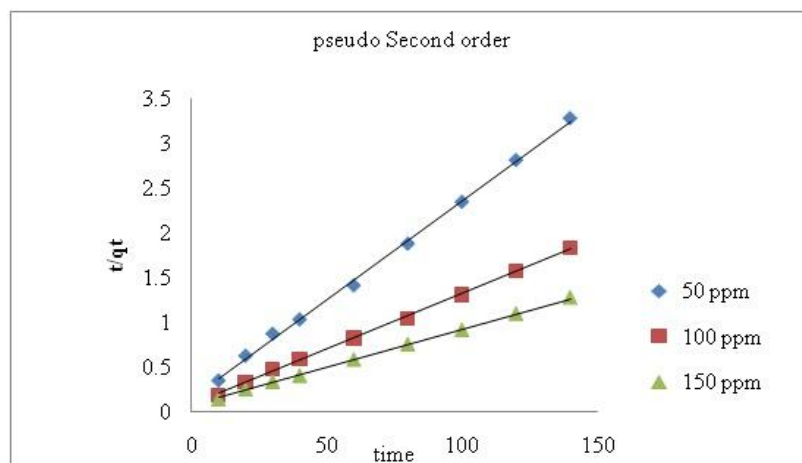


Figure 6: Pseudo-Second-Order Model

#### 4.3 Test for kinetics models:

The sum of error squares (SSE%) [14] is one method which has been used in literature to test the validity of models used. The sum of error squares is given as follows;

$$SSE (\%) = \frac{[(q_e)_{exp} - (q_e)_{cal}]^2}{N}$$

Where N is the number of data points.

Experimental  $q_e$ , calculated  $q_e$  and SSE for the pseudo first order and pseudo second order kinetics were given in Table 3. It shows that  $q_e$  (exp) is close to  $q_e$  (cal) for second order kinetics for DRZAC. It can be seen that SSE (%) value is lower for the second order kinetic model than that of pseudo first order kinetic model. This confirms the applicability of the pseudo second order kinetic model well. The determination coefficient ( $R^2$ ) for pseudo first order model ranged between 0.956 to 0.981 whereas these values for the second order model were ranged between 0.998 to 0.999. This also indicates that the experimental data best fitted into pseudo

second order. The  $q_e$  values and SSE% values suggest that the process of adsorption follows pseudo second order kinetics

**Table 3:** Kinetic parameters

C <sub>i</sub> mg/L	Rate constants		q <sub>e(cal)</sub> mg/g		q <sub>e(exp)</sub> mg/g		q <sub>e</sub>		R <sup>2</sup>		(SSE %)	
	k <sub>1</sub> (10 <sup>-2</sup> ) (min <sup>-1</sup> )	k <sub>2</sub> (10 <sup>-3</sup> ) (gmg <sup>-1</sup> min <sup>-1</sup> )	First Order	Second order	First order	Second order	First order	Second Order	First order	Second order	First order	Second Order
50	5.29	3.46	42.67	42.67	28.91	45.45	13.76	2.78	0.956	0.998		
100	4.37	1.82	76.33	76.33	41.96	83.33	34.37	7.00	0.977	0.999		
150	3.68	1.00	110	110	59.57	125	50.43	15.00	0.981	0.999	5.56	2.73

## 5. Adsorption Isotherm

The adsorption isotherms of the Bismarck Brown R using the DRZAC at 303k, 313k and 323k have been verified.

### 5.1 Langmuir isotherms:

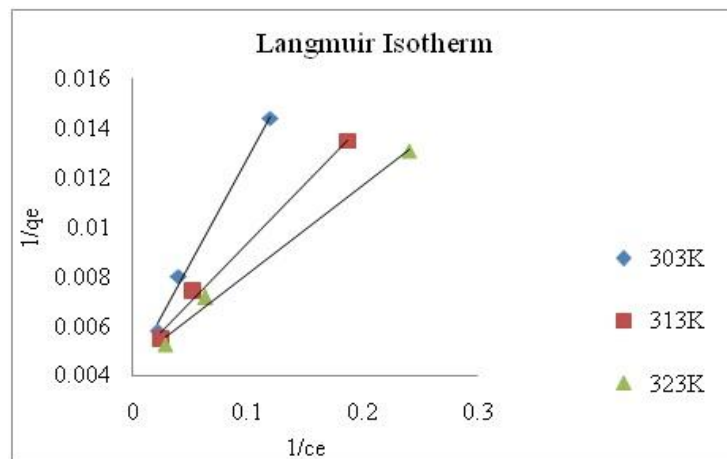
The Langmuir adsorption isotherm assumes that adsorption takes place at specific homogeneous sites within the adsorbent and has found successful application in many sorption processes of monolayer adsorption [16]. Figure 7 shows the Langmuir isotherm for adsorption BBR onto DRZAC. The following equation is the Langmuir isotherm:

$$\frac{C_e}{q_e} = \frac{1}{q_0 b} + \frac{C_e}{q_0}$$

where  $C_e$  is the equilibrium concentration (mg/l),  $q_e$  is the amount adsorbed at equilibrium (mg/g), and  $q_0$  and  $b$  are Langmuir constants related to adsorption efficiency and energy of adsorption, respectively [17]. The constants  $q_0$  and  $b$  calculated from the slope and intercept of the plot of  $C_e/q_e$  vs  $C_e$  are listed in Table 4. The essential characteristics of Langmuir isotherm can be expressed by dimensionless separation factor,  $R_L$  [18].

$$R_L = \frac{1}{(1 + K_L \cdot C_0)}$$

The value of separation factor  $R_L$  indicates the nature of the adsorption process as given below  
 $R_L > 1$  Unfavourable,  $R_L = 1$  Linear,  $0 < R_L < 1$  Favourable,  $R_L = 0$  Irreversible



**Figure 7:** Langmuir isotherm

### 5.2 Freundlich isotherm:

The Freundlich isotherm is an empirical equation employed to describe heterogeneous systems [19]. The Freundlich model [20] is based on the distribution of adsorbate between the adsorbent and aqueous phases at equilibrium. Figure 8 shows the Freundlich isotherm for adsorption BBR onto DRZAC. The basic Freundlich equation is:

$$\text{Log } q_e = \text{Log } K_F + \frac{1}{n} \text{Log } C_e$$

where  $q_e$  is the amount of BBR dye adsorbed (mg/g),  $C_e$  is the equilibrium concentration of BBR dye in solution (mg/l),  $K_F$  and  $n$  are constants incorporating all factors affecting the adsorption capacity and intensity of

adsorption, respectively. The linear plot of  $\log q_e$  versus  $\log C_e$  gives a linear line with a slope of  $1/n$  and intercept of  $\log k_f$  and the results are given in table 4.

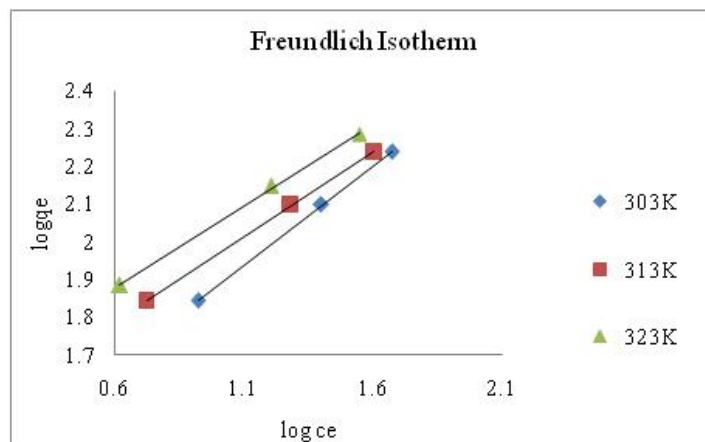


Figure 8: Freundlich isotherm

### 5.3 Temkin Isotherm:

This isotherm takes into accounts of indirect adsorbent - adsorbate interactions and suggests that, because of these interactions the heat of adsorption of all the molecules in the layer would decrease linearly with coverage [21]. Figure 9 shows the Temkin isotherm for adsorption BBR onto DRZAC. The Temkin isotherm has been used in the form given below.

$$q_e = B_1 \ln C_e + B_1 \ln K_T$$

where  $K_T$  and  $B_1$  are the Temkin constants ( $K_T$  is the equilibrium binding constant (L/g) and  $B_1$  is related to the heat of adsorption). The values of the isotherm parameters are given in table 4.

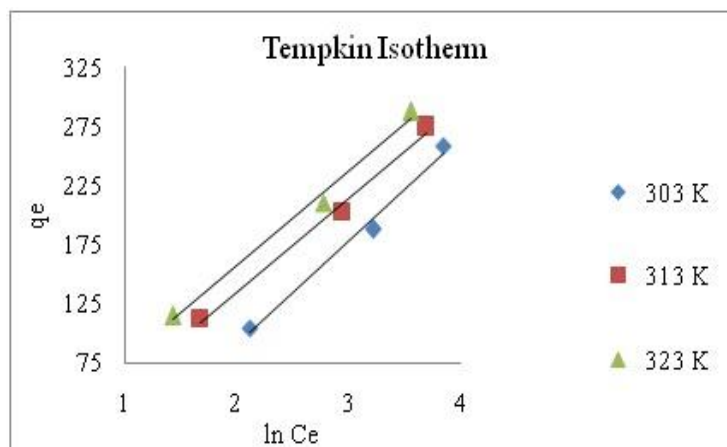


Figure 9: Temkin Isotherm

**5.4. Dubinin–Radushkevich isotherm:** The Linear form of Dubinin-Radushkevich isotherm [22] is

$$\ln q_e = \ln q_D - B^{-2}$$

Where,  $q_D$  is the theoretical saturation capacity (mg/g)  $B$  is a constant related to the mean free energy of adsorption per mole of the adsorbate ( $\text{mol}^2/\text{J}^2$ ) and  $\ln q_e$  is polanyi potential which is related to the equilibrium as follows;

$$= RT \ln(1+1/C_e)$$

A plot of  $\ln q_e$  vs  $\ln(1+1/C_e)$  gives a linear trace and the constants  $q_D$  and  $B$  calculated from the slope and intercept respectively. The mean free energy of adsorption  $E$  calculated from  $B$  using the following equation

$$E = 1/(2B)^{1/2}$$

Based on this energy of activation we can predict whether an adsorption is physisorption or chemisorption. If the energy of activation is  $<8$  kJ/mol, the adsorption is physisorption and if the energy of activation is  $8-16$  kJ/mol, the



adsorption is chemisorption in nature. Based on the mean free energy calculated from the constant B (table 4), we can predict that physisorption dominates chemisorption.

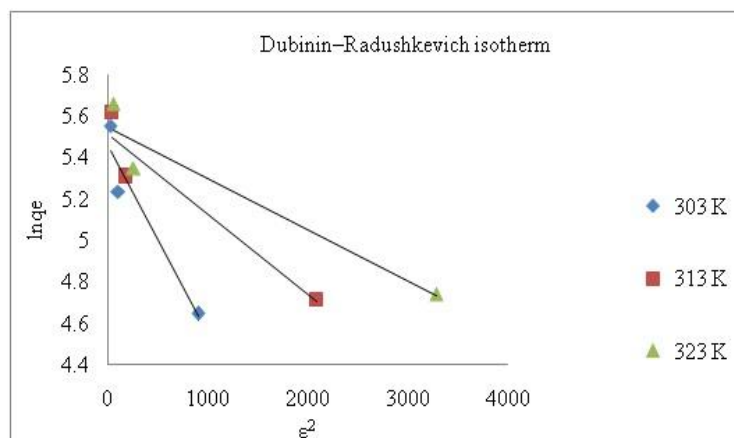


Figure 10: Dubinin-Radushkevich isotherm

Table 4: Results of various isotherms plots for the adsorption of BBR dye onto DRZAC

Isotherm Models	Temp (K)	Parameters and their results			
		$Q_0$ (mg/g)	B	$R_L$	$R^2$
Langmuir	303	11.76	21.26	0.0007	0.995
	313	21.28	11.75	0.0004	0.994
	323	28.57	8.75	0.0005	0.992
Freundlich		$1/n$	$k_f$ (mg/g <sup>-1</sup> )		$R^2$
	303	1.9	22.70		0.999
	313	2.22	32.66		0.999
	323	2.31	41.40		0.999
Tempkin		$B_1$ (J/Mol)	$K_T$ (L/g)		$R^2$
	303	28.67	0.3770		0.987
	313	32.53	0.7304		0.992
	323	33.56	0.3277		0.991
Dubinin Raduskevich		$q_D$ (mg/g)	E	$B \times 10^{-4}$ (mol <sup>2</sup> /J <sup>2</sup> )	$R^2$
	303	234.42	2.73	0.0007	0.925
	313	245.47	2.75	0.0003	0.924
	323	257.03	2.77	0.0002	0.920

## 6. Analysis of Isotherm

### 6.1 Langmuir isotherm:

In the present study  $Q_0$  value ranges from 11.76 to 28.57, as the temperature increases the monolayer adsorption capacity also found to increase. This kind of results were obtained in various similar studies [16]. The separation factor  $R_L$  values in between 0 to 1 indicates the favourable adsorption.  $R^2$  values of isotherm ranged between 0.992 to 0.995.

### 6.2 Freundlich isotherm:

The values of  $n$  were between 1 and 10 which indicates cooperative adsorption [23]. The  $R^2$  values were close to unity which reached to good fitting into Freundlich isotherm.

### 6.3 Temkin Isotherm:

$B_1$ -Temkin constant is related to the heat of adsorption. This  $B_1$  value increased from 28.67 to 33.56 as the temperature of adsorption increased. The Temkin parameter  $K_T$  value gives an idea about the nature of adsorption [24]. In our present study the  $K_T$  values ranged from 0.3277 to 0.7304 which indicate the adsorption is physical nature. The  $R^2$  value was low compared to Langmuir and Freundlich isotherm.

### 6.4 Dubinin-Raduskevich:

The activation energy  $E$  value ranges from 2.73 to 2.77 and  $B$  value from 0.0002 to 0.0007 indicates the physisorption. The  $R^2$  value was very low when compared to other three isotherms. In general the fitting data in isotherm equation were in the following order: Freundlich > Langmuir > Temkin > Dubinin-Raduskevich.

## 7. Conclusion

This study shows that DRZAC can be used effectively for the removal of BBR dye from aqueous solution. The adsorption of BBR dye onto DRZAC increased with the increasing of initial concentration of dye. The adsorption data fitted with pseudo-second order kinetic model. The adsorption equilibrium data well described by the Freundlich isotherm when compared to other three isotherm such as Langmuir, Temkin and Dubinin–Radushkevich studied constant predicted that the adsorption was favorable adsorption and physical nature.

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