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RESEARCH PAPER

Thermodynamics and adsorption studies of rhodamine-b dye onto organoclay

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Abstract

Thermodynamics and adsorption studies were conducted with a dye of Rhoda mine-B (RB) on organoclay (OC). Adsorption of the dye was investigated with an initial dye concentration at pH 7±0.3, 298, 308 and 318 K. The adsorption experiments were carried out isothermally at three different temperatures. The Langmuir and Freundlich isotherm models were used to describe the equilibrium data and the results were discussed in details. The thermodynamic parameters such as standard free energy (ΔG°), entropy change (ΔS°) and enthalpy (ΔH°) were calculated for OC. These values showed that adsorption of RB on OC was a spontaneous and endothermic process.

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Introduction

Dyes are organic compounds consisting of two main groups of compounds, chromophores (responsible for color of the dye) and auxochromes (responsible for intensity of the color) (Christie, 2001). Dyes usually have synthetic origin and complex aromatic molecular structures which make them more stable and more difficult to biodegrade (Sayan, 2006). It is estimated that 10 -15% of the dye is lost in the effluent during the dyeing process (Garg, *et al*, 2003; Young, L. *et al*, 1997). Removal of synthetic dyes from wastewater before discharging to environment and from raw wastewater before offering it to public use is essential for the protection of health and environment (Numan *et al*, 2006). Most of the dyes are toxic and carcinogenic compounds; they are also recalcitrant and thus stable in the receiving environment, posing a serious threat to human/animal health which is not only limited to themselves but may be passed onto further generations by the way of genetic mutations, birth defects, inherited diseases and so on (Crini, 2008).

Many treatment processes such as photo catalytic degradation (Sohrabi *et al*, 2008; Sleiman *et al*, 2007), electrochemical degradation (Fan *et al*, 2008), cation exchange membranes (Wu *et al*, 2008), micellar enhanced ultra- filtration (Zaghbani *et al*, 2008), adsorption/precipitation processes (Zaghbani *et al*, 2007), Fenton-biological treatment (Lodha *et al*, 2007) have been applied for the removal of dyes from wastewater. Adsorption has proven to be more versatile and efficient compared to conventional physico-chemical methods of dye removal (Senthilkumar *et al*, 2006). The utilization of waste materials is increasingly becoming of vital concern because these wastes represent unused resources and, in many cases, present serious disposal problems. During the past decade, a great deal of attention has been given to methods of converting these materials into useful products (Jain *et al*, 2003). Consequently, many investigators have studied the feasibility of using low cost sorbents such as: coconut husk (Tan *et al*, 2008; Tamai *et al*, 2008), wheat bran (Ozer *et al*, 2007), vetiver roots (Altenor *et al*, 2009), cottons talk and its hull (Tunc *et al*, 2009) date stones (Girgis *et al*, 2002; Haimour *et al*, 2006), pistachio nut shell (Yang *et al*, 2006), apple prom ace and wheat straw (Chandran *et al* 2002).

The aim of the present study is to explore the capability of organically to remove basic dye, Rhoda mine B, from aqueous solution under different experimental conditions such as contact time, adsorbent dosage, pH, initial dye concentration, temperature, and agitation speed. The adsorption, equilibrium isotherm models and thermodynamic parameters were also evaluated.

Material and methods

Preparation of Adsorbent

Organically was prepared via cationic surfactants modification of sodium-montmorillonite by reacting Na-MMT with solution containing the, cationic surfactants, cetyltrimethylammonium bromide (CTMAB). A typical experimental procedure is as follows: 10.0 g of the Na-MMT is dispensed in 0.5 L of distilled water. Then a certain amount of CTMAB is added and the suspension is continuously stirred for 1 hr. After that, subjected to mechanical stirring for 4 hrs. in water bath at 80˚C. The final MMT precipitate was separated by centrifugation and washed with 50% (v/v) of water and ethanol mixture at 60˚ several times until no chloride ions were detected with 0.1 N Ag NO3 solution. The organophilic montmorillonite sample is dried at 80˚C, and mechanically ground with a mortar and pestle to less than 400 mesh to collect the Nan particles. All samples are identified by a prefix number that states the amount of the added surfactant expressed as the percent of the Na-MMT'S CEC followed by the abbreviation for the specific type of the surfactant. In this experiment, the amount of the cationic surfactant CTMAB is fixed at the 110% CEC of the Na-MMT. Therefore, the prefix number in the front of CTMAB is omitted.

Preparation of Adsorb ate Solutions

Rhoda mine-B (RB) as a dye was obtained from Koch-Light Laboratories Ltd, England, and used without further purification.

The structural form of Rhoda mine-B is given in Fig. 1. A RB has the maximum absorption wavelength at 554 nm. The maximum absorption wavelength of RB was determined by finding out absorbance at the characteristic wavelength using a double beam UV-visible spectrophotometer. Dye stock solution (200mg/L) was prepared by dissolving accurately the weighed quantity of the dye in distilled water. Experimental dye solution of different concentrations was prepared by diluting the stock solution with suitable volume of distilled water.

Adsorption Experiments

Adsorption of RB by OC was carried out by batch method and the influence of various parameters such as contact time (5-60 min), adsorbent dosage (1.0-5.0g/L), pH (2-10), initial dye concentration (5-20mg/L), temperature (25-45°C), and agitation speed (50-400rpm) were studied. Adsorption was carried out on RB by mixing 0.1 g of the OC samples with initial concentrations of 5, 10, 15, and 20mg/L into several 20mL bottles, each containing 20mL solution of RB dye. Then the bottles were stirred at 300 rpm for 60 min using a magnetic stirrer at $25^{o}C$ temperature. OC in the samples was separated by filtration and the concentrations of the dye at a given time (C_t) were determined in the supernatant solutions. Adsorption isotherms were determined by introducing 0.1g OC to respective 20mL of dye concentration 20mg/L at 298, 308, and 318°K. The pH of the solution was adjusted to the desired value by adding small amount of HCl or NaOH (0.1M).

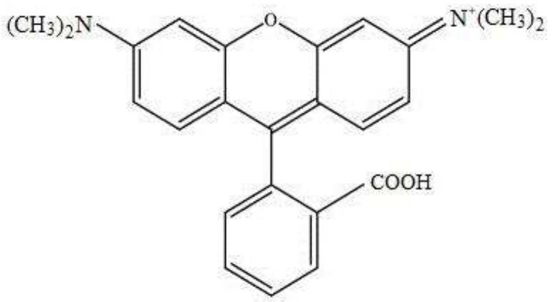

Fig. 1. Chemical structure of rhodamine-B.

The concentration of the dye in the aqueous solutions after adsorption was measured by using a UV-V is (Lambda 3b Shimadzu UV-V is spectrophotometer). The measurements of the pH values of the dye solutions were determined by using an Orion 410 pH meter (HANNA Instruments). The percentage of adsorption was estimated using following equation (XIANGHENG, *et al*, 2012):

$$\% \, Removal = \frac{Ci - Ct}{Ci} \, .100 \qquad (1)$$

Where, C_i and C_e are initial and equilibrium concentrations (mg/L), respectively.

D) Thermodynamic Parameters

The thermodynamic parameters such as standard Gibbs fee energy (ΔG°), entropy change (ΔS°) and enthalpy (ΔH°) were calculated using following equations (Laidler *et al*, 1999; Caliskan *et al*, 2011):

$$\text{kd} = \frac{Ci - Ct}{Ci} \, . \frac{V}{m} \qquad (2)$$

$$\ln \text{k}_d = \frac{\Delta S^\circ}{R} - . \frac{\Delta H^\circ}{RT} \qquad (3)$$

$$\Delta G^\circ = \Delta H^\circ - T \Delta S^\circ \qquad (4)$$

where K_d is the equilibrium constant, C_i initial concentration (mg/L), C_e equilibrium concentration (mg/L), V volume (L), m mass of the adsorbent (g), T (Kelvin), and R gas constant (8.314 J/mol). The changes in enthalpy (ΔH°) and entropy (ΔS°) were determined from the slope and intercept of the plots of $\ln K_d$ versus 1/T. The Gibbs free energy (ΔG°) was calculated using Eq. (4).

Results and discussion

Effect of Initial Dye Concentration

The relative RB removal by the adsorbents as a function of RB concentration was studied (ranging from 5 to 20mg/L, at 298 K). Fig. 2. shows the equilibrium adsorption capacity increases with the increasing the initial RB concentration. The RB concentrations of 5, 10, 15 and 20mg/L increase the removal to 44.5, 72.1,80.1 and 84.5%, respectively. This increase in the proportion of adsorption may be probably due to the equilibrium shift during the adsorption process, and which may result from the increased number of ions competing for the available binding sites on the surface of organoclay.

Adsorption of the dye was a little increase for concentrations higher than 20mg/L. It indicates that the saturation of adsorption sites was achieved. It can be seen that the rate of adsorption decreases with time and gradually reaches equilibrium.

In Fig. 2, where measurements were undertaken over a period of 5-60 min in all cases, the contact time of 60 min was sufficient to ensure, that the adsorption equilibrium was attained.

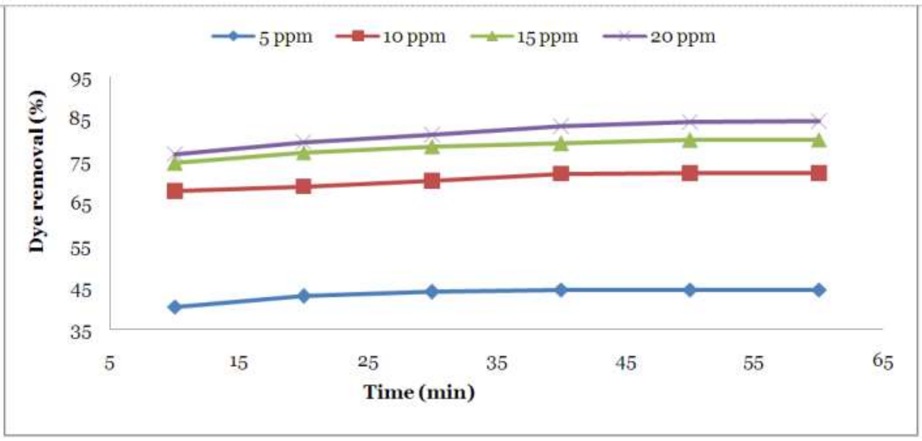

Fig. 2. Effect of contact time on adsorption of RB by OC at different initial dye concentration (adsorbent dosage = 5g/L; agitation speed = 300 rpm; temperature=25°C).

Effect of Temperature

The temperature has two major effects on the adsorption process. Higher temperature increases the rate of diffusion of the adsorb ate molecules across the external boundary layer and in the internal pores of the adsorbent particle, owing to decrease in the viscosity of the solution. In addition, temperature changing will change the equilibrium capacity of the adsorbent for the particular adsorb ate.

In this case of study, a series of experiments was conducted at 298, 308 and 318°K to study the effect of temperature on the adsorption rate. Adsorption of RB onto organoclay at 298, 308, and 318°K is shown in Fig. 3. The results indicate that adsorption increases with the temperature. It indicates the endothermic nature of the adsorption process. Similar results were also reported for RB adsorption onto acid-heat activated recto rite (Xiangheng *et al*, 2012).

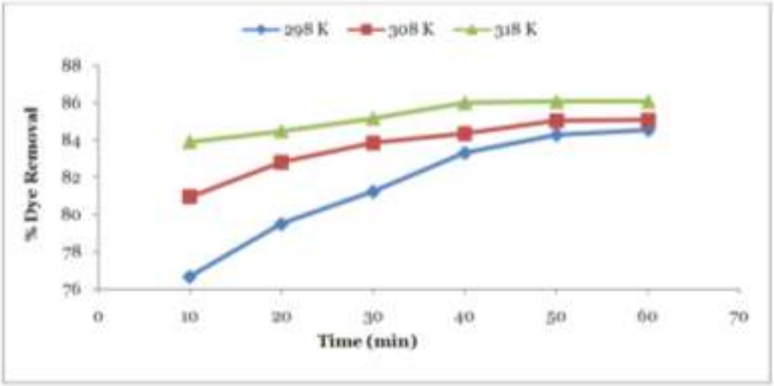

Fig. 3. Effect of temperature on RB adsorption by OC
(C = 20 mg/L; adsorbent dosage = 5g/L; agitation speed=300 rpm).

Effect of PH on the Adsorption of Rhodamine-B

The pH value of the solution is an important controlling parameter in the adsorption process.

Fig. 4 shows the concentration variation of waste at different pH. As can be seen in Fig. the adsorption is higher at acidic pHs and it is maximum at pH=4.

By increasing the pH the adsorption is decreased which can be because of negatively charged surface of adsorbent or functional groups on dyes. Also, the adsorption capacity of RB onto organoclay increases slightly (significantly) at low pH value. This result indicates that both organoclay and Rhoda mine B can be recovered by simply washing with acid solution (Ho *et al*, 2003).

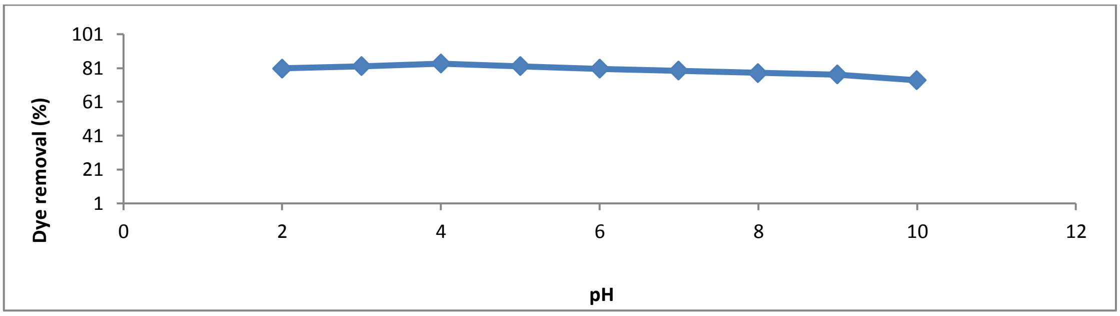

Fig. 4. Effect of pH on RB adsorption by OC (C = 20 mg/L; adsorbent dosage = 5 g/L; agitation speed=300 rpm)

Effect of Agitation Speed

Agitation speed is an important parameter in sorption phenomena, which has a serious action on the distribution of the solute in the bulk solution and the formation of the external boundary film. The effect of agitation on the uptake of RB by OC was studied at different agitation speeds (50-400rpm). From Fig. 5, it can be observed that agitation speed significantly.

Affects the adsorption of RB, thus confirming that the influence of external diffusion on the sorption kinetic control plays a significant role.

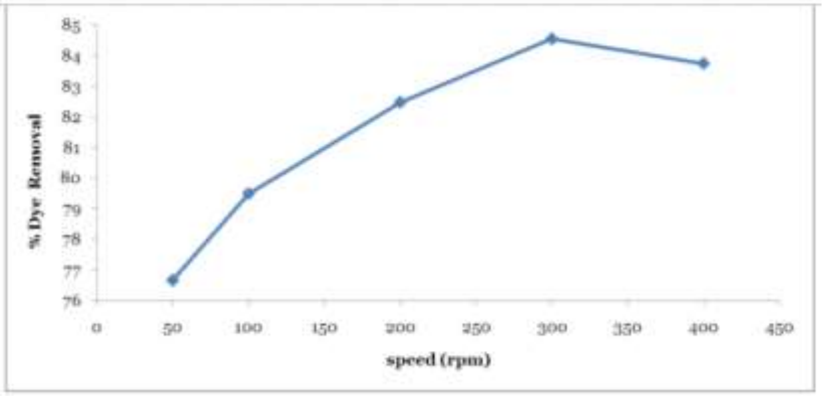

Fig. 5. Effect of agitation speed on adsorption of RB onto OC
(C = 20mg/L; contact time = 60 min; temperature = 25°C; adsorbent dosage = 5 g/L).

The percent of dye adsorbed was found to increase from 76.6 to 84.5% with increased in agitation speed from 50 rpm to 300 rpm, thus confirming that the influence of external diffusion on the sorption kinetic control plays a significant role.

With increasing the agitation speed, the rate of diffusion of dye molecules from bulk liquid to the liquid boundary layer surrounding the particle becomes higher because of an enhancement of turbulence and a decrease of thickness of the liquid boundary layer (Mall *et al*, 2005).

Also it is clear that while increasing the speed from 300 to 400 rpm, the percent removal of dye was decreased to 83.7%. This decrease in percent removal may be attributed to an increase desorption tendency of dye molecules and/or having similar speed of adsorbent particles and adsorbate ions.

This desorption tendency may be attributed to high mixing speed which means more energy input and higher shear force causing break of bonds between RB and the adsorbent (Abd El-Latif *et al*, 2010).

This also indicates that a 300 rpm agitation speed is sufficient to assure that all the surface binding sites are made readily available for dye uptake.

Adsorption Isotherms

A) Langmuir Isotherm

In the solid/liquid adsorption process, adsorption of the solute is usually characterized by either mass transfer (boundary layer diffusion) or intraparticle diffusion or even both (Ghosh *et al*, 2002).

The adsorption data of RB removal with OC was analyzed by the Freundlich and Langmuir isotherm models. The Langmuir isotherm model is valid for monolayer adsorption. The linear equation of the Langmuir isotherm is (Dogan *et al*, 2006):

$C_e/q_e= 1/bq_m + 1/q_m \, Ce$ (5)

Where, C_e is the equilibrium concentration of RB in the solution, q_e amount of RB adsorbed at equilibrium, q_m Langmuir adsorption capacity, and b Langmuir constant. Fig. 6. shows the relationship between C_e/q_e and C_e for the adsorption of RB onto the OC at 298, 308, and 318°K. The slope calculation and intercept of the linear plots give the values of q_m and b. The values of the Langmuir constants and coefficient determination R^2 are given in Table 2. The Langmuir adsorption capacity (q_m) was found to be 6.2, 11, and 17.9mg/g at different temperatures (298, 308 and 318°K). The essential characteristic of the Langmuir isotherm can be expressed by the equilibrium parameter and dimensionless constant (R_L) using equation:

$R_L = 1/1+bC_O$ (6)

Where, R_L can be $0< R_L< 1$.

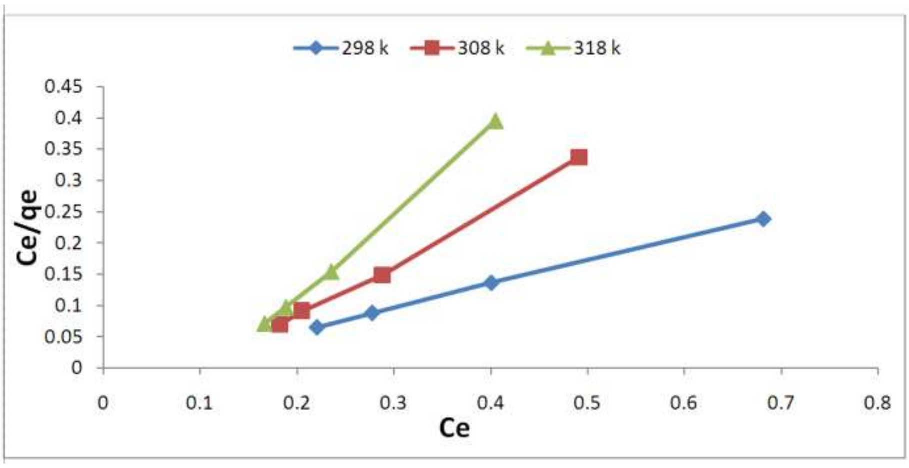

Fig. 6. Langmuir adsorption isotherm plots for adsorption of RB onto OC at different temperatures.

Table 1. Isotherm parameters for adsorption of RB onto OC.

Model	Isotherm constants	298 K	308 K	318 K
Langmuir	q_m	6.2	11	17.9
	b	23.6	9.5	8.5
	R_L	0.01	0.03	0.04
	R^2	0.999	0.996	0.998
Freundlich	K_f	0.42	0.31	0.19
	n	1.2	4.3	5.7
	R^2	0.9978	0.9971	0.9952

The dimensionless separation factor (R_L) was found to be in the range of 0.0 to 1.0 indicating that adsorption of RB is favorable for OC (Rahchamani *et al*, 2011).

B) Freundlich Isotherm

The Freundlich isotherm assumes an empirical equation based on the heterogeneous surface of adsorbent. The linear form of the Freundlich isotherm is expressed as (Khan, *et al*, 2011):

$\log qe = \log Kf + n \log Ce$ (7)

Where Kf is the Freundlich coefficient related to adsorption capacity, and n relates to adsorption intensity.

The values of the Freundlich constants were obtained from the linear correlations between the values of log q_e and log C_e as shown in Fig. 7. The values of K_f, n and coefficient determination R^2 are collected in Table 1.

In the Freundlich adsorption constant, n should be between 1 and 10 for beneficial. Adsorption (Vasu, 2008). Table 1 show that n values are in the range of 1 to 10 for OC and R^2 is 0.99. It means that adsorption of RB on OC can be described by the Freundlich model.

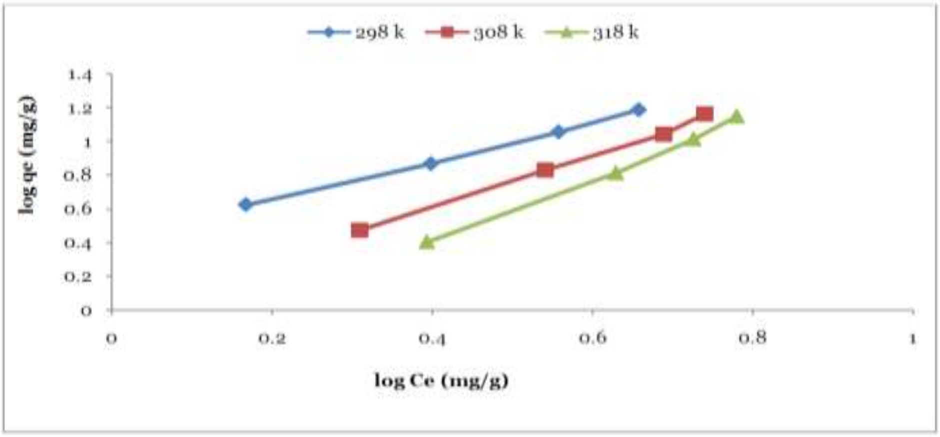

Fig. 7. Freundlich adsorption isotherm plots for adsorption of RB onto OC at different temperatures.

Thermodynamic Parameters of Adsorption

The thermodynamic parameters (ΔG°, ΔH° and ΔS°) are presented in Table 2. The Gibbs free energy is calculated for adsorption of RB on OC using Eq. (4). The values of ΔH° and ΔS° were determined from the slope and intercept from the plot of 1/T versus $\ln K_d$ given in Eq. (3), respectively. Fig. 8 illustrates Von't Hoff plot of effect of temperature on adsorption of RB on OC. The ΔG° value is negative for RB on OC and it indicates that adsorption is spontaneous.

The ΔG° decreases with temperature. Furthermore, better adsorption is obtained at higher temperatures (Khan, *et al*, 2012). The positive values of the enthalpy change (ΔH°) indicates that the adsorption process is endothermic. When ΔH° is more than 40 kJ/mol the type of adsorption can be accepted to be a chemical process. It indicates that adsorption is chemical by nature and involves strong forces of attraction (Rahchamani, *et al*, 2011). The positive value of ΔS° shows the increasing randomness at the solid/solution interface during adsorption of RB on the adsorbents. The positive mean values of ΔS° may be due to some structural changes in the adsorb ate and adsorbents during the adsorption process from the aqueous solution (Unuabonah, *et al*, 2007).

Table 2. Thermodynamic parameters for the adsorption of RB onto OC.

Initial concentration (mg/L)	ΔH° (KJ/mol)	ΔS° (KJ/molk)	ΔG°(KJ/mol)		
			298 K	308 K	318 K
5	218.6	0.12	-1.7	-3.5	-5.3
10	313.2	0.26	-3.8	-5.2	-8.6
20	482.5	0.32	-4.8	-6.04	-10.27

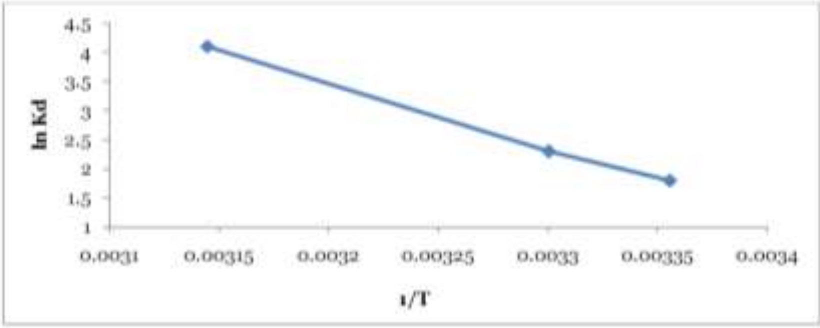

Fig. 8. Von't Hoff plot for effect of temperature on adsorption of RB onto OC.

Conclusion

The adsorption of RB onto OC was proven to be an efficient process. The amount of dye adsorbed was found to vary with contact time, adsorbent dosage, pH, initial concentration, temperature and agitation speed. The maximum uptake of dye by OC occurred at a pH of 4. The equilibrium adsorption was attained after 60 min. The Langmuir and Freundlish isotherm models were used for the mathematical description of the adsorption equilibrium of RB onto OC. The thermodynamic parameters ΔG (J/mole), ΔH (J/mole) and ΔS (J/K/mole) indicated the endothermic nature of adsorption and the increased randomness at the solid-solution interface during adsorption respectively. Results obtained from this study indicated that OC could be used as a potential adsorbent for the removal of RB from the aqueous solution in a batch system. Since clay are freely, abundantly and locally available, it can be used as an economical sorbent for the real industrial effluent.

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