ADSORPTION OF REACTIVE DYE BY A NOVEL ACTIVATED CARBON PREPARED FROM THEVETIA PERUVIANA

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ABSTRACT

This investigation was to evaluate the adsorption capability of activated carbon prepared from *Thevetia Peruviana* wood for removal of reactive textile dye (RO4) from aqueous solution. Batch adsorption experiments were performed as a function of initial dye concentration, contact time, temperature and pH. The amount of dye adsorbed increased while increasing the initial concentration and temperature of the solution. The experimental data fitted well to Pseudo second order kinetic model with Intra particle diffusion as the rate determining step. Adsorption data fitted with Langmuir model. The negative value of Gibb's free energy ($\Delta G°$) indicated that the adsorption was spontaneous in nature. The results showed that the activated carbon of *Thevetia Peruviana* was suitable for the removal of the reactive dyes from the aqueous solutions. The positive value of enthalpy ($\Delta H°$) indicated that the adsorption of the selected dyes were endothermic.

Keywords*:* Activated Carbon, Adsorption, Dyes, Reactive Orange4.

INTRODUCTION

Dyes and pigments are extensively used in paper, textile, food, dye-houses and printing to color the final products and they generated wastewater. Dyes are synthetic aromatic water-soluble dispersible organic colorants, having potential application in industries. Recently, it was estimated about 10,000 of different commercial dyes and pigments exist and over $7x 10^5$ metric tones are produced annually worldwide[1]. Presence of colour in the wastewater is one of the main problems in textile industries. From the environmental point of view, the removal of synthetic dyes is of great concern, because some dyes and their degradation products are carcinogenic and toxic[2].

In general, reactive dyes are the most problematic among other dyes, as they can not be removed by the conventional treatment systems[3]. Removing colour from waste water can be done via several methods namely chemical, biological and physical methods. Chemical methods use coagulation or flocculation combined with floatation and filtration, precipitation –flocculation, electro-floatation, electro kinetic coagulation and ozonisation to remove colour. Biological treatment utilizes fungi, bacteria or other biomass (either dead or alive) and is widely accepted due to its economical advantage. Physical methods often applied are membrane filtration and adsorption techniques.

Among these methods, adsorption using activated carbon as adsorbent has been found to be superior due to low cost, simplicity of design, flexibility, ease of operation and insensitivity to toxic pollutants[4]. Since commercially available activated carbon is very expensive, now the research is focused on the use of low cost adsorbents derived from agricultural and wood wastes. A number of agricultural waste and by-products of cellulosic origin have been analyzed for their capacity to remove dyes from waste water, such as coir pith[5], maize bran[6], rice husk[7], orange peel[8], lemon peel[9], saw dust[10], barley straw[11], egg shell[12], sunflower stalks [13]and pea nut hulls[14].

In the present work, the adsorption of Reactive Orange4 onto activated carbon prepared from *Thevetia Peruviana* wood by carbonization with Phosphoric acid (TPAC) was investigated. The thermodynamic and kinetic parameters such as free energy changes (ΔG), enthalpy changes (ΔH), entropy changes (ΔS), pseudo first order rate constant (k_1) and second order constant (k_2) were calculated for the adsorption of Reactive Orange 4.

MATERIALS AND METHODS

Preparation of adsorbent

Thevetia Peruviana wood was used as precursor for the preparation of activated carbon. The wood cut into pieces of 2 cm to 3 cm size, dried in sunlight for 10 days. The dried material soaked in a boiling solution of 40 % H_3PO_4 for one hour and kept at room temperature for 24 hours. After 24 hours the wood material separated, air dried and carbonized in muffle furnace at 400°C. The carbonized material was powdered and activated in a muffle furnace at 800°C for a period of 10 minutes. Then the material was washed with plenty of water to remove residual acid, dried, sieved to a desired particle size and stored in a tight lid container for further adsorption studies. The characteristics of the activated carbon are studied as per the standard procedures [15,16] and given in Table 1.

Preparation of adsorbate

The dye used in this study was Reactive Orange4 having molecular formula $C_{24}H_{13}Cl_2N_6O_{10}S_3Na_3$ (Mol.Wt:781.46) with CI No. 18260. All the chemicals were reagent grade. The structure of the dye is given in Fig. 1. The stock solution of 1000mg/L of the RO4 dye was prepared using double distilled water. The experimental solutions were obtained by diluting the dye stock solutions in accurate proportions to different initial concentrations.
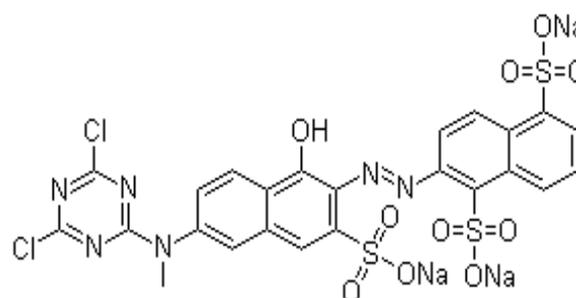

Fig. 1: Structure of Reactive Orange 4

Batch mode adsorption studies

The adsorption experiments were carried out by agitating 100 mg adsorbent with 200 mL of dye solutions of 25 to 100 mg/L concentration at 150rpm on an Orbital shaker (Universal make). The mixture was withdrawn at specified intervals, centrifuged using electrical centrifuge (Universal make) at 5000rpm for 20 minutes and unadsorbed supernatant liquid was analyzed for the residual dye concentration using Elico make UV Spectrophotometer (CI 73) at 479 nm for RO4. The effect of pH was studied by adjusting the pH of the adsorptive solutions by using dilute HCl and NaOH solutions. The effect of temperature was studied at four different temperatures

(30, 35, 40 and 45°C). All experiments were carried out in duplicate and the mean values are reported, where the maximum deviation was within 4%.

The amount of dye adsorbed on TPAC adsorbent was calculated from the following equation

$$q_t = \frac{(C_0 - C_e)}{M} \qquad (1)$$

Where, q_t (mg/g) is the amount of dye adsorbed at time t, C_0 and C_e (mg/L) are the concentrations of dye at initial and equilibrium respectively. V (L) and W (g) are the volume of the solution and the mass of dry adsorbent used, respectively.

Desorption Studies

Desorption studies helps to elucidate the mechanism of adsorption and recovery of the adsorbate and adsorbent. The regeneration of the adsorbent may make the treatment process economical. After centrifugation, the supernatant was separated and the adsorbent was separated and allowed to agitate with 100 mL of distilled water at different pH (2 – 11) above the equilibrium time of adsorption. The desorbed dye solution was separated by centrifugation and estimated as given in the adsorption studies[17].

RESULTS AND DISCUSSION

Characterization studies

Characteristics of carbon prepared from Thevetia Peruviana are presented in Table 1. The surface morphology of TP carbon

visualized via Scanning Electron Microscope (SEM), (Make -Jeol, Model 6390LA) at 2000X magnification. Examination of SEM micrograph of the TPAC particles showed rough areas of the surface of the carbon. The characteristics (Table 1) showed that carbon had a high porosity and more bulk density.

Table 1: Physico – chemical characteristics of TPAC

S. No.	Properties	Values
1.	pH	6.68
2.	Moisture Content,%	12.2
3.	Bulk density, g/mL	0.41
4.	Porosity,%	55.43
5.	Methylene Blue Value , mg/g	420
6.	Iodine Number, mg/g	825
7.	Surface Area,m^2 /g	862.394

Effect of agitation time and initial dye concentration

The variation in percentage removal of dye RO4 with contact time at different time initial dye concentrations ranging from 25 to 100mg/L at 30° C is shown in Fig. 2. It was observed that the maximum amount of dye adsorption taking place within the contact time of 30 min and it reached equilibrium at 90 minutes. After that no significant change was observed in the extent of adsorption. The percentage of dye removal decreased from 94.44 % to 79.49 % for RO4 while increasing the initial dye concentrations from 25 to 100 mg/L. This is due to the fact that with increase in dye concentration, there will be increased competition for the active adsorption sites and the adsorption process will increasingly slow down.
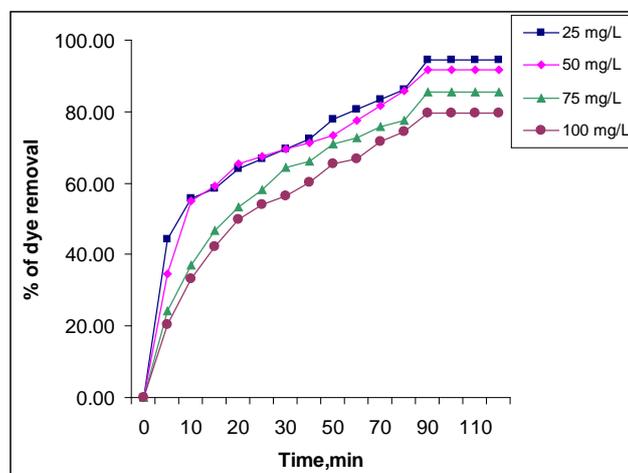

Fig. 2: Effect of agitation time on the adsorption of RO4 on to TPAC at 30° C

The adsorption curves are single, smooth and continuous till the saturation of dye on the carbon surface. The adsorption capacity at equilibrium increased from 47.22 mg/g to 158.97 mg/g for RO4 with an increase in the initial concentrations from 25 to 100mg/L.This is due to the increase in availability of the dye molecules near adsorbent. Similar behaviors were reported for the adsorption of Reactive Red 120 by palm-oil industry waste [18]and the adsorption of Reactive Orange onto activated carbons prepared from sugarcane bagasse pith[19].

Effect of pH

The percentage of dye adsorption decreases with increase in pH from 2 to 5 and remains constant there after. In the acidic range 1 – 3, the positive surface charge of adsorbent increases and this would attract the negatively charged functional groups on the reactive dyes. When the pH is increased, the number of negatively charged sites increases and there will be competition between the negatively

charged hydroxyl ions and anionic dye for the sorption sites and the adsorption rate get decreased.

Effect of temperature

To observe the effect of temperature on the percentage of dye removal, experiments were carried out for four different temperatures (30, 35, 40 and 45°C) at an initial dye concentration of 50 mg/L and the normal pH of dye solution (6.5). Fig. 3 showed that the percentage removal of RO4 by TPAC increases from 91.84 % to 97.96% while increasing the temperature. This indicates that the sorption of RO4 on to TPAC is endothermic in nature.

Adsorption kinetics

In order to optimize the design of an adsorption to remove the dye, it is important to establish the most appropriate correlations for the equilibrium data for each system. In this present study, the following four kinetic models were applied for the experimental data.

Pseudo first –order kinetic model assumes that the rate of change of solute uptake with time is directly proportional to difference in saturation concentration and the amount of solid uptake with time. It was proposed by Lagergren[20]. The rate constant of adsorption is expressed as a first – order rate expression given as:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \tag{2}$$

Where q_t and q_e are the amount of dye adsorbed (mg/g) at contact time t (min) and at equilibrium, and k_1 is the pseudo-first-order rate constant (min[-1]). After integrating with the boundary conditions at initial time (t = 0), q_t = 0 and at any time (t >0), amount of dye adsorbed is q_t and rearranging Eq. (2), the rate law for a pseudo-first-order reaction becomes:

$$\log (q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \tag{3}$$

The plot of log (q_e-q_t) versus t should give a straight line with slope of –k_1/2.303 and intercept log q_e which allows calculation of adsorption rate constant k_1 and equilibrium adsorption capacity q_e. It was observed that the experimental data point does not fit a straight line. Calculated values of k_1 and q_e are summarized in Table 2. The correlation coefficient r^2 are found to range from 0.9109 to 0.9755 .The pseudo first-order kinetic model of Lagergren does not fit well with the experimental data over the whole range of initial concentrations studied. Hence the adsorption of both RO4 onto TPAC does not follow the pseudo first-order model.

The pseudo second-order kinetic equation is expressed as

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{4}$$

The initial adsorption rate, h, (mg/g min) is expressed as h = $k_2 q_e^2$ (5)

Where, k_2 is the rate constant of pseudo second –order adsorption (g/mg min) and q_e is the equilibrium adsorption capacity (mg/g). Fig. 4 shows the pseudo second -order plots for the adsorption of RO4 by TPAC at various initial dye concentrations.

When the pseudo second order kinetic is applicable, the plot of t/q_t against t should give a linear relationship from which k_2 and q_e can be determined respectively from the intercept and slope of the plot. The correlation coefficients are higher than pseudo first order model. Further, the rate constant, k_2 decreases with increase in initial dye concentration.

It can be shown from Table 2 that the adsorption of RO4 dye onto TPAC fitted well to the pseudo second order kinetic model than the pseudo first order kinetic model.
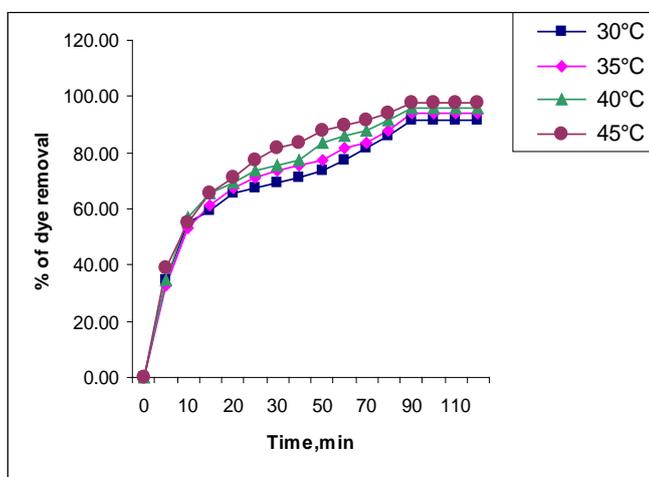

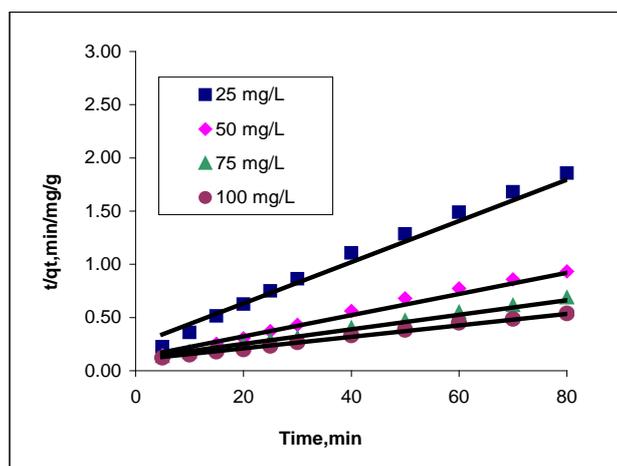**Fig. 3: Effect of temperature on the adsorption of RO4 on to TPAC**

Fig. 4: Pseudo second order plot for the adsorption of RO4 on to TPAC at 30° C

Adsorption is a multi step process involving transport of the solute molecules from the aqueous phase to the surface of the solid particulates followed by diffusion into the interior of the pores. Assuming that the rate is controlled by pore and intra particle diffusion, the amount adsorbed (q_t) is proportional to the $t^{1/2}$

$$q_t = k_{id} t^{1/2} + I \quad (6)$$

Where q_t is the amount of dye adsorbed (mg/g) at time t (min) and I is the intercept (mg/g). k_{id} and I values were obtained from the slopes and intercepts of the linear plot. Fig. 5 represents the plot of

q_t versus $t^{1/2}$ for adsorption of RO4 onto TPAC for the initial dye concentration of 25 to 100mg/L.

It seems that the plots are non linear for all the concentrations but careful observations shows the data points can be represented by double linear. In first straight line, the sudden increase in slope signifies that the dye molecules are transported to the external surface of the adsorbent through film diffusion. Intra -particle diffusion of dye molecules through pore is represented by second straight line. The linear portion does not pass through the origin which concludes that the pore diffusion is the only rate determining step and not the film diffusion.

Table 2: Kinetic parameters for RO4 dye with different initial dye concentration and different temperatures onto TPAC

Parameter	Initial dye concentration, mg/L				Temperature, °C			
	25	50	75	100	30	40	45	50
$q_e$exp.(mg/g)	47.22	91.84	128.23	158.97	91.84	93.88	95.92	97.96
Pseudo first order kinetics								
$k_1 \times 10^{-2}$ (min^{-1})	2.49	2.65	2.76	3.06	2.65	2.79	3.22	3.52
$q_e$cal (mg/g)	29.17	57.61	93.78	127.61	57.61	58.61	61.87	62.31
r^2	0.9435	0.9109	0.9562	0.9755	0.9109	0.917	0.9426	0.9561
Pseudo second order kinetics								
$k_2 \times 10^{-4}$(g/mg min)	15.41	8.11	3.95	2.88	8.11	8.15	8.97	9.76
h	4.09	8.11	8.55	9.87	8.11	8.67	9.74	11.05
$q_e$cal(mg/g)	51.55	100.00	147.06	185.19	100.00	103.09	104.17	106.38
r^2	0.9915	0.9923	0.9961	0.9969	0.9923	0.9956	0.9977	0.9993
Intra particle diffusion model								
k_{id} (mg/g/min^{½})	3.5924	7.1017	11.093	14.08	7.1017	7.3774	7.4751	7.6267
r^2	0.8907	0.8768	0.9298	0.9394	0.8768	0.8687	0.8548	0.8424

The values of k_{id} and r^2 values are given in Table 2.
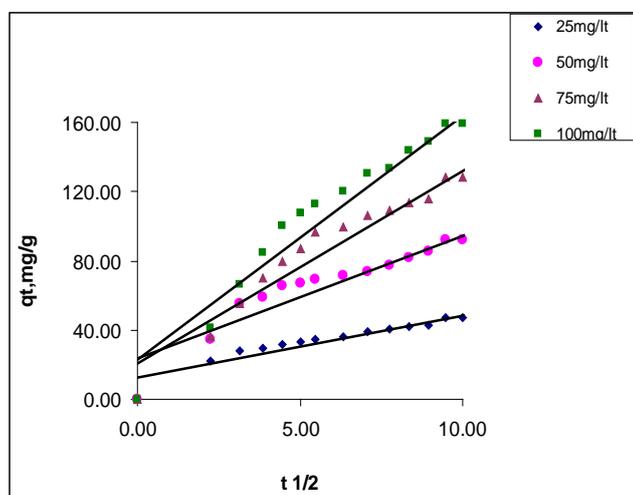

Fig. 5: Intra particle diffusion plot for the adsorption RO4 on to TPAC at 30° C

Adsorption isotherm

Adsorption isotherm indicates how the adsorption molecules distribute between the liquid phase and solid phase when the adsorption reaches an equilibrium state. Adsorption isotherms are important to describe the interaction of adsorbate molecules with adsorbent surface. In this study, Langmuir and Dubinin – Raduskevich isotherms were employed for the treatment of the equilibrium adsorption data. The applicability of the isotherm equation is compared by judging the correlation coefficients r^2.

The Langmuir adsorption isotherm is the best known linear model for monolayer adsorption on the homogeneous surface and most frequently utilized to determine the adsorption parameters. Langmuir model is represented by the following equations:

$$\frac{C_e}{q_e} = \frac{1}{Q_o b_L} + \left(\frac{1}{Q_o} \right) C_e \quad (7)$$

Where, q_e the amount is adsorbed at equilibrium (mg/g), Q_o is the monolayer adsorption capacity (mg/g), C_e is the equilibrium concentration of adsorbate (mg/l) and b_L is Langmuir constant related to energy of adsorption.

Fig. 6 shows a linearized plot of C_e/q_e against C_e. Values of Q_o and b_L were calculated and given in Table 3. The Langmuir adsorption capacities varies from 113.64 mg/g to 123.46 mg/g for RO4 with increase in temperature from 30º C to 45º C, indicating that the adsorption is favor to high operating temperature. Similar values were reported for the adsorption of reactive dye brilliant red by activated charcoal[22] .From the values it is concluded that the maximum adsorption corresponds to a saturated mono layer of dye molecules on the adsorbent surface with constant energy and there is no transmission of dye molecules on the adsorbent surface occurs. Further it confirms the endothermic nature of the processes involved in the system. The Langmuir isotherm fits quite well with the experimental data with good correlation coefficient which varies from 0.9854 to 0.9930 for RO4.

The separation factor R_L is calculated by the following equation to confirm the favorability of the adsorption process. $R_{L=}$ 1/ (1+ $k_L.C_0$)
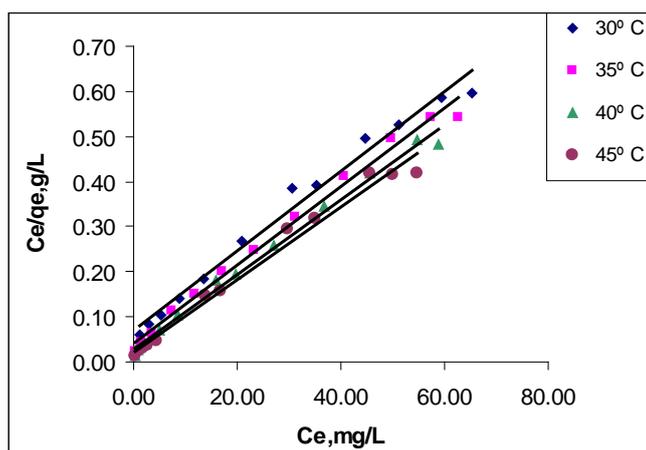

Fig. 6: Langmuir adsorption isotherm plot of adsorption of RO4 on to TPAC.

Where, k_L is the Langmuir constant and C_0 is the initial concentration of dye (mg/L). The values of R_L are found to be between 0 and 1 and confirm that the adsorption process is favorable.

The D-R isotherm describes the adsorption on a single uniform pore. Dubinin-Radushkevich isotherm is generally expressed as follows[25]:

$$q_e = q_D . e^{-B\varepsilon^2} \quad (8)$$

The linear form of D-R isotherm equation is represented as:

$$\ln q_e = \ln q_D - B\varepsilon^2 \quad (9)$$

$$\varepsilon = RT \ln(1 + \frac{1}{C_e}) \quad (10)$$

Where, q_D is the theoretical saturation capacity (mol/g), B is a constant related to the mean free energy of adsorption per mole of the adsorbate (mol²/ J²), ε is the Polanyi potential, C_e is the equilibirium concentration of adsorbate in solution (mol/L). The D-R constants q_D and B were calculated from the linear plots of ln q_e versus ε^2 and are given in Table 3. The constant B gives an idea about the mean free energy E (kJ/mol) of adsorption per molecule of the adsorbate when it is transferred to the surface of the solid from infinity in the solution and can be calculated from the following relationship [26]

$$E = 1/ (2B)^{1/2} \quad (11)$$

The adsorption is physisorption if the energy of activation is less than 8 kJ/ mol and chemisorption if the energy of activation is between 8 and 16 kJ/mol. From the Table 3, we can predict that the adsorption of RO4 by TPAC is physisorption in nature. D-R isotherm is not able to describe the experimental data properly because of the poor linear correlation coefficient which vary from 0.7520 to 0.7938.

Thermodynamics of adsorption

Thermodynamic parameters provide in-depth information of inherent energetic changes associated with adsorption; therefore, these parameters should be accurately evaluated. Langmuir isotherm equation was applied to calculate the thermodynamic parameters as follows:

$$\Delta G° = -RT \ln k_L \quad (12)$$

$$\ln k_L = \frac{\Delta S°}{R} - \frac{\Delta H°}{R} \frac{1}{T} \quad (13)$$

Where, k_L is the Langmuir equilibrium constant, $\Delta H°$ and $\Delta S°$ is the standard enthalpy and entropy changes of adsorption respectively.

Table 3: Isotherm Constants for the adsorption of RO4 on TPAC at various temperatures

Parameter	Temperature °C			
	30	35	40	45
Langmuir Isotherm				
Q_0 (mg/g)	113.64	114.94	120.48	123.46
b_L (L/mg)	0.0702	0.0411	0.0280	0.0215
r^2	0.9854	0.9918	0.993	0.9847
Dubinin-Raduskevich Isotherm				
q_D (mg/g)	79.7103	84.3015	90.5047	95.259
E (kJ/mol)	0.9129	1.581	2.236	2.357
r^2	0.7808	0.7520	0.7711	0.7938

Table 4: Thermodynamical Parameters for the adsorption of RO4 dye onto TPAC

Temperature, ºK	RO4		
	ΔHº, kJ/mol	ΔSº, kJ/K/mol	ΔGº, kJ/mol
303	63.1330	0.2312	-6.9206
308			-8.0766
313			-9.2326
318			-10.3886

Thermodynamic parameters like ΔH°, ΔS° and ΔG° were determined from the slope and intercept of Von't Hoff's plot of ln k_L versus 1/T. Table 4 shows the thermodynamic parameters ΔH°, ΔS° and ΔG° for the removal of RO4 by TPAC. The ΔG° values were negative at all tested temperatures (30 to 45°C), indicating that the adsorption of RO4 onto TPAC was spontaneous and thermodynamically favorable. The positive ΔH° values indicate that the adsorption of RO4 onto TPAC was an endothermic process, which was supported by the increase of adsorption of the dye with increase in temperature. Furthermore, the positive ΔS° indicates that the degrees of freedom increased at the solid – liquid interface during adsorption of of reactive dyes onto TPAC. Generally, ΔG° for physisorption is between -20 to 0 kJ/mol and for chemisorption is between -80 to -400 kJ/mol[27] .The ΔG° value ranges from -6.9206 kJ/mol to -10.3886 kJ/mol for RO4. This confirms that the adsorption of RO4 onto TPAC is physisorption in nature.

Desorption studies

The desorption of dyes by mineral acids and alkaline medium indicates the dyes are adsorbed onto the activated carbon by physisorption. Maximum desorption of 24.5% for RO4 observed at pH 8. There is no change in desorption above pH 8 for the dye.

CONCLUSION

In this investigation, low cost activated carbon can be prepared from the wood of Thevetia Peruviana for the adsorption of reactive dye (Reactive Orange 4) from aqueous solution. The Physico- chemical characteristics of the activated carbon are comparable with the commercial Activated Carbon. The adsorbed amount of RO4 increased from 47.22 mg/g to 158.97 mg/g while increasing the initial dye concentration. The adsorption of the dye increased with increase in temperature indicates that the adsorption is endothermic in nature. Kinetic studies showed that adsorption of these reactive dyes followed pseudo- second order model with multi step intra particle diffusion model. The data obtained from adsorption isotherms are well fitted with Langmuir model which suggests the monolayer coverage of the reactive dye on surfaces of TPAC. The negative ΔG° value confirms that the adsorption is spontaneous in nature. The Positive ΔH° value suggests that the adsorption is endothermic in nature.

ACKNOWLEDGEMENT

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