ORIGINAL PAPER

Adsorption of C.I. Acid Red 97 dye from aqueous solution onto walnut shell: kinetics, thermodynamics parameters, isotherms

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Abstract This study aims to investigate the adsorption of C.I. Acid Red 97 (AR97) onto walnut shell (WS) in order to find out the desired conditions. The effect of operational parameters including adsorbent dosage, agitation speed, contact time, pH and temperature was studied in a batch mode. The results revealed that 73.33 % of AR97 (50 mg/L) was decolorized at 60 min of contact time, agitation rate of 140 rpm and 12 g/L of adsorbent at 291 K. The rate of adsorption follows pseudo-second-order kinetics with the high correlation coefficient, which was more than 0.99. Specific surface area of WS was calculated as 1.28 m^2/g by BET method. It was observed from the values of thermodynamics parameters such as Gibbs free energy ($\Delta G°$), enthalpy ($\Delta H°$) and entropy ($\Delta S°$) that the nature of adsorption process is spontaneous, exothermic and mainly physical. Langmuir, Fruendlich and Redlich–Peterson isotherms were applied for mathematical description of equilibrium data and findings indicated that it is in good agreement with Fruendlich model.

Keywords C.I. Acid Red 97 · Adsorption · Walnut shell · Kinetics · Thermodynamics parameters · Isotherm

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Introduction

Dyes and pigments play the essential role in the textile industry, and nowadays, approximately 10,000 of dyes were used in textile industry, besides they are significant in the industrial chemistry (Behnajady et al. 2007; Daneshvar et al. 2004). Discharging of these dyes up to 10 %, during their production and consumption into water supplies, can create various problems. It can affect food and water cycles, and also cause allergic dermatitis and skin sensitivity. Moreover, these dyes can be toxic and hazardous and cause mutation for watery organisms (Doğan et al. 2009). Cationic (basic dyes), anionic (direct, acid and reactive dyes) and nonionic (disperse) are three types of dyes (Mall et al. 2006). Over 50 % of all textile dyes utilized in the industries are azo dyes (Behnajady et al. 2006). The chemical structure of these dyes can be defined as single or multiple units of azo ($-N=N-$) bonds connected to aryl or naphthalene groups, it creates chromophore feature in them (Ince and Tezcanli-Güyer 2004). The major problem for the industries and also for environment is treatment of these colored wastewaters (Behnajady et al. 2008).

In recent years, many methods including coagulation and flocculation (Furlan et al. 2010), reverse osmosis (Gupta et al. 1990), chemical oxidation (Neamtu et al. 2004), biological treatment (Ledakowicz et al. 2001), photodegradation (Behnajady et al. 2006) and adsorption (Rafatullah et al. 2010) have been developed for treating of dyes in wastewaters. Among these methods, adsorption has become proper method for treatment of aqueous effluent (Nandi et al. 2009a, b).

Activated carbon owing to its high surface area and high adsorption capacity is one of the common adsorbents. However, it is uneconomical for industrial application due

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to its high price; so, the process of dye removal through adsorption can be economically feasible by substituting low-cost adsorbents (Nandi et al. a, b; Demirbas 2009; Dural et al. 2010). For this reason, researches have focused on the usage of low-cost, locally available, reusable, biodegradable adsorbents made from natural sources like rice hull shell (Wang et al. 2010), wood apple shell (Jain and Jayaram 2010), fly ash (Sun et al. 2010) and powdered peanut hull (Gong et al. 2005). Natural and modified clays like sepiolite (Eren et al. 2010), zeolite (Mustafaa et al. 2010), perlite (Doǧan et al. 2004) and bentonite (Li et al. 2010) are being considered as alternative low-cost adsorbents (Rafatullah et al. 2010).

Adsorption experiments using walnut shell (WS) as an adsorbent have been reported on other dyes like malachite green (Dahri et al. 2014), reactive brilliant red K-2BP (Cao et al. 2014) and heavy metals such as Cr(VI) (Altun and Pehlivan 2012). But from the survey of the literature, no information for the adsorption removal of Acid Red 97 (AR97) by WS is available. The objective of this study was to investigate the effectiveness of WS for the removal of AR97 from aqueous solutions in a batch mode. Then, the effect of operational parameters including adsorbent dosage, agitation speed, contact time, pH and temperature was investigated on the adsorption process. Eventually, the applicability of known adsorption isotherms and kinetic models, and also thermodynamic parameters was studied.

Materials and methods

Materials

The azo acid dye, AR97 (C.I. 22890, FW: 698.63274 and λ_{max}: 498 nm) is a biorefractory contaminant but commonly used in the textile and tanning industries supplied by Aldrich, USA. Due to these characteristics, it was selected as a model adsorbate (Kayan et al. 2010). Its molecule exists as an anion in aqueous medium. The walnut shell as an adsorbent was provided from Mahabad region in Iran. It was ground up by hand mill and used after sieving as an adsorbent, and other chemicals were purchased from Merck Company in Germany.

Experimental methods

The experiments were performed in a batch mode, using AR97 solution with concentration of 50 mg/L. Stock solution of AR97 was prepared by dissolving proper amount of dye in distilled water. Experimental solution with the desired concentration was obtained by dilution. The experiments were carried out by feeding 50 mL of solution with known concentration of the dye into a 100 mL Erlenmeyer, and certain amount of the adsorbent was added and agitated for distinct time at known speed by shaker. At the end of the time, the sorbent was centrifuged and the adsorption of dye solution was determined by UV/Vis spectrophotometer. The dye removal efficiency can be calculated as follows:

$$\% \text{ dye removal} = \frac{(C_o - C)}{C_o} \times 100 \qquad (1)$$

where C is the concentration of dye at any time (t), and C_o is the initial dye concentration in the solution (mg/L). In order to study the sorption isotherms, the amount of adsorbed dye at equilibrium, q_e (mg/g), and at time t, q_t (mg/g), was calculated by:

$$q_e = \frac{(C_o - C_e)V}{W} \qquad (2)$$

$$q_t = \frac{(C_o - C_t)V}{W} \qquad (3)$$

where C_e is the equilibrium dye concentration (mg/L), W is the adsorbent mass (g) and V is the volume of the solution (L) (Mall et al. 2006).

Results and discussion

Characterization of WS

The nitrogen adsorption–desorption isotherm at 77 K onto WS is shown in Fig. 1, which is in correspondence with classical type III isotherm of IUPAC classification (Sing
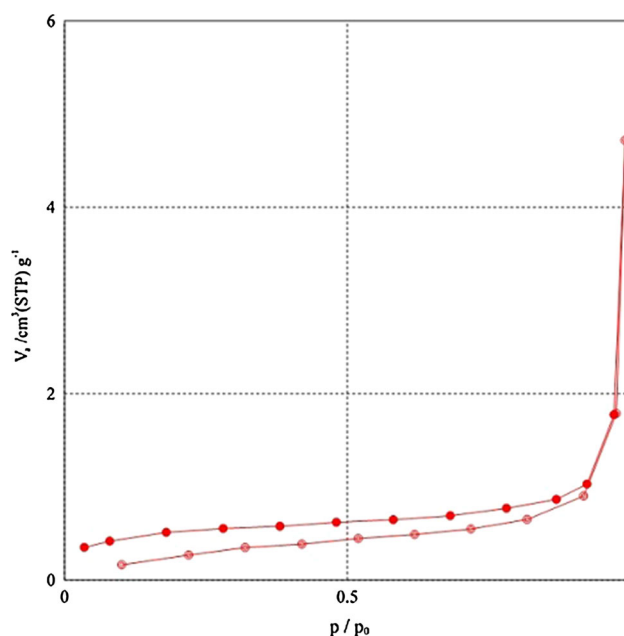

Fig. 1 Physisorption isotherm of WS

et al. 1985). According to the results of BET method, the average specific surface area and total pore volume of WS were 1.28 m^2/g and 6.89 × 10^{-3} cm^3/g, respectively. The adsorption capacity of WS depends on porosity and chemical reactivity of functional groups at the surface. Knowledge on surface functional groups would give insight into the adsorption capability of the WS (Senthil Kumar et al. 2010). The FT-IR analysis for WS showed broad band near 3,435 cm^{-1}, which indicates the presence of hydroxyl groups on the WS surface.

Effect of adsorbent dose

The adsorption of the AR79 dye onto the WS was studied by varying the dosage of the adsorbent (0.1–1.2 g) at dye concentration of 50 mg/L. The effect of WS dose on the adsorption of AR97 is shown Fig. 2. The removal percent enhanced with increasing WS amount up to a certain limit (0.6 g), and then, it remained approximately constant. This behavior can be attributed to availability of more adsorption sites and greater surface area (Nandi et al. 2009a, b).

Effect of agitation speed

Adsorption process strongly depends on agitation speed owing to the distribution of the solute in the solution and the formation of the external boundary film. Figure 3 demonstrates the removal percent of AR97 (50 mg/L) at different agitation speeds (80–180 rpm) within contact time of 1 h. From the figure, it is clear that with increasing agitation speed, the percentage removal of dye enhanced from 50.08 to 73.95 %. The increase in removal percentage can be described by the fact that increasing agitation speed reduces the film boundary layer and surrounding particles, therefore increasing the external film transfer coefficient (Nandi et al. 2009a, b).
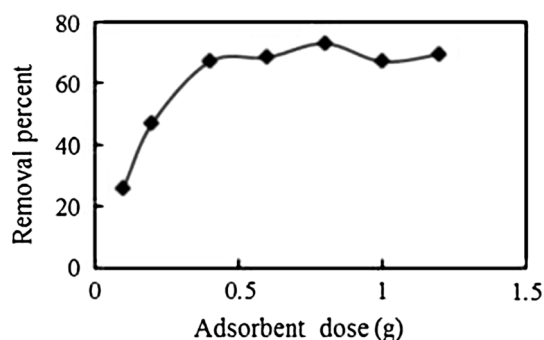

Fig. 2 Effect of adsorbent dose on the removal percentage of AR97. ([AR97] = 50 mg/L, T = 297 K, agitation speed= 120 rpm, pH= 5.45)
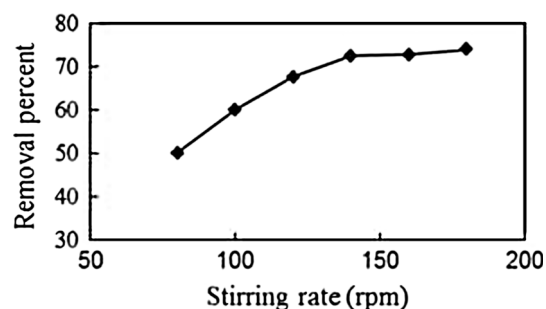

Fig. 3 Effect of agitation speed on the percentage removal of AR97 by WS. ([AR97] = 50 mg/L, T = 293 K, adsorbent dose = 12 g/L, pH= 5.45)
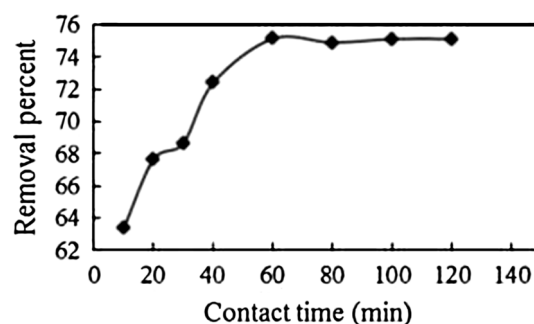

Fig. 4 Effect of contact time on the removal percentage of AR97 by WS. ([AR97] = 50 mg/L, T = 298 K, agitation speed = 140 rpm, adsorbent dose = 12 g/L, pH = 5.45)

Effect of contact time

The mixture of 0.6 g of the adsorbent and 50 mL of the dye solution (50 mg/L) was agitated at various times. It is obvious from Fig. 4 that the maximum amount of dye removal took place within the contact time of 60 min, and then, it remained almost constant due to the desorption process and the solution that has reached equilibrium (Govindasmay et al. 2009). The fast adsorption at the initial contact time can be explained by the electrostatic attraction, and the slow rate of dye adsorption after 60 min may occur as a result of the slow pore diffusion of the solute molecules into the bulk of the adsorbent (Khaled et al. 2009).

Point of zero charge measurements and Effect of pH

It is essential to realize the adsorption mechanism by determining the zero point charge (pH$_{ZPC}$) of the adsorbent (Govindasmay et al. 2009). The pH$_{ZPC}$ of the WS was measured by pH drift method. It was performed by adjusting the pH of a solution containing 0.01 M NaCl between 2 and 12 using NaOH and HCl, and then, 0.1 g of WS was added to 50 mL of the solution. The final pH was recorded after stabilizing the initial pH after 24 h. The
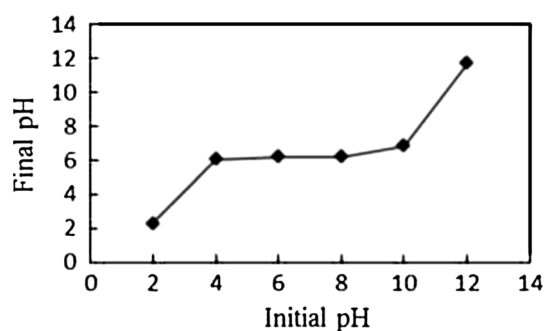
Fig. 5 Zero point charge (pH$_{ZPC}$) of the WS used for the adsorption experiments
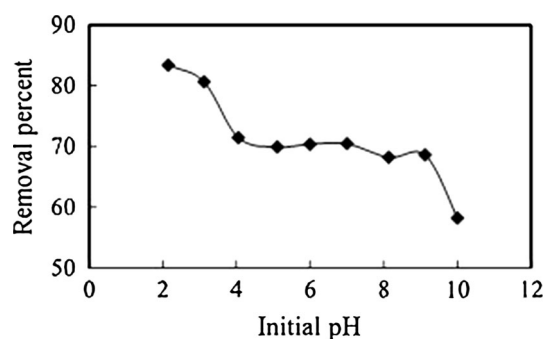

Fig. 6 Effect pH on the removal percentage of AR97 by WS. (contact time = 60 min, T = 292 K, agitation speed = 140 rpm, adsorbent dose = 12 g/L, pH = 5.45)

graph of final pH versus initial pH was used to identify pH$_{ZPC}$ that is the point in which both initial and final pH values are equal (Govindasmay et al. 2009). In this study, the pH$_{ZPC}$ of the WS was found to be 6.18 (Fig. 5).

The pH of dye solution is an effective parameter, which controls the adsorption particularly the adsorption capacity. Adsorption of anions is favored at lower pH (<pH$_{zpc}$), while the adsorption of cations is favored at a higher pH (>pH$_{zpc}$) (Govindasmay et al. 2009). The results indicated that acidic pH supported the adsorption of negatively charged AR97 (an anionic dye) on WS, where the surface change of WS was positive (Fig. 6).

Adsorption isotherm study

Adsorption isotherms are mathematical models that the equilibrium studies have an essential role in optimizing the design parameters for adsorption system. They provide adequate information on the physicochemical data of the adsorption process as a unit operation. In order to find a suitable model that can be utilized for design process, equilibrium data should appropriately fit into diverse isotherm models. The purpose of the adsorption isotherm

Table 1 The value of parameters for each isotherm models used in the studied

Langmuir nonlinear			
Equation	K_L (L/mg)	q_m (mg/g)	R^2
$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$	−0.00232	−29.154	0.744
Fruendlich nonlinear isotherm			
Equation	K_F (L/mg)	$1/n$	R^2
$q_e = K_F C_e^{1/n}$	0.0000023	5.1058	0.8312
Redlich–Peterson nonlinear isotherm			
Equation	A	B	g	R^2
$q_e = \frac{A C_e}{1 + B C_e^g}$	0.04	0.05	0.7	0.7818

[AR97] = 10–70 mg/L, contact time = 60 min, agitation speed = 140 rpm, adsorbent dose = 12 g/L, pH = 5.45

analysis is to relate adsorbate concentration in the solution and adsorbent surface (Khaled et al. 2009). The Langmuir, Fruendlich and Redlich–Peterson models are the most common types of isotherm equations, which were applied for analyzing experimental sorption data. The adequacy of model for predicting the experimental data was evaluated by correlation coefficient (R^2), and the results are given in Table 1.

Langmuir isotherm

The Langmuir isotherm assumes that the highest adsorption happens when a saturated monolayer of solute molecules exists on the adsorbent surface (homogenous system). The energy of adsorption is stable, and there is no migration of adsorbate molecules in the surface plane (Senthil Kumar et al. 2010). The equation of Langmuir isotherm is represented as follows:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \qquad (4)$$

where q_e is the equilibrium dye concentration on adsorbent (mg/g), q_m is the monolayer capacity of the adsorbent (mg/g), C_e is the equilibrium dye concentration in solution (mg/L), and K_L is the Langmuir adsorption constant representing the energy constant related to the heat of adsorption (Senthil Kumar et al. 2010). Correlation coefficient (R^2) of 0.744 (Table 1) indicates that this isotherm is not suitable for adsorption prediction.

Fruendlich isotherm

The Fruendlich isotherm model is an exponential equation that considering the adsorption on heterogeneous surface with interaction between adsorbed molecules and is not

Table 2 Different kinetic model parameter

Pseudo-first order		
k_1 (min^{-1})	q_e (mg/g)	R^2
0.1082	0.9005	0.9351

Pseudo-second order		
k_2 (g/mg min)	q_e (mg/g)	R^2
2.269	2.823	0.9994

Intra-particle diffusion		
$K_{i,1}$ (mg/g min$^{0.5}$)	I_1 (mg/g)able	R^2
0.0778	2.3995	0.9316
$K_{i,2}$ (mg/g min$^{0.5}$)	I_2 (mg/g)	R^2
0.0031	2.8563	0.7914

[AR97] = 50 mg/L, T = 298 K, agitation speed = 140 rpm, adsorbent dose = 12 g/L, pH = 5.45

restricted to the formation of a monolayer (Nandi et al. 2009a, b). This model is given as:

$$q_e = K_F C_e^{1/n} \qquad (5)$$

where n and K_F are Fruendlich constants related to adsorption intensity of the adsorbent and adsorption capacity, respectively (Li et al. 2010). The correlation coefficient (R^2) values demonstrate that the adsorption isotherm for the present system is explained better by Fruendlich model ($R^2 = 0.8312$) (Table 1).

Redlich–Peterson isotherm

Combination of Langmuir and Fruendlich models makes the Redlich–Peterson isotherm. It is in accordance with the Langmuir equation at low concentration and is close to the Fruendlich model at high concentration (Senthil Kumar et al. 2010). In addition, the Redlich–Peterson equation adds three parameters into an empirical isotherm and, consequently, can be utilized both in homogenous or in heterogeneous systems because of the high versatility of the equation (Mall et al. 2006). The equation is given as:

$$q_e = \frac{A\, C_e}{1 + B\, C_e^g} \qquad (6)$$

where A and B are Redlich–Peterson isotherm constants, and g is the exponent which lies between 0 and 1. It is obvious from Table 1 that this isotherm cannot be considered as favorable isotherm ($R^2 = 0.8718$).

Adsorption kinetics

In order to study the controlling mechanism of adsorption process, three kinetic models were investigated. Pseudo-

first-order kinetic model, pseudo-second-order kinetic model and intra-particle diffusion model were applied to find the best-fitted model for the experimental data, and the obtained results are presented in Table 2.

Pseudo-first-order kinetic model

This model is based on the assumption that the rate of solute uptake with time was directly correlative with difference in saturation concentration and the adsorbed amount. The first-order rate expression (linear form) is given as:

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

where k_1 is the pseudo-first-order rate constant (min^{-1}), q_t and q_e are the amount of dye adsorbed (mg/g) at contact time t (min) and at equilibrium. The plot of $\log(q_e - q_t)$ versus t revealed that the adsorption process is not following the pseudo-first-order kinetic model by correlation coefficient of 0.9351 (Table 2) (Rao et al. 2011).

Pseudo-second-order kinetic model

The linear form of the pseudo-second-order kinetic model is defined as:

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

where k_2 is the rate constant for pseudo-second-order sorption (g/mg min). The straight-line plot of t/q_t versus t has been used to determine rate parameters (Ghasemi et al. 2011). The results of Table 2 showed that the highest correlation coefficient ($R^2 = 0.9994$) among kinetic models is related to the pseudo-second-order kinetic model, and also, the calculated q_e value in this model was very close to what experimentally obtained q_e (2.882 mg/g). Thus, the adsorption of AR97 on WS was explained better by the pseudo-second-order kinetic model.

Intra-particle diffusion model

This model is based on the hypothesis that the mechanism for dye removal by adsorption on a sorbent material occurs through four steps: (1) bulk diffusion, which is the migration of dye molecules from bulk solution to the surface of the adsorbent; (2) film diffusion, which is diffusion of dye molecules through the boundary layer to the surface of the adsorbent; (3) dye adsorption on active site of the surface of adsorbent; and (4) intra-particle diffusion, which is migration of dye to the interior pore structure of adsorbent (Doğan et al. 2009). The adsorption process is a diffusive mass transfer process where the rate can be

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Table 3 Removal of AR97 onto WS with varying temperature

C_o (mg/L)	Removal percent of dye at temperature (°C)			
	18	30	40	50
30	61.62	50.77	41.61	37.54
40	66.55	61.49	56.5	46.91
50	71.69	65.32	60.43	57.11
60	73.33	70.21	65.49	61.82

[AR97] = 30–60 mg/L, contact time = 60 min, agitation speed = 140 rpm, adsorbent dose = 12 g/L, pH = 5.45

Table 4 Thermodynamic parameters calculated for the adsorption of AR97 onto WS

C_o (mg/L)	T (°C)	C_e (mg/L)	K_c	$\Delta G°$ (kJ/ mol)	$\Delta H°$ (kJ/ mol)	$\Delta S°$ (J/ mol K)
30	18	10.613	1.826	−1.456	−26.566	−87.388
	30	16.267	0.884	0.427		
	40	17.517	0.712	0.883		
	50	18.738	0.601	1.367		
40	18	13.380	1.989	−1.663	−19.241	−59.977
	30	13.404	1.596	−1.177		
	40	15.404	1.229	−0.680		
	50	21.238	0.883	0.334		
50	18	14.154	2.532	−2.518	−19.834	−59.977
	30	17.339	1.883	−1.594		
	40	21.744	1.229	−0.680		
	50	21.446	1.331	−0.767		
60	18	16	2.75	−2.447	−13.240	−36.903
	30	17.875	2.356	−2.158		
	40	20.702	1.898	−1.667		
	50	22.904	1.619	−1.294		

[AR97] = 30–60 mg/L, contact time = 60 min, agitation speed = 140 rpm, adsorbent dose = 12 g/L, pH = 5.45

expressed in terms of the square root of time (t). The intra-particle diffusion model can be represented as:

$$q_t = k_i \, t^{0.5} + I \qquad (9)$$

where k_i is the intra-particle diffusion rate constant (mg/g min$^{0.5}$), and I is the effect of boundary layer thickness (Nandi et al. 2009a, b). The R^2 value is calculated by the plot of q_t versus $t^{0.5}$. The intra-particle diffusion plot gave multi-linearity (Table 2), indicating the different stages (Ghasemi et al. 2011).

The effect of temperature and evaluation of thermodynamic parameters

In any adsorption process, the amounts of thermodynamic parameters are the significant indicators for practical application. To study the effect of temperature on the adsorption capacity, experiments were accomplished for four different dye concentrations (30, 40, 50 and 60 mg/L) at various temperatures (18, 30, 40 and 50 C) using 0.6 g of WS. The equilibrium contact time for adsorption was maintained at 60 min. It was observed that with increase in temperature, removal percentage decreased (Table 3). This means that the adsorption is exothermic in nature (Nandi et al. 2009a, b). The thermodynamic parameters including Gibbs free energy ($\Delta G°$), enthalpy ($\Delta H°$) and entropy ($\Delta S°$) for the adsorption of AR97 onto WS have been calculated by using equations as follows and presented in Table 4 (Senthil Kumar et al. 2010):

$$K_c = \frac{C_A}{C_S} \qquad (10)$$

$$\Delta G° = -RT \ln K_c \qquad (11)$$

$$\ln K_c = \frac{\Delta S°}{R} - \frac{\Delta H°}{RT} \qquad (12)$$

where K_c is the equilibrium constant, C_S is the equilibrium concentration of the dye in the solution (mg/L), and C_A is the amount of dye adsorbed on the adsorbent in the solution at equilibrium (mg/L). R is the gas constant, and T is the solution temperature (K). $\Delta S°$ and $\Delta H°$ were obtained from the intercept and slope of Van't Hoff plot of ln K_c versus 1/T from Eq. 12. The results are given in Table 4. The negative values of $\Delta G°$ indicated the feasibility and spontaneous nature of AR97 sorption onto WS. The value of enthalpy of a sorption process is used to determine the chemical or physical sorption. For chemical sorption, enthalpy values vary from 83 to 830 kJ/mol, and for physical sorption, they vary from 8 to 25 kJ/mol (Govindasmay et al. 2009). It can be concluded from the low value of $\Delta H°$ that the interaction between AR97 dye molecules and WS is physical. The exothermic nature is also indicated by the negative value of $\Delta H°$.

Conclusion

This study investigated the removal of AR97 from aqueous solution by WS under different operational conditions such as effect of adsorbent dose, agitation speed, contact time,

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pH and temperature. The adsorption process follows Fruendlich isotherm, which fits well heterogeneous surfaces. It was found that the adsorption mechanism follows pseudo-second-order kinetic model. BET analysis of WS showed the surface area to be $1.28 \text{ m}^2/\text{g}$. The effect of temperature was investigated to calculate thermodynamic characteristics. The analysis of these parameters revealed that the process is: (a) spontaneous, (b) exothermic and (c) mainly physical because of the low ΔH° value, negative ΔG° and ΔS°.

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