Desalination and Water Treatment
www.deswater.com
doi: 10.1080/19443994.2014.967303

57 (2016) 440–448
January

Taylor & Francis
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Removal of acid turquoise blue 2G from aqueous solution by adsorbent derived from sludge and straw: kinetic, isotherm and thermodynamic study

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Received 21 January 2014; Accepted 12 September 2014

ABSTRACT

The main objective of this work was to evaluate the adsorption capacity of acid turquoise 2G onto the adsorbent derived from sludge and straw. The sludge–straw adsorbent was prepared by pyrolysis in a dried state. The BET surface area of the adsorbent was 459.77 $m^2 \cdot g^{-1}$, micropore volume was 0.164 $cm^3 \cdot g^{-1}$, and average pore radius was 3.345 nm. The experimental data were analyzed using four adsorption kinetic models (pseudo-first-order, pseudo-second-order, intra-particle diffusion, and elovich) and related parameters were calculated. The results showed pseudo-second-order kinetic model was better fitted with experimental data than the others; the pseudo-second-order adsorption mechanism was predominant for the adsorption of acid turquoise 2G. Adsorption equilibrium was analyzed by Langmuir and Freundlich models. Langmuir isotherm was found to be more suitable than Freundlich isotherm for correlation of equilibrium data. The maximum amount of acid turquoise blue 2G adsorbed (q_m) was 1.215, 1.413, and 1.574 $mmol \cdot g^{-1}$ at 288, 298, and 308 K, respectively. Free energy of adsorption (ΔG^0), enthalpy change (ΔH^0), and entropy change (ΔS^0) were calculated, and the results indicated that the adsorption processes of acid turquoise blue 2G on the sludge–straw adsorbent were spontaneous ($\Delta G^0 < 0$), endothermic in nature ($\Delta H^0 > 0$), and accompanied by an increase in entropy change ($\Delta S^0 > 0$).

Keywords: Acid turquoise blue 2G; Adsorption; Sludge; Kinetic; Thermodynamic

1. Introduction

Many industries such as textile, tanneries, pharmaceuticals, pulp and paper, paint and electroplating industries, leather, cosmetic, printing, and plastic produce large amount of wastewater containing dyes [1]. Wastewater containing dyes are highly colored and contain various organic compounds, acids or alkalis, salts, surfactants, and toxicant which can cause damage not only to aquatic life, but also to human life due to their mutagenic, carcinogenic, and toxic effect [2,3]. So, it is essential to remove dyes before they are discharged into conventional systems. However, wastewater containing dyes are difficult to treat, since the dyes are recalcitrant organic molecules, resistant to aerobic digestion, and stable to light, heat, and oxidizing agents [4]. Therefore, colored wastewater

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treatment has aroused the attention of environmental researchers. Various treatment methods have been applied for the removal of dyes from colored wastewater, which include photo-catalytic degradation [5], electro-coagulation [6], oxidation [7,8], membrane filtration [9], biotechnology, [10] and adsorption [11–16]. Among these, adsorption process has shown to be highly efficient to remove the dyestuff, pigments, and other chemicals from wastewater. It is well known that activated carbon is the most commonly used adsorbent, but the high cost limits its application [17]. In recent years, more and more researchers applied non-conventional low-cost adsorbents to remove the various dyestuffs from an aqueous solution. Some of these are as follows: waste materials [18], fruit peels [19], Portland cement [20], Tripoli [21], date stones and palm-trees [22], pomegranate peel [23], fly ash [24,25], betonies [26], princess tree leaf [27], sepiolite [28], kaolin [29,30], etc.

Sludge is inevitably by-product of wastewater treatment plants. With the rapid growth of population and urbanization, the output of excess sludge is expected to rise in next decades. Previous investigations showed that sludge is rich in carbonaceous organic material and can be converted chemically into relatively cheap adsorbents for dyes removal from industrial effluents [10,12,13,17,31,32]. The aim of this study was to investigate the adsorption capacity of acid turquoise blue 2G onto the adsorbent derived from sludge and straw. The best-fit equilibrium isotherms and adsorption kinetic models were determined by applying adsorption equations. Thermodynamic parameters such as free energy of adsorption (ΔG^0), enthalpy change (ΔH^0), and entropy change (ΔS^0) were estimated to know the nature of adsorption.

2. Materials and methods

2.1. Materials

The molecular formula of acid turquoise blue 2G is $C_{37}H_{35}N_2O_6S_2$, molecular weight is 690.820, CAS number is 3486-30-4, and the UV–vis spectrophotometer UV-2100PC scanning spectrum indicates that the maximum absorption wavelength is 637 nm.

Dewatered surplus sludge was collected from the northern suburb municipal wastewater treatment plant in Taiyuan, China. The sludge–straw adsorbent was prepared by dry pyrolysis method [33]. In order to improve the adsorption capacity, the straw was added to the sludge in a proportion of 20%. The physical characteristics of the adsorbents were measured with a surface area analyzer (ASAP 2020 M, Micro-meritics Instrument Co., USA) by using the adsorption isotherms for gas adsorption (N_2, 77 K) [34]. The specific surface area of the adsorbent was 459.77 $m^2 \cdot g^{-1}$, calculated by using the BET equation. Micropore volume (V_{mi}) was 0.164 $cm^3 \cdot g^{-1}$ obtained by the t-plot method. Desorption average pore radius (2 $V \cdot A^{-1}$) was 3.345 nm, calculated by the Barrette Joyner Halenda method.

2.2. Methods

The study of adsorption kinetics and equilibrium is essential in supplying the fundamental information required for the design and operation of adsorption equipment for wastewater treatment. Batch kinetic experiments were carried out in an orbital shaker with initial dye concentration of 0.145, 0.290, and 0.435 $mmol \cdot L^{-1}$, respectively at the adsorbent dose of 0.8 $g \cdot L^{-1}$. The solution samples were separated at predefined time intervals and determined by using a UV–vis spectrophotometer at a wavelength of 637 nm. The adsorption capacity (q_t, $mmol \cdot g^{-1}$) at time was calculated by using Eq. (1):

$$q_t = \frac{(c_0 - c_t)V}{m} \quad (1)$$

where q_t is the amount of dye adsorbed at time ($mmol \cdot g^{-1}$), c_0 is the initial dye concentration ($mmol \cdot L^{-1}$), c_t is the dye concentration at time ($mmol \cdot L^{-1}$), m is the mass of adsorbent (g), and V is the volume of the solution (L).

Batch equilibrium experiments were conducted at different temperatures (288, 298, and 303 K), the initial concentration varied from 0.275 to 1.057 $mmol \cdot L^{-1}$ with an adsorbent dosage of 0.5 $g \cdot L^{-1}$. The samples were agitated for 12 h to reach equilibrium. The amount of dye adsorbed at equilibrium (q_e, $mmol \cdot g^{-1}$) was calculated by using Eq. (1).

3. Results and discussion

3.1. Adsorption kinetics

3.1.1. Time profiles of acid turquoise blue 2G adsorption

The contact time between the dye and adsorbent is of great importance parameter for the adsorption of dye onto the adsorbents during the wastewater treatment process. Effect of the contact time on the amount of dye adsorbed (q_t) was investigated, as given in Fig. 1.

It can be observed from Fig. 1 that the adsorption capacity of turquoise blue 2G increased with the

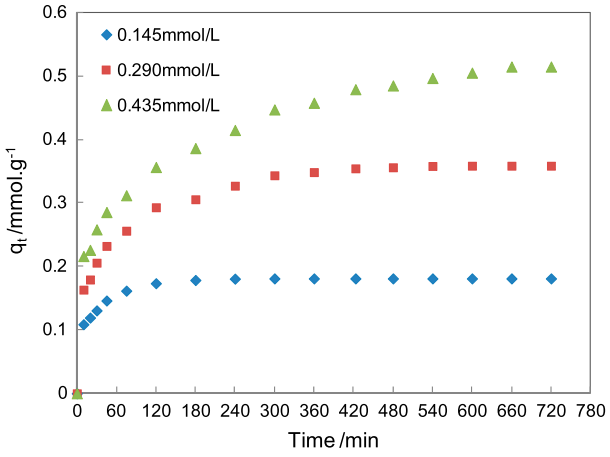

Fig. 1. Effect of the contact time on the values of q_t.

increase of the contact time, and reached to a constant value at a certain time, where adsorption and desorption process reached equilibrium. The rapid increase in the amount of the dye absorbed during the initial period may be attributed to large available amount of surface area of the adsorbent. As the surface area became gradually filled up, the adsorption rate decreased. Another significant parameter to the adsorption performance is the initial dye concentration in the solution. The amount of dye adsorbed increased with increasing initial dye concentration. The lower initial dye concentration achieved equilibrium more rapidly than the higher initial concentration. When the initial dye concentrations were 0.435, 0.290, and 0.145 mmol·L^{-1}, the equilibrium times were, respectively, 660, 300, and 120 min. It is shown that in low initial concentrations, the equilibrium would be reached faster [13,17,29]. This may be because the competition among dye molecules for active adsorption sites will be more intense in higher initial concentrations.

3.1.2. Analysis of adsorption kinetics

Kinetic analysis also helps to explore the controlling mechanism of adsorption process such as chemical reaction, mass transfer, and diffusion control. In the present work, four kinetic models including the pseudo-first-order, pseudo-second order, intra-particle diffusion, and elovich were used to fit experimental data. The equations and linear expressions of these models are presented in Table 1 [10].

The pseudo-first-order kinetic equation has been widely used to describe adsorption dynamics. The model assumes that the adsorption is a pseudo-chemical reaction process and the limiting factor for adsorption is the resistance of mass transfer inside granules. The pseudo-second-order kinetic equation has also been widely used to describe the adsorption dynamics. The model also assumes that the adsorption is a pseudo-chemical reaction process. However, unlike the pseudo–first-order kinetic equation, this model describes the whole process of adsorption and assumes that the limiting factor for adsorption is the adsorption mechanism rather than the resistance of mass transfer inside granules. Intra-particle diffusion is known as a limiting step when adsorbate is transferred onto the adsorbent from solution phase, and the intra-particle mass transfer rate controls the dyestuff adsorption onto the adsorbent [15].

The plots of four kinetic models for the adsorption of acid turquoise blue 2G onto the sludge–straw adsorbent at different initial dye concentrations are shown in Fig. 2. The derived rate constants together with the corresponding linear regression correlation coefficient R^2 values of four kinetic models are given in Table 2.

It can be concluded from the linear regression correlation coefficient R^2 values presented in Table 2, for acid turquoise blue 2G, that the best model to generate a good fit to the experimental data was the pseudo-second-order model (R^2: 0.9954–0.9999), and the amount of dye absorbed (q_e) obtained by pseudo-second-order model was closer to the experimental amount of dye absorbed (q^e), indicating that the adsorption of acid turquoise blue 2G by the sludge–straw adsorbent is complex, and includes more than one mechanism, such as external liquid film diffusion, surface adsorption, and intra-particle diffusion. The second best-fit model was the pseudo-first-order model (R^2: 0.9871–0.9943), but the amount of dye absorbed (q_e) obtained by pseudo-first-order model differ considerably from the experimental amount of dye absorbed (q^e). The intra-particle diffusion and elovich models showed limited applicability for the adsorption process. The fitting lines of q_t vs. $t^{0.5}$ did not pass through the origin, indicating that besides the intra-particle diffusion, other processes such as surface adsorption and liquid film diffusion also assume the control on the rate of adsorption simultaneously. The rate constants k_1 and k_2 decreased with increasing initial dye concentration. However, intra-particle diffusion rate constant k_p and elovich rate constant α increased with increasing initial dye concentration. These findings were in agreement with those obtained by other workers [10,12,13,17,35,36].

Table 1
Features of kinetics equations

Kinetic model	Equations	Linear expressions	Plot	Parameters
Pseudo-first order	$\frac{dq_t}{dt} = k_1(q_e - q_t)$	$\ln(q_e - q_t) = \ln q_e - k_1 t$	$\ln(q_e - q_t)$ *vs.* t	$q_e = \exp(\text{intercept})$ $k_1 = -(\text{slope})$
Pseudo-second order	$\frac{dq_t}{dt} = k_2(q_e - q_t)^2$	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$	$\frac{t}{q_t}$ vs. t	$q_e = \frac{1}{\text{slope}}$ $k_2 = \frac{(\text{slope})^2}{\text{intercept}}$
Intra-particle diffusion	$q_t = k_p t^{0.5} + C$	$q_t = k_p t^{0.5} + C$	q_t *vs.* $t^{0.5}$	$k_p = \text{slope}$
Elovich	$\frac{dq_t}{dt} = \alpha \exp(-\beta q_t)$	$q_t = \left(\frac{1}{\beta}\right)\ln(\alpha\beta) + \left(\frac{1}{\beta}\right)\ln t$	q_t vs. $\ln t$	$\beta = (\text{slope})^{-1}$ $\alpha = (\text{slope})\exp\left(\frac{\text{intercept}}{\text{slope}}\right)$

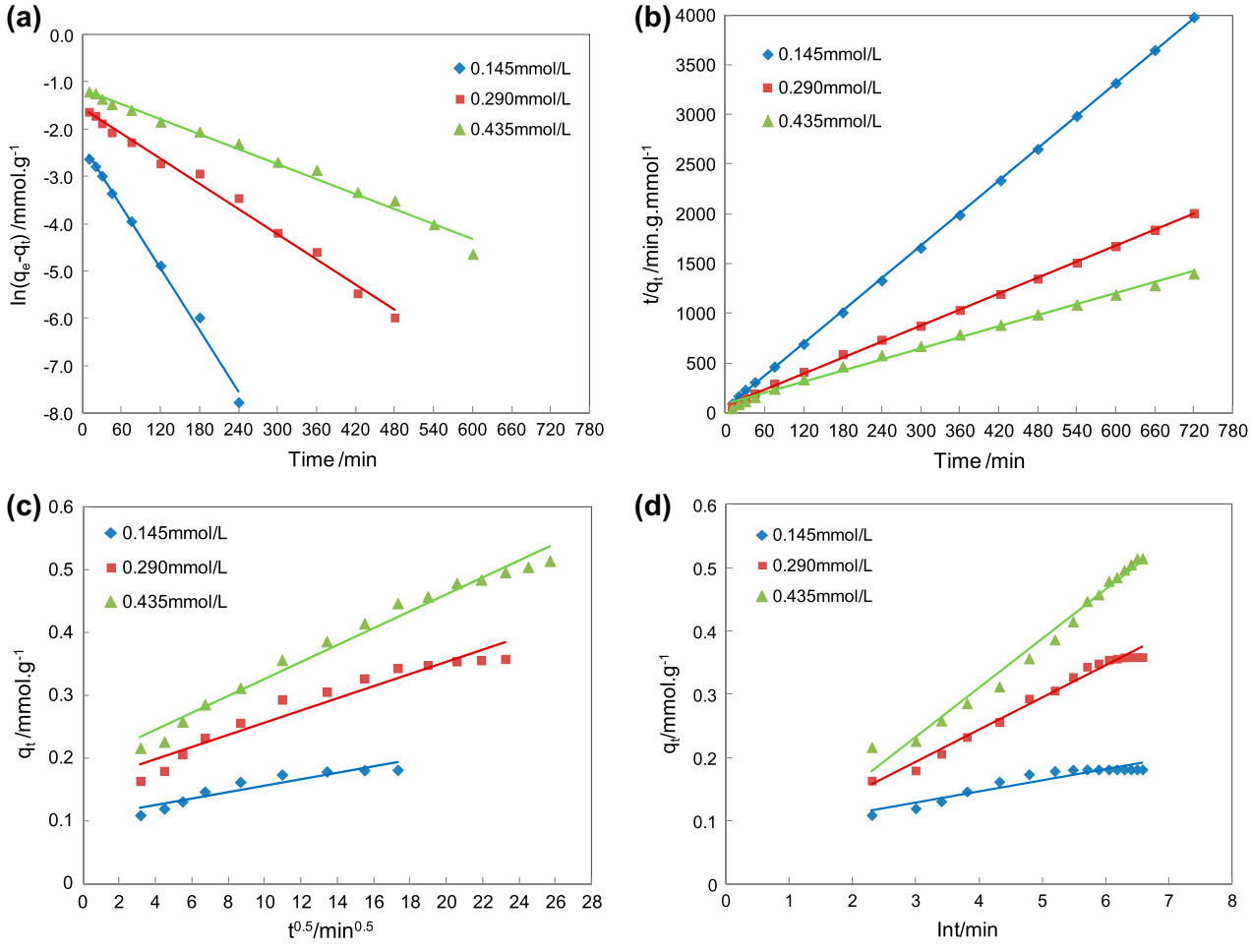

Fig. 2. Plots of four kinetic models for adsorption of acid turquoise blue 2G onto the sludge–straw adsorbent at different initial dye concentrations: (a) pseudo-first-order; (b) pseudo-second-order; (c) intra-particle diffusion, and (d) elovich models.

Table 2
Kinetic parameters for the adsorption of acid turquoise blue 2G onto sludge–straw adsorbent

Model	Variable	C_0/mmol L^{-1}		
		0.145	0.290	0.435
Pseudo-first-order	k_1/min^{-1}	−0.0217	−0.0089	−0.0053
	q_e/mmol·g^{-1}	0.0964	0.2125	0.3193
	R^2	0.9943	0.9916	0.9871
Pseudo-second-order	k_2/g·mmol^{-1} min^{-1}	0.6555	0.0995	0.0396
	q_e/mmol·g^{-1}	0.1838	0.3743	0.5383
	R^2	0.9999	0.9993	0.9954
Intraparticle diffusion	k_p/mmol·g^{-1}·$min^{-0.5}$	0.0052	0.0098	0.0136
	R^2	0.8767	0.9324	0.9789
Elovich	α/g·mmol^{-1}·min^{-1}	1.4656	0.1110	0.0763
	β/g·mmol^{-1}	56.1798	19.6464	12.8205
	R^2	0.8965	0.9838	0.9817
q^e		0.1809	0.3585	0.5147

Note: q^e and q_e represent the amount of dye absorbed at equilibrium obtained by experiment and calculation, respectively.

3.2. Adsorption isotherms

3.2.1. Isotherms of acid turquoise blue 2G adsorption

Effect of the equilibrium concentration of the adsorbate (c_e) on the amount of acid turquoise blue 2G adsorbed (q_e) at different temperatures (288, 298, and 308 K) has been investigated, and the results are shown in Fig. 3.

It has been observed that the amount of acid turquoise blue 2G adsorbed (q_e) increased with the increase of the equilibrium concentration of the adsorbate (c_e), until it reached a certain limit (0.058 mmol·g^{-1}), and then the rate of increase slowed down. This might be because there was availability of more adsorption sites in the beginning. After the critical concentration (0.058 mmol·g^{-1}), the amount of dye adsorbed (q_e) increasingly slowed down. It could be attributed to the fact that the active sites of adsorbent were filled up. It can also be seen that with the increase in temperature, the amount of acid turquoise blue 2G adsorbed (q_e) increased. The findings implied that the adsorption for acid turquoise blue 2G onto the sludge-straw adsorbent was endothermic.

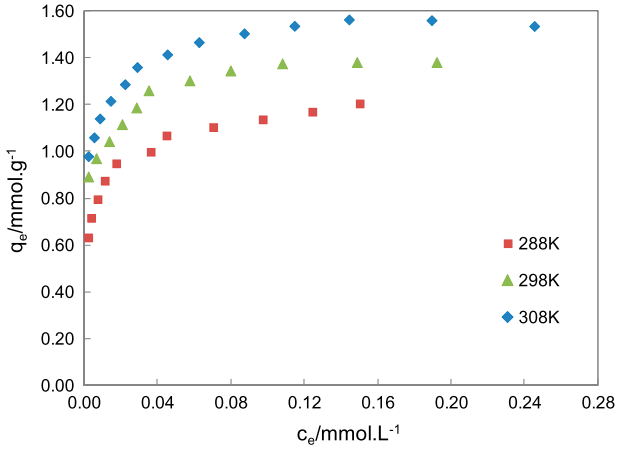

Fig. 3. Adsorption isotherms of acid turquoise blue 2G onto the sludge–straw adsorbent.

3.2.2. Analysis of adsorption isotherms

Adsorption isotherm describes the equilibrium of the adsorption of a material at a surface boundary at constant temperature. It represents the amount of material bound at the surface (the adsorbate) as a function of the material present in the solution and or in the gas phase. The most frequently used isotherms are Langmuir and Freundlich.

Langmuir isotherm is based on the fact that intermolecular force drops rapidly with the increase of distance. It proposes that the molecules of adsorbate could be adsorbed only when the molecules of adsorbate come into contact with the surface of adsorbent. Collision and contact are considered to be the preconditions of adsorption. The following assumptions are made: (1) the adsorption is localized in a monolayer on the surface of adsorbent; (2) the adsorption performance is uniform; and (3) there was no interaction between adsorbed molecules. So, Langmuir isothermal adsorption equation is written as:

$$q_e = \frac{q_m K_L c_e}{1 + K_L c_e} \tag{2}$$

where c_e is the dye concentration at equilibrium (mmol·L^{-1}), q_e is the amount of dye adsorbed at equilibrium (mmol·g^{-1}), q_m is the maximum amount of dye adsorbed (mmol·g^{-1}), and K_L is a constant related to the affinity between the adsorbent and the adsorbate (L·mmol^{-1}).

The Eq. (2) can be expressed in linear form as in Eq. (3):

$$\frac{c_e}{q_e} = \frac{1}{q_m K_L} + \frac{c_e}{q_m} \tag{3}$$

The values of q_m and K_L can be determined by plotting c_e/q_e vs. c_e.

The essential feature of the Langmuir isotherm can be expressed in terms of a dimensionless factor called separation factor (R_L), which is defined by the following Eq. (4):

$$R_L = \frac{1}{1 + a_L c_0} \tag{4}$$

where c_0 is the initial concentration (L·mmol^{-1}) and a_L is Langmuir constant (L·mmol^{-1}). The value of R_L indicates the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$).

Freundlich isotherm is an empirical model and describes the heterogeneous adsorption model with different energies of adsorption. There is no limitation for the formation of the monolayer, and the isotherm commonly refers to the reversible adsorption.

Freundlich isothermal adsorption equation is written as:

$$q_e = K_F c_e^{\frac{1}{n}} \tag{5}$$

where c_e is the dye concentration at equilibrium (mmol·L^{-1}), q_e is the amount of dye adsorbed at equilibrium (mmol·g^{-1}), and n is the concentration index. $1/n$ is between 0.1 and 0.5, indicating the adsorption process is thought to be acceptable, $1/n > 2$ indicates the adsorption is difficult, and K_F is Freundlich constant (mmol·g^{-1}) (L·g^{-1})$^{1/n}$, correlated to the maximum adsorption capacity.

The linear form of Eq. (5) can be expressed in Eq. (6):

$$\ln q_e = \ln K_F + \frac{1}{n} \ln c_e \tag{6}$$

The values of n and K_F can be determined by plotting ln q_e vs. ln c_e.

The experimental data were fitted with Langmuir and Freundlich models. Fig. 4 describes the experimental isotherm and its representation. The parameters of model are summarized in Table 3.

As can be seen from Fig. 4 and Table 3, based on the correlation coefficient R^2 values, experimental data yielded good linear plots with both Langmuir isotherm (R^2: 0.9983–0.9996) and Freundlich isotherm (R^2: 0.9627–0.9779) for acid turquoise blue 2G onto the sludge–straw adsorbent, and the Langmuir isotherm showed comparatively better applicability for the adsorption. These results are consistent with those in the literatures [12,13,17,29]. The maximum amount of acid turquoise blue 2G adsorbed (q_m) was 1.215, 1.413, and 1.574 mmol·g^{-1} at 288, 298, and 308 K, respectively. It was also found that the values of q_m, K_L, and K_F all increased with increasing temperature, indicating the adsorption process was endothermic [13]. The values

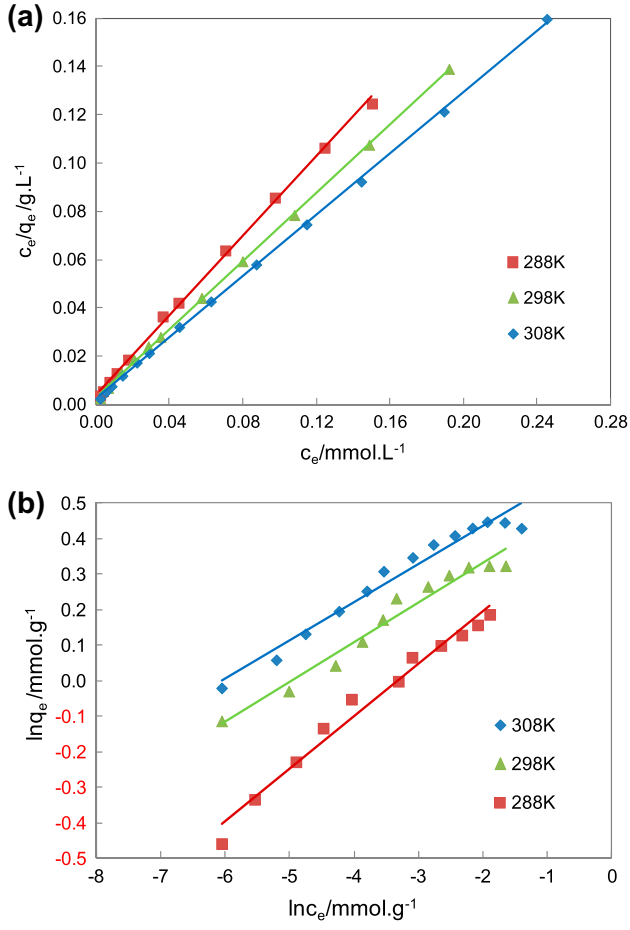

Fig. 4. Adsorption isotherms of acid turquoise blue 2G onto the sludge–straw adsorbent (a) Langmuir model; and (b) Freundlich model.

Table 3
Isotherm parameters for the adsorption of acid turquoise blue 2G onto sludge–straw adsorbent

Model	Variable	T/K		
		288	298	308
Langmuir	q_m/mmol·g^{-1}	1.2149	1.4134	1.5738
	K_L/L·mmol^{-1}	211.0512	252.6786	288.8182
	R_L	0.0063–0.0147	0.0045 (0.00447)–0.0123	0.0034–0.0070
	R^2	0.9983	0.9996	0.9996
Freundlich	K_F/(mmol·g^{-1}) . (L·g^{-1})$^{1/n}$	1.6317	1.7412	1.9142
	1/n	0.1475	0.1115	0.1074
	R^2	0.9779	0.9627	0.9631

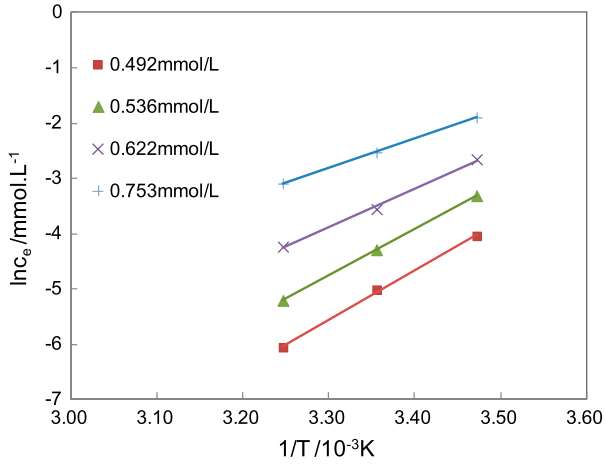

Fig. 5. Plot of ln c_e vs. $1/T$ at different initial concentrations.

of R_L (0.0034–0.0147) and $1/n$ (0.1074–0.1475) indicated that the adsorption was favorable.

3.3. Adsorption thermodynamic

The enthalpy change (ΔH^0) for the adsorption of acid turquoise blue 2G onto the sludge–straw adsorbent was determined by using Clausius–Clapeyron equation [13].

$$\ln c_e = \frac{\Delta H^0}{RT} + K_c \tag{7}$$

where c_e is the dye concentration at equilibrium (mmol·L^{-1}), ΔH^0 is change in enthalpy (J·mol^{-1}), R is the gas constant (8.314 J·mol^{-1}·K^{-1}), T is the temperature (K), and K_c is the constant.

Fig. 5 gives the plots of ln c_e vs. $1/T$ at different initial concentrations with slope of $\Delta H^0/R$, the ΔH^0 values could be calculated from the slope of the linear plot.

Since the Langmuir isotherm showed good applicability for the adsorption of acid turquoise blue 2G onto the sludge–straw adsorbent, the free energy of adsorption (ΔG^0) could be obtained from Langmuir isotherms [37].

$$\Delta G^0 = -RT \ln K_L \tag{8}$$

where K_L is the Langmuir constant (L·mmol^{-1}).

Thus, the entropy change (ΔS^0) could be calculated from the Gibbs–Helmholtz equation [13,38,39]:

$$\Delta S^0 = \frac{(\Delta H^0 - \Delta G^0)}{T} \tag{9}$$

The values of ΔH^0, ΔS^0, and ΔG^0 calculated from Eqs. (7)–(9) were presented in Table 4.

Table 4
Thermodynamic parameters for the adsorption of acid turquoise blue 2G onto the sludge–straw adsorbent at different temperatures

c_0 mmol·L^{-1}	ΔH^0 kJ·mol^{-1}	ΔG^0 (kJ·mol^{-1})			ΔS^0 (J·mol^{-1}·K^{-1})		
		288 K	298 K	308 K	288 K	298 K	308 K
0.492	74.24	−29.35	−30.81	−32.20	359.704	352.5268	345.5852
0.536	69.82				344.3549	337.6927	331.2327
0.622	58.41				304.719	299.387	294.1706
0.753	44.15				255.2219	251.5508	247.8876

The positive values of ΔH^0 indicate that the adsorption was endothermic in nature, which was also indicated by the increase in the amount of adsorption with temperature (Fig. 4 and Table 3). The positive values of ΔS^0 reflected that the molecules of acid turquoise blue 2G onto the sludge–straw adsorbent surface were organized in a more disorder state compared to those in the aqueous phase. The negative value of free energy change (ΔG^0) indicated the spontaneous nature of adsorption and confirmed affinity of the sludge–straw adsorbent for the acid turquoise blue 2G. Hence, the adsorption of acid turquoise blue 2G molecules onto the sludge–straw adsorbent was a spontaneously endothermic process. Similar observations have been reported in the literature [13,21].

4. Conclusions

The adsorption kinetics and isotherms of acid turquoise blue 2G onto the sludge–straw adsorbent were investigated in batch experimental system. It was found that for the adsorption process of acid turquoise blue 2G, the pseudo-second-order model and pseudo-first-order model both showed better applicability, and the best-fit model was found to be pseudo-second-order model (R^2: 0.9954–0.9999). The intra-particle diffusion and elovich models showed limited applicability for the adsorption process. The adsorption of acid turquoise blue 2G onto the sludge–straw adsorbent is complex and includes more than one mechanism such as external liquid film diffusion, surface adsorption, and intra-particle diffusion. Experimental data yielded good linear plots with both Langmuir isotherm (R^2: 0.9983–0.9996) and Freundlich isotherm (R^2: 0.9627–0.9779). The maximum amount of acid turquoise blue 2G adsorbed (q_m) was 1.215, 1.413, and 1.574 mmol·g^{-1} at 288, 298, and 308 K, respectively. The values of ΔH^0, ΔS^0, and ΔG^0 showed that the adsorption process for acid turquoise blue 2G onto sludge-straw adsorbent was endothermic and spontaneous.

Acknowledgments

This work was financially supported by Shanxi Science and Technology Agency Research Project (No. 20100321086) and Scientific and Technological Innovation Programs of Higher Education Institutions in Shanxi (No. 20111029).

Symbols

q_t — the amount of dye adsorbed at time, mmol·g^{-1}
q_e — the amount of dye adsorbed at equilibrium, mmol·g^{-1}
q_m — the maximum amount of dye adsorbed, mmol·g^{-1}
c_e — the dye concentration at equilibrium, mmol·L^{-1}
c_0 — the initial dye concentration, mmol·L^{-1}
c_t — the dye concentration at time, mmol·L^{-1}
k_1 — rate constant of pseudo-first-order equation, min^{-1}
k_2 — rate constant of pseudo-second-order equation, g·mmol^{-1}·min^{-1}
k_p — rate constant of intra-particle diffusion equation, mmol·g^{-1}
m — the mass of adsorbent, g
V — volume of the solution, L
K_L — the Langmuir constant, L·mmol^{-1}
R_L — equilibrium parameter
n — the concentration index
R — the gas constant, 8.314 J·mol^{-1}·K^{-1}
T — the temperature, K
K_F — Freundlich constant, (mmol·g^{-1})·(L·g^{-1})$^{1/n}$
ΔH^0 — change in enthalpy, kJ·mol^{-1}
ΔG^0 — the free energy of adsorption, kJ·mol^{-1}
ΔS^0 — the entropy change, J·mol^{-1}·K^{-1}

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