



Removal of Congo Red from aqueous solution by cattail root

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ABSTRACT

In this study, cattail root was used to remove Congo Red (CR) from aqueous solution. The effects of operation variables, such as cattail root dosage, contact time, initial pH, ionic strength and temperature on the removal of CR were investigated using batch adsorption technique. Removal efficiency increased with increase of cattail root dosage and ionic strength, but decreased with increase of temperature. The equilibrium data fitted well to the Langmuir model ($R^2 > 0.98$) and the adsorption kinetic followed the pseudo-second-order equation ($R^2 > 0.99$). Thermodynamics parameters such as standard free energy change (ΔG°), standard enthalpy change (ΔH°), and standard entropy change (ΔS°) were analyzed. The values of ΔG° were between -7.871 and -4.702 kJ mol⁻¹, of ΔH° was -54.116 kJ mol⁻¹, and of ΔS° was -0.157 kJ mol⁻¹ K⁻¹, revealing that the removal of CR from aqueous solution by cattail root was a spontaneous and exothermic adsorption process. The maximum adsorption capacities of CR on cattail root were 38.79, 34.59 and 30.61 mg g⁻¹ at 20, 30 and 40 °C, respectively. These results suggest that cattail root is a potential low-cost adsorbent for the dye removal from industrial wastewater.

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1. Introduction

More than 10,000 dyes have been widely used in textile, paper, rubber, plastics, leather, cosmetic, pharmaceutical, and food industries, which generated huge volume of wastewater every year [1]. The disposal of dye wastewater without proper treatment is a big challenge and has caused harms to the aquatic environment, such as reducing light penetration and photosynthesis [2]. Some of dyes contained in wastewater even decompose into carcinogenic aromatic amines under anaerobic conditions, which will cause serious health problems to human and animals [3]. Due to the complex molecular structure, dyes are usually very difficult to be biodegraded, making them hardly eliminated under natural aquatic environment [4].

Due to the low biodegradability, conventional biological wastewater treatment processes were not efficient in treating dyes wastewater [1]. Therefore, dyes wastewater was usually treated by physical and/or chemical methods, such as coagulation and flocculation [5], membrane separation [6], activated carbon adsorption [7], electrochemical removal [8], and photochemical degradation [9]. However, for the developing counties, these methods are still too expensive to be used widely. Developing economical adsorbents to treat dyes wastewater has attracted great interest in recent years [10]. Gupta and Suhas [11] recently reviewed the

application of low-cost adsorbents for the dye removal. Many non-conventional, low-cost adsorbents such as coir pith [12], anaerobic granular sludge [13], hazelnut shells [14], bottom ash and de-oiled soya [15–21], carbon slurry [22,23], hen feathers [24], and other waste materials [25–29], have been attempted to remove dyes from wastewater. But the adsorption capacities of most of the above were still limited. New economical, locally available and highly effective adsorbents are still under development.

Cattail is an aquatic plant and has been widely used in artificially constructed wetlands for the removal and mineralization of phenol [30], the treatment of high-strength wastewater [31], and the removal of phosphorous and heavy metals [32]. However, the cattail biomass and its root produced in the phytoremediation probably become a potential pollution sources like water hyacinth if they are not properly managed. Previous studies have shown that cattail biomass could be subjected to anaerobic digestion for biogas production [33]. Cattail root has a porous structure and a large surface area, which might be utilized as adsorbent to treat dyes wastewater.

Congo Red (CR) is an anionic dye, which has been widely used in textiles, paper, rubber and plastic industries [34]. In this study, we investigated the feasibility using cattail root to remove CR from synthetic dye wastewater.

2. Materials and methods

2.1. Material

Cattail root used in this study was collected from a local pond in Hefei, China. The root was washed with tap water to remove soil and

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Nomenclature

C_0	initial dye concentration in aqueous solution (mg L^{-1})
C_e	equilibrium concentration in solution (mg L^{-1})
C_s	equilibrium concentration on adsorbent (mg L^{-1})
h	initial adsorption rate at time approaching 0 ($\text{mg g}^{-1} \text{min}^{-1}$)
k_{ad}	rate constant of pseudo-first-order adsorption (min^{-1})
K_c	equilibrium constant
k_{id}	rate constant of intraparticle diffusion ($\text{mg g}^{-1} \text{min}^{-0.5}$)
k_2	rate constant of pseudo-second-order adsorption ($\text{g mg}^{-1} \text{min}^{-1}$)
q_e	amount of dye adsorbed on adsorbent at equilibrium time
q	amount of dye adsorbed on adsorbent at time t
Q^0	maximum adsorption capacity (mg g^{-1})
t	time (min)
R	ideal gas constant, $8.314 \text{ J mol}^{-1} \text{ K}^{-1}$
R^2	regression coefficient
T	temperature in Kelvin (K)
V	volume of aqueous solution to be treated (L)
ΔG°	standard free energy change (kJ mol^{-1})
ΔH°	standard enthalpy change ($\text{kJ mol}^{-1} \text{ K}^{-1}$)
ΔS°	standard entropy change (kJ mol^{-1})

dust, sprayed with distilled water, and dried to a constant weight at 75°C . The dry cattail root was ground and sieved to obtain particle sizes of 0.25–0.40 mm as adsorbent, and then stored in desiccators for use.

2.2. Preparation of dye solution

Congo Red used in this study was of commercial purity (C.I. 22120, FW=696.7, $\lambda_{\text{max}}=500 \text{ nm}$; Shanghai Chemical Reagent Ltd.). The chemical structure is shown in Fig. 1. Stock solution of 500 mg L^{-1} was prepared by dissolving accurately quantity of the dye in double-distilled water. The experimental solution was obtained by diluting the stock solution to the designed initial dye concentration.

2.3. Adsorption studies

The batch tests were carried out in 100 mL flasks with 50 mL of working volume. The ground cattail root was added into flasks with designed concentration of the dye solution. The initial pH of the solution was adjusted to 7.0 ± 0.3 using 0.01 M HCl or 0.01 M NaOH. The flasks were stirred at a rotary shaker at 140 rpm. Samples were collected from flasks at predetermined time intervals, and centrifuged at $10,000 \times g$ for 10 min to remove adsorbent. The concentration of CR in the supernatant was measured immediately by a

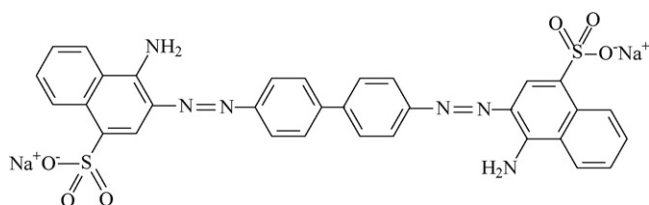


Fig. 1. Molecular structure of Congo Red.

722 Grating Spectrophotometer (Shanghai Instrument Ltd., China), at 498 nm of wavelength.

In the test of contact time and adsorbent dosage, the initial pH of the dye solution was adjusted to 7.0 ± 0.3 , the concentration of CR was 50 mg L^{-1} , and the temperature was controlled at 20°C . The cattail root dosage used was 0.5, 1.0, 3.0, 5.0, 7.0 and 10.0 g L^{-1} , respectively.

In the investigation of pH, the pH of dye solutions was adjusted to the designed values using 0.01 M HCl or 0.01 M NaOH. The CR concentration was 50 mg L^{-1} and the temperature was controlled at 20°C . The adsorbent dosage used was 5.0 g L^{-1} .

In the investigation of ionic strength, NaCl was used to adjust the ionic strength and seven different concentrations of 0, 0.001, 0.01, 0.05, 0.1, 0.5, and 1.0 mol L^{-1} were applied to adjust the ionic strength. The CR concentration used was 50 mg L^{-1} and the temperature was 20°C with adsorbent dosage 5.0 g L^{-1} .

Three different temperatures of 20, 30 and 40°C were applied to investigate the influence of temperature. The CR concentration, temperature, pH, and adsorbent dosage were the same as the test of ionic strength.

2.4. Equations and calculations

In this study, Lagergren's pseudo-order equation was used to investigate the dynamics of the adsorption process from aqueous solution. The first-order Lagergren equation is given as

$$\log(q_e - q) = \log q_e - \frac{k_{ad}}{2.303} t \quad (1)$$

where k_{ad} is the rate constant of first-order adsorption (min^{-1}), q_e and q are the amounts of dye adsorbed on adsorbent at equilibrium and at time t (min), respectively.

The second-order Lagergren equation is expressed as

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

and

$$h = k_2 q_e^2 \quad (3)$$

where k_2 is the pseudo-second-order rate constant ($\text{g mg}^{-1} \text{min}^{-1}$) and h is the initial adsorption rate at time approaching 0 ($\text{mg g}^{-1} \text{min}^{-1}$).

The effect of intraparticle diffusion resistance on adsorption can be evaluated by the following equation:

$$q = k_{id} t^{1/2} + I \quad (4)$$

where k_{id} is the rate constant of intraparticle diffusion ($\text{mg g}^{-1} \text{min}^{-0.5}$). Values of I give the information regarding the thickness of boundary layer.

Thermodynamic parameters were calculated using the following equations:

$$\Delta G^\circ = -RT \ln K_c \quad (5)$$

$$K_c = \frac{C_s}{C_e} \quad (6)$$

$$\ln K_c = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (7)$$

where ΔG° , ΔH° and ΔS° are respectively standard free energy change, standard enthalpy change, standard entropy change, K_c is the equilibrium constant, C_s is the equilibrium concentration of CR on adsorbent (mg L^{-1}), C_e is the equilibrium concentration of CR in solution (mg L^{-1}), R is the ideal gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$), and T is the adsorption temperature in Kelvin.

Langmuir isotherm model was applied to describe the adsorption of dye:

$$\frac{C_e}{q_e} = \frac{1}{Q^0 b} + \frac{C_e}{Q^0} \quad (8)$$

where Q^0 is the maximum adsorption capacity of cattail root (mg g^{-1}) and b is the equilibrium constant related to the adsorption energy (L mg^{-1}).

R_L , a dimensionless constant, was used to determine whether an adsorption is favorable, which was calculated by

$$R_L = \frac{1}{1 + bC_0} \quad (9)$$

where C_0 is the initial dye concentration and b is the Langmuir constant.

3. Results and discussion

3.1. Effect of adsorbent dosage and contact time

The effect of adsorbent dosage and contact time on the removal of CR is shown in Fig. 2. With increase of adsorbent dosage from 0.5 to 10.0 g L^{-1} , the removal of CR increased from 14.4 to 98.4%. Rapid removal of CR was observed at the beginning of the contact time. Following the rapid removal, the removal rate decreased, and an apparent equilibrium was reached after 1–6 h depending on the adsorbent dosages. The high removal rate at the start of the contact time was due to the large amount of surface area available for adsorption of the dye. At this stage, the adsorption mainly occurred on the surface of the adsorbent. After the rapid uptake, the capacity of the adsorbent became exhausted and the adsorption would be replaced by the transportation of dye from the external sites to the internal sites of the adsorbent particles. Therefore, the uptake rate began to drop down, which can be explained by intraparticle diffusion model [35], as discussed in the following Section 3.2.

3.2. Adsorption dynamics

Lagergren's pseudo-order equation is widely used to investigate the dynamics of the adsorption process from aqueous solution [36]. In this study, pseudo-first-order equation and pseudo-second-order equation were separately used to describe the adsorption process of CR by cattail root.

$\log(q_e - q)$ was calculated using the first-order Lagergren equation, as shown in Eq. (1). A plot of $\log(q_e - q)$ versus t is shown in Fig. 3a, in which the shape of the lines indicates that the first-order Lagergren equation did not fit well to the whole range of the adsorption process and was generally applicable over the initial stage of the contact time [35]. Therefore, the second-order Lagergren equation

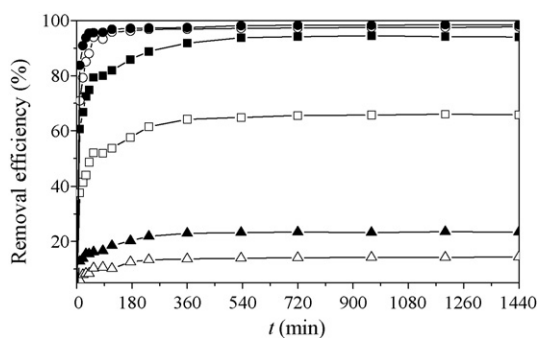


Fig. 2. Effect of adsorbent dosage and contact time on Congo Red removal (cattail root dosage: Δ , 0.5 g L^{-1} ; \blacktriangle , 1.0 g L^{-1} ; \square , 3.0 g L^{-1} ; \blacksquare , 5.0 g L^{-1} ; \circ , 7.0 g L^{-1} ; \bullet , 10.0 g L^{-1} . Congo Red 50 mg L^{-1} , initial pH 7.0 ± 0.3 , and temperature 20 °C).

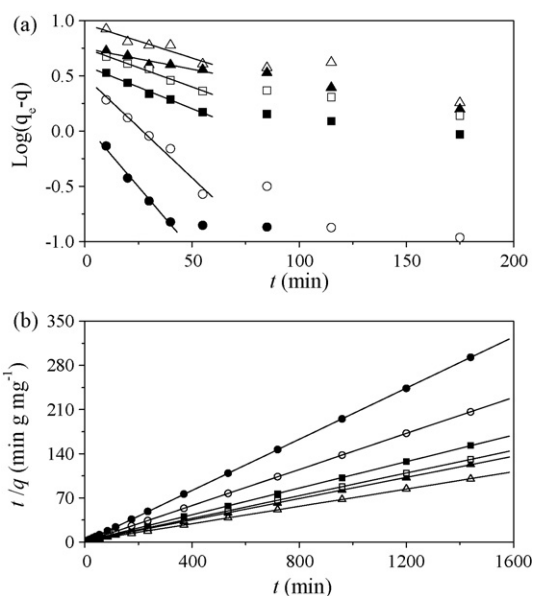


Fig. 3. Adsorption kinetic for the adsorption of Congo Red on cattail root (cattail root dosage: Δ , 0.5 g L^{-1} ; \blacktriangle , 1.0 g L^{-1} ; \square , 3.0 mg L^{-1} ; \blacksquare , 5.0 g L^{-1} ; \circ , 7.0 g L^{-1} ; \bullet , 10.0 g L^{-1} . Congo Red 50 mg L^{-1} , initial pH 7.0 ± 0.3 , and temperature 20 °C). (a) Pseudo-first-order and (b) Pseudo-second-order.

was also applied to describe the adsorption process, as listed in Eqs. (3) and (4). The intercept of the plots of t/q versus t was used to calculate the rate constant k_2 and the initial adsorption rate h . Table 1 lists the values of the rate constant k_2 , the initial adsorption rate h and the regression coefficient R^2 . All regression coefficients were more than 0.99 and the pseudo-second-order equation fitted very well to the adsorption. As seen in Fig. 3b, the pseudo-second-order equation is more likely to predict the behavior over the whole range of the adsorption process.

The effect of intraparticle diffusion resistance on adsorption was evaluated by intraparticle diffusion model to identify the adsorption mechanism [35,37,38], as expressed in Eq. (4). A plot of q against $t^{1/2}$ should give a linear line where the slope is k_{id} . Table 1 lists the values of k_{id} . Different cattail root dosages showed similar features (Fig. 4). The plot of q versus $t^{1/2}$ was linear within a certain extent but not linear over the whole time range. It can be separated into two or more linear regions. This revealed that more than one mode of adsorption functioned in the uptake of CR by cattail root. The initial linear portion might be attributed to external surface adsorption, in which the adsorbate diffuses through the solution to the external surface of the adsorbent and the uptake rate is high, and the later linear portion refers to the gradual adsorption

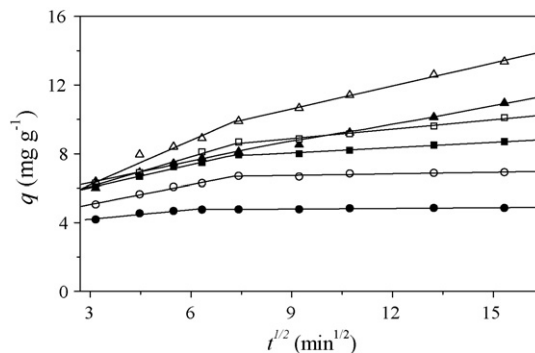


Fig. 4. Intraparticle diffusion kinetic for the adsorption of Congo Red on cattail root (cattail root dosage: Δ , 0.5 g L^{-1} ; \blacktriangle , 1.0 g L^{-1} ; \square , 3.0 mg L^{-1} ; \blacksquare , 5.0 g L^{-1} ; \circ , 7.0 g L^{-1} ; \bullet , 10.0 g L^{-1} . Congo Red 50 mg L^{-1} , initial pH 7.0 ± 0.3 , and temperature 20 °C).

Table 1
Adsorption parameters of kinetic for the adsorption of Congo Red on cattail root.

Cattail root dosage (g L ⁻¹)	k_2 (g mg ⁻¹ min ⁻¹)	h (mg g ⁻¹ min ⁻¹)	R^2	k_{id} (mg g ⁻¹ min ^{-0.5})
0.5	0.0030	0.6230	0.9997	0.4456
1.0	0.0045	0.6167	0.9998	0.2845
3.0	0.0056	0.6787	0.9999	0.1826
5.0	0.0099	0.8750	0.9999	0.1027
7.0	0.0417	2.0300	1	0.0304
10.0	0.1014	2.4546	1	0.0119

stage and the final equilibrium stage, in which the intraparticle diffusion starts to slow down and level out [35,38] because of the extremely low dye concentration remained in the solution or maximum adsorption attained [39].

3.3. Effect of pH

pH is one of the most important factors affecting the adsorption process. In order to investigate the influence of pH on the CR removal by cattail root, experiments were carried out over a pH range of 2.5–10.0 at CR concentration 50 mg L⁻¹, adsorbent dosage 5.0 g L⁻¹ and temperature 20 °C. As shown in Fig. 5, the maximum removal efficiency, approximately 100%, was achieved around pH 2.5, which was due to the very low solubility of CR at pH < 2.5. The dye removal was not affected over a pH range of 5.5–10.0.

Several parameters such as adsorption capacity of adsorbent, surface charges and active sites might be attributed to the adsorption behavior of the adsorbent at various pHs. The surface of cattail root contains a large number of active sites. The dye uptake can be related to the active sites and also to the chemistry of the dye in the solution [39]. Theoretically, at pH < isoelectrical point, the surface gets positively charged, which enhances the adsorption of the negatively charged dye anions through electrostatic forces of attraction. At pH > isoelectrical point, the surface of cattail root particles gets negatively charged, which makes OH⁻ ions compete effectively with dye anions causing a decrease of the adsorbed dyes [39]. However, in this case, the amount of the adsorbed dyes was far less than the adsorbent capacity (as calculated and discussed in Section 3.6), which made adsorption capacity become the key factor as compared with surface charges in determining the adsorption behavior, which might be the reason why the dye removal was not affected over pH range of 5.5–10.0.

3.4. Effect of ionic strength

Since NaCl is often used as a stimulator in dying processes and ionic strength affects the activity coefficients for OH⁻, H₃O⁺ and specifically absorbable ions [39], the ionic strength of NaCl may have impact on the removal of dyes from wastewater. As shown in Fig. 6, the NaCl concentrations had strong impact on the

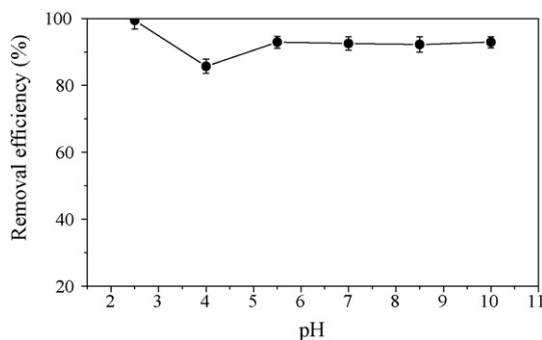


Fig. 5. Effect of pH on the removal of Congo Red by cattail root (cattail root dosage 5.0 g L⁻¹, Congo Red 50 mg L⁻¹, and temperature 20 °C).

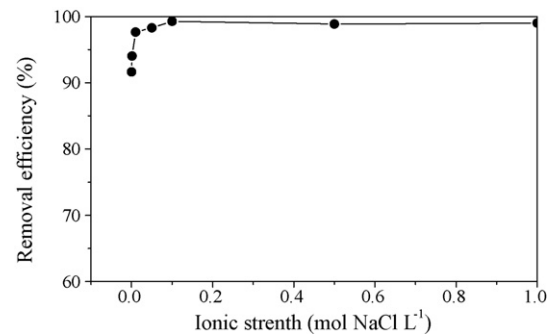


Fig. 6. Effect of ionic strength on the removal of Congo Red by cattail root (cattail root dosage 5.0 g L⁻¹, Congo Red 50 mg L⁻¹, initial pH 7.0 ± 0.3, and temperature 20 °C).

removal at CR concentration of 50 mg L⁻¹, temperature 20 °C, and pH 7.0 ± 0.3. With increase of ionic strength of NaCl from 0 to 0.1 M, the removal of CR reached 97%. With further increase to 1.0 mol L⁻¹, the CR removal increased to 99%. The reason is that increase in the ionic strength increases the positive charge of the surface, thus increases the electrostatic interaction between the dye and cattail root.

3.5. Effect of temperature

In order to understand the effect of temperature on the influence of the removal of CR by cattail root, experiments were carried out at temperatures of 20, 30 and 40 °C, respectively, pH 7.0 ± 0.3, CR concentrations of 25–400 mg L⁻¹, and adsorbent dosage 5.0 g L⁻¹. As shown in Fig. 7, the removal efficiency decreased with rise in temperature from 20 to 40 °C at the same dye concentration. A similar result on the CR removal by clay materials was obtained by Vimonses et al. [35]. The effect may be contributed to the weakening of hydrogen bonds and van der Waals interaction at higher temperatures, resulting in the weakening of physical interaction between the active sites of cattail and CR [39]. Equilibrium time at various temperatures and dye concentrations was observed to

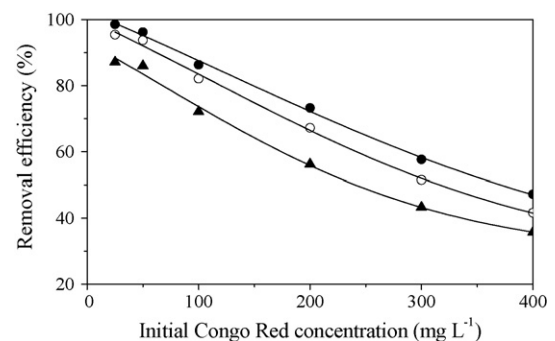


Fig. 7. Effect of temperature on the removal of Congo Red by cattail root at various dye concentrations (temperature: ●, 20 °C; ○, 30 °C; ▲, 40 °C. Initial pH 7.0 ± 0.3, cattail root dosage 5.0 g L⁻¹, and Congo Red concentration 25–400 mg L⁻¹).

Table 2
Thermodynamic parameters for the adsorption of Congo Red on cattail root.

Temperature (°C)	K_c	ΔG° (kJ mol ⁻¹)	ΔS° (kJ mol ⁻¹ K ⁻¹)	ΔH° (kJ mol ⁻¹)
20	25.32	-7.871	-0.157	-54.116
30	14.87	-6.800		
40	6.09	-4.702		

Table 3
Adsorption parameter of Langmuir isotherm and R_L for the adsorption of Congo Red on cattail root.

Temperature (°C)	Q^0 (mg g ⁻¹)	b (L mg ⁻¹)	R_L	R^2
20	38.79	0.0936	0.0260–0.2994	0.992
30	34.59	0.0743	0.0326–0.3500	0.997
40	30.61	0.0401	0.0587–0.4994	0.985

be about 6 h, indicating that equilibrium time was independent of temperature.

The variation of dye removal efficiency with respect to temperature can be explained by thermodynamic parameters, such as ΔG° , ΔH° , and ΔS° [38], which were evaluated from Eqs. (5), (6) and (7). ΔG° was calculated at the initial dye concentration of 50 mg L⁻¹. The plot of $\ln K_c$ against $1/T$ of the dye adsorption process was carried out as indicated in Fig. 8, in which the slope and intercept obtained by a curve-fitting program were used to calculate the ΔH° and ΔS° . The slope of the plot equals to $-\Delta H^\circ/R$ and its intercept value equals to $\Delta S^\circ/R$. These thermodynamic parameters are presented in Table 2.

Equilibrium constant values decreased with increasing temperature. The overall ΔG° was negative values and increased with increasing temperature as listed in Table 2. Since negative ΔG° indicates that the adsorption process is spontaneous and the shift of ΔG° to higher negative values is indicative of a rapid and more spontaneous adsorption [35], the ΔG° and its trend in this study revealed that the adsorption of CR by cattail root was spontaneous and more favorable at a low temperature in the studied range. This was confirmed by the negative value of ΔH° , which revealed that the adsorption of CR by cattail root was exothermic and likely to be dominated by physical processes in nature. The negative value of ΔS° suggests a slight decrease in randomness at the solid/solution interface with increasing temperature [38].

3.6. Adsorption isotherm

The adsorption isotherm represents the relationship between the amount adsorbed by a unit weight of solid adsorbent and the amount of adsorbate remained in the solution at equilibrium time. Langmuir isotherm models have been applied to describe the adsorption of dyes by different materials [35]. In this study, the values of $C_e q_e^{-1}$ and C_e were calculated using Eq. (8). The linear plots of $C_e q_e^{-1}$ versus C_e showed that the adsorption followed a

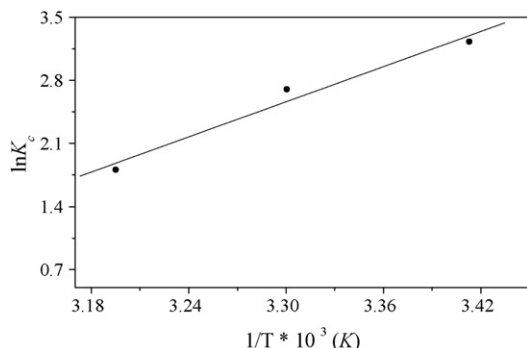


Fig. 8. Plot of $\ln K_c$ vs. $1/T$ for the removal of Congo Red by cattail root.

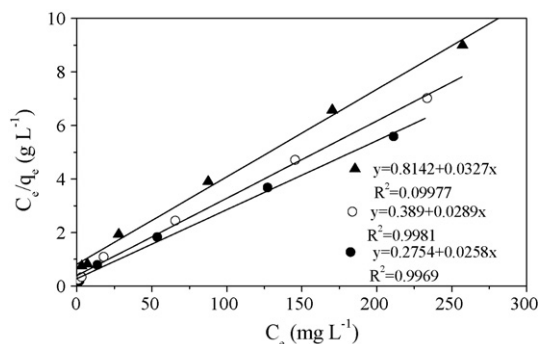


Fig. 9. Langmuir isotherm for adsorption of Congo Red on cattail root (temperature: ●, 20 °C; ○, 30 °C; ▲, 40 °C. Initial pH 7.0 ± 0.3, cattail root dosage 5.0 g L⁻¹, and Congo Red concentration 25–400 mg L⁻¹).

Langmuir isotherm, as shown in Fig. 9 ($R^2 > 0.98$). The Q^0 and b were determined from the slopes and intercepts of the respective plots and are listed in Table 3. The values of Q^0 and b decreased with increase of temperature. The maximum adsorption capacities on the adsorption of CR by cattail root at 20, 30 and 40 °C were 38.79, 34.59 and 30.61 mg g⁻¹, respectively, indicating that increasing temperature induced a lower maximum adsorption capacity. As listed in Table 4, in comparison with other materials, the maximum adsorption capacities of CR on cattail root was lower than that of activated carbon, but higher than that of wheat bran, waste orange peel and sodium bentonite [35,39–41].

R_L , a dimensionless constant, was usually used to determine whether an adsorption is favorable, which was calculated by Eq. (9). The values of R_L are basically classified into four groups, indicating the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$) [35]. The values of R_L in this study are listed in Table 3 and all are between 0 and 1, revealing that cattail root is a favorable adsorbent for CR removal, especially at lower CR concentrations.

Table 4
Adsorption capacities of Congo Red on various adsorbents.

Adsorbent	Adsorption isotherm	q_m (mg g ⁻¹)	Reference
Coal-based mesoporous activated carbons	Langmuir	52–189	[21]
Cattail root	Langmuir	38.79	Present study
Sodium bentonite	Langmuir	35.84	[17]
Wheat bran	Langmuir	22.73	[23]
Waste orange peel	Langmuir	22.44	[22]
Rice bran	Langmuir	14.63	[23]
Kolin	Langmuir	5.44	[17]

4. Conclusions

This study investigated the removal of CR by cattail root from aqueous solution. The removal efficiency of CR increased with increasing adsorbent dosage and decreased with increasing temperature from 20 to 40 °C. The equilibrium time was about 1–6 h, depending on the adsorbent dosage. Over a pH range of 5.5–10.0, the removal efficiency was independent of pH at CR concentration of 50 mg L⁻¹ and adsorbent dosage 5.0 g L⁻¹. Adsorption dynamics analysis indicates that pseudo-second-order equation fitted very well to the adsorption of CR on cattail root ($R^2 > 0.99$). Intraparticle diffusion model shows that more than one mode of diffusion functioned in the adsorption of CR on cattail root. The adsorption process followed well to the Langmuir model ($R^2 > 0.98$). The maximum adsorption capacities of CR on cattail root were 38.79, 34.59 and 30.61 mg g⁻¹ at 20, 30 and 40 °C, respectively. Further thermodynamic analysis reveals that the removal of CR from aqueous solution by cattail root was a spontaneous and exothermic process. These results suggest that cattail root is a potential low-cost adsorbent for the dye removal from industrial wastewater.

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