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Study on the kinetics of the adsorption of reactive brilliant red K-2BP onto modified soybean straw activated carbon

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ABSTRACT

In this paper, the soybean straw was modified into activated carbon adsorbent by chemical activation using zinc chloride as activator, and the influence of pH on the adsorption effect and the adsorption kinetic characteristics of reactive brilliant red K-2BP under different dosage, oscillation speed and temperature were investigated by static experiments. In addition, the kinetic date were respectively fitted by pseudo-first-order equation, pseudo-second-order equation and the intra particle diffusion equation. It was found that the adsorption effect could reach the best and the decolorization rate was as high as 95.6% when pH was 4. The adsorption effect was mainly affected by the dosage of adsorbent, and temperature and oscillation speed had little effect on it. The pseudo-second-order equation could provide good description to the adsorption process which was about the adsorption of adsorbent to reactive brilliant red K-2BP, and the correlation coefficient R^2 of which could reach at 0.999. The intra particle diffusion equation indicated that intra particle diffusion was the main control step of the adsorption rate but not the sole rate controlling step. Thermodynamic analysis suggested that the adsorption activation energy was small and its value was 16.6 KJ/mol, which indicated that the adsorption type of the whole process mainly was physical adsorption and its adsorption rate was fast. When the temperature was 20°C, 25°C or 30°C, the ΔG° was greater than zero. It indicated that the adsorption was a non-spontaneous reaction, which required additional energy to promote the reaction.

Keywords: Modified carbonization straw; Reactive brilliant red K-2BP; Adsorption kinetics; Dyestuff wastewater

1. Introduction

China is a major producer of straw [1], and there are many kinds of straw produced in agriculture. However, most of the straws are usually burned and abandoned rather than fully reused, which causes serious environmental pollution and wastes a lot of resources [2]. In response to the call of building a resource-saving and environment-friendly society, it is a good way to deal with agricultural waste by reusing these residues and treating waste by waste [3]. The wastewater from textile printing and dyeing has the characteristics of large amount of water, high content of organic pollutants, high

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alkalinity, quality changes greatly and so on. Therefore, it is a refractory industrial wastewater [4]. At present, in industrial production, the removal methods of dye wastewater mainly includes flocculation, chemical oxidation, membrane separation, adsorption of activated carbon and so on [5–8]. However, these technologies are limited by the low efficiency, high cost and the regeneration. Reactive brilliant red K-2BP is one of the dyes used widely in textile, printing and dyeing industry, and the wastewater treatment of which is typical [9]. For the moment, the study on the adsorption kinetics of agricultural straw by chemical modification is not enough. Therefore, this paper used the common soybean straw as material and used $ZnCl_2$ as activator to modify soybean straw. It investigated the influence of pH on the adsorption effect and the adsorption kinetic characteristics of reactive brilliant red K-2BP under different dosage, oscillation speed and temperature. The aim of this paper is to find an efficient and low-cost treatment technology for waste disposal, which is very important to realize the utilization of agricultural solid waste.

2. Materials and methods

2.1. Materials, reagents and instruments

Soybean straw was taken from the suburb of Chengdu, Sichuan. Zinc chloride and hydrochloric acid were analytical pure. Reactive brilliant red K-2BP was produced by Ji'nan Mingxin chemical factory, and its structure and molecular formula are shown in Table 1.

Visible spectrophotometer: V-1200, Chengdu Shengye instrument factory. Electronic analytical balance: Dragon ESJ120-4, Chengdu Shengye instrument factory. DHG series heating and drying oven: DHG-9140, Chengdu new instrument factory owned by state. Analytical grinding machine: IKA A11BASIC, Chengdu new instrument factory owned by state. Constant temperature oscillator: THZ-032, Chengdu Shengye instrument factory. Muffle furnace: XL high temperature oven, Beijin Tiantong technology company.

2.2. Preparation of modified soybean straw activated carbon

In this paper, soybean straw was used as raw material to prepare activated carbon by using chemical activation method, and the activator was zinc chloride [10]. First, distilled water was applied to clean soybean straw. Then soybean straw was dried under the condition of 100°C in DHG series heating and drying oven and it was cut into long strips of 5 cm. Next, the dried material was transferred to the crucible in the muffle furnace, and the temperature was raised to 800°C in the heating rate of 10°C/min and kept it for 90 min [11]. Next, the raw material was cooled to room temperature and then it was removed and sieved (40 ~ 60 mesh). The screened carbonized straw was mixed with 3 mol/L $ZnCl_2$ according to the mass ratio of 1:3 in a 100 mL conical flask and closed it immediately, and then it was oscillated in the constant temperature oscillating machine(temperature: 25°C, oscillation speed: 150 rpm, oscillation time: 120 min). At the end of the oscillation, solid modified carbonized straw was obtained by filtering the mixed liquor. Firstly, it was washed with 1:9 dilute hydrochloric acid solution (1 mol/L). Secondly, it was washed repeatedly with distilled water to neutral. Thirdly, it was dried to constant weight. After grinding, the activated carbon in 200 mesh was selected for use.

2.3. Adsorption experiment of dye wastewater

2.3.1. Standard curve of reactive brilliant red K-2BP

The absorbance of reactive brilliant red K-2BP at different concentrations was measured by visible spectrophotometer of V-1200 at 535 nm and the results are shown in Table 2. The absorbance of Table 2 was taken as abscissa and the concentration was taken as the ordinate [12], and the standard curve of reactive brilliant red K-2BP was drawn as shown in Fig. 1.

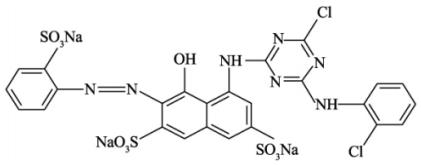
2.3.2. Adsorption experiment

The 50 mL 100 mg/L reactive brilliant red K-2BP was used to simulate the dye wastewater and it was installed in 100 mL conical flask with stopper. Then a certain amount of modified soybean straw was added in it, and adsorption

Table 2
The absorbance value of different concentrations of reactive brilliant red K-2BP

Concentration(mg/L)	Absorbance
0	0
4	0.029
8	0.060
12	0.084
16	0.113
20	0.151
24	0.171
28	0.200
32	0.241

Table 1
Structure and molecular formula of reactive brilliant red K-2BP

Dye	Structural formula	Molecular formula	Molecular weight
Reactive brilliant red K-2BP		$C_{25}H_{14}C_{12}N_7Na_3O_{10}S_3$	808.48

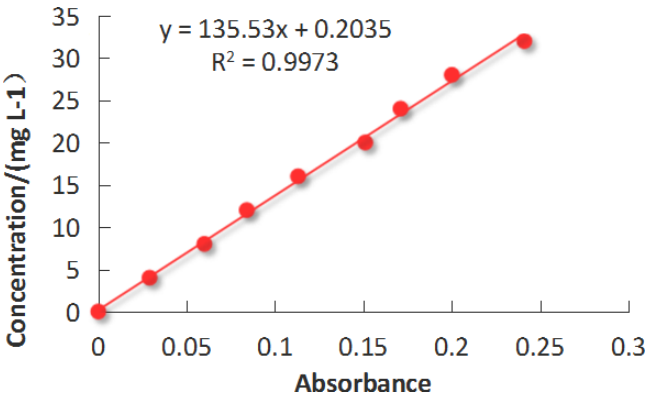


Fig. 1. Standard curve of reactive brilliant red K-2BP.

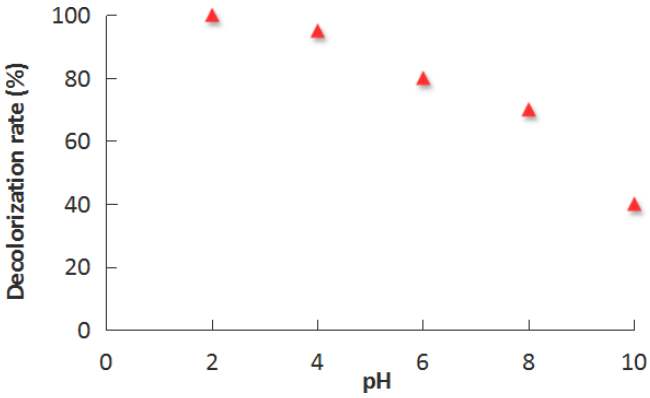


Fig. 2. Effect of pH on decolorization rate

occurred about 4 h in the reactor with a certain oscillation speed and temperature. Next it was filtered to obtain the clear liquid. Finally, the absorbance was determined at 535 nm, and the concentration was measured according to the standard curve [13]. Eqs. (1) and (2) were used to calculate the amount of dye adsorption capacity and decolorization rate.

$$q = (\rho_0 - \rho_e)\frac{V}{m} \tag{1}$$

$$\eta = \left(1 - \frac{\rho_e}{\rho_0}\right) \times 100\% \tag{2}$$

where q is the dye adsorption capacity (mg/g), ρ_0 is the concentration of dye before adsorption (mg/L), ρ_e is the equilibrium dye concentration after adsorption (mg/L), V is the volume of the dye solution (L), m is the quality of modified soybean straw (g), η is the dye decolorization rate (%).

3. Results and discussion

3.1. The effect of pH on the removal rate of reactive brilliant red K-2BP

The effects of pH on the removal rate of reactive brilliant red K-2BP can be shown from Fig. 2. The decolorization efficiency of adsorbent decreased with the increase of pH value. When pH is 4, the adsorption effect is the best and the decolorization rate is as high as 95.6%. This is because reactive brilliant red K-2BP is a soluble anionic dye, and its molecular structure contains a large number of $–SO_3^-$ (it can be seen in Table 1). In acidic environment, the protonation effect of $–SO_3^-$ is enhanced, which causes a large number of $–SO_3^-Na$ in the dye molecule to exist in the form of $–SO_3H$. However, $–SO_3H$ combines with the amine groups of modified soybean straw adsorbent to produce $=NH_2^-SO_3^-$ or $–NH_2^-SO_3^-$ [14,15]. So that the $–SO_3^-H$ of the dye molecule exchange with the exchangeable Cl^- in the adsorbent and then combined with the modified soybean straw adsorbent, which enhances the adsorption capacity. With the increase of the pH value of the system, the protonation effect of $–SO_3^-$ decreases so that $–SO_3^-H$ decrease with it, which results in the reduction of the exchangeable ion exchange between the dye molecules and the modified soybean straw adsorbent [15]. In addition, the pH of reactive brilliant red (100 mg/L) shows weak acid. Therefore, the choice of pH is about 4, everything considered.

3.2. Adsorption kinetics

3.2.1. Adsorption kinetic model

In fact, the adsorption of solution is a complicated process. Therefore, in order to design a high efficient adsorption system, the analysis of adsorption kinetics and mass transfer process is very important. The pseudo-first-order equation, pseudo-second-order equation and intra particle diffusion equation are usually used to describe and analyze the adsorption kinetics process.

The pseudo-first-order model and pseudo-second-order model are widely used to describe the adsorption kinetics, and they assume that adsorption is a pseudo chemical reaction. The pseudo-first-order kinetics considered that the mass transfer resistance is the limiting factor of adsorption, and its linear expression is as follows [16].

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{3}$$

In the formula, q_e is equilibrium adsorption capacity (mg/g), q_t is adsorption amount of t (mg/g), k_1 is rate constant of pseudo-first-order equation (min^{-1}). If the adsorption process conforms to the pseudo-first-order model, the $ln(q_e - q_t) - t$ curve should be a straight line.

The pseudo-second-order model considered that the limiting factor of adsorption is the adsorption matrix rather than the mass transfer within the particle. The linear expression is as follows [17].

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

where k_2 is rate constant of pseudo-second-order ($g \cdot mg^{-1} \cdot min^{-1}$). If the adsorption process conforms to the pseudo-second-order model, the $t/q_t - t$ curve should be a straight line.

Intra particle diffusion is one of the main processes for the transfer of adsorbate from solution to solid phase. Therefore, the intra particle diffusion process is often the controlling step of adsorption rate. The linear expression of intra particle diffusion is as follows [18].

$$q_t = k_p t^{0.5} + C \quad (5)$$

where C is intercept, which reflects the thickness of the boundary layer. The larger the C value, the greater the boundary layer effect. k_p is rate constant of intra particle diffusion, which related to D (g·mg^{-1}·min$^{-0.5}$).

The relationship between the intra particle diffusion rate constant k_p and the particle diffusion coefficient D is as follows.

$$K_p = \frac{6q_e}{R}\sqrt{\frac{D}{\pi}} \quad (6)$$

where R is particle radius (cm^{-1}). If the q–t is a straight line and passes though the origin, the intra particle diffusion process is the only rate controlling process.

3.2.2. Effect of adsorbent dosage on adsorption kinetics

Modified soybean straw with different dosage (1.6 g/L, 2.0 g/L, 2.4 g/L) was used for the adsorption of 100 mg/L reactive brilliant red, and the adsorption kinetics curve is shown in Fig. 3, and the reaction conditions were as follows: the temperature was 25°C, pH was 4, the oscillation speed was 150 rpm. The results shown that the larger the amount of adsorbent was, the smaller the adsorption capacity was which caused the adsorption balance to take longer time. This phenomenon may be caused by the lack of dye molecules. Moreover, under different dosage of adsorbent, the adsorbent showed similar adsorption kinetics, and each curve can be divided into the following three stages: for example, when the adding amount was 1.6 mg/L, the slope of the first stage was very large, and the adsorption capacity increased from 21.43 mg/L to 56.48 mg/L in 50 min. Such rapid adsorption may be caused by the van Edward force between the adsorbent and the adsorbate, which shown that the adsorption process was a fast physical process [19]. In the second stage, the slope was small, and the adsorption capacity increased from 56.48 mg/L to 60.56 mg/L in 25 min, which shown that the growth was slow. In the third stage, the amount of adsorption increased and then reached equilibrium. That is because when the adsorption time was more than 75 min, almost all of the dye molecules were adsorbed so that the adsorption amount increased little [20].

The pseudo-first-order equation, pseudo-second-order equation and intra particle diffusion equation were used respectively to fit the experimental data of Fig. 3, and the result is shown in Figs. 4a~c and the fitting parameters are shown in Table 3.

From the pseudo-first-order equation fitting curve (Fig. 4a and Table 3), it can be known that the correlation coefficient R^2 of pseudo-first-order equation was 0.798~0.951, which can describe the initial stage of adsorption process well. However, with the increase of adsorption amount, the adsorption data gradually deviated from the fitting curve. Therefore, the pseudo-first-order equation can only describe the adsorption process before adsorption saturation [21].

From the pseudo-second-order equation fitting curve (Fig. 4b and Table 3), it can be known that the correlation coefficient R^2 of pseudo-second-order equation was almost 0.999. Therefore, the pseudo-second-order kinetic equation

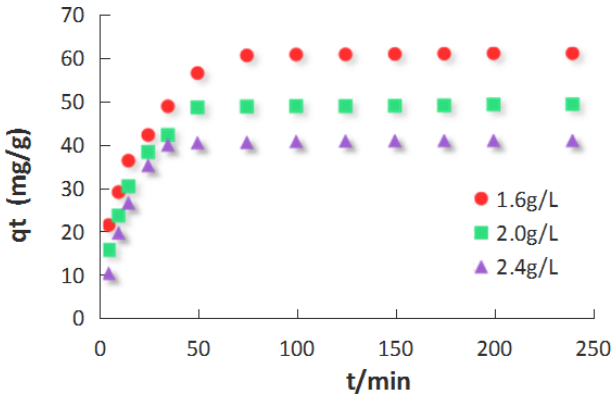


Fig. 3. Adsorption kinetic curves of adsorbents with different dosage.

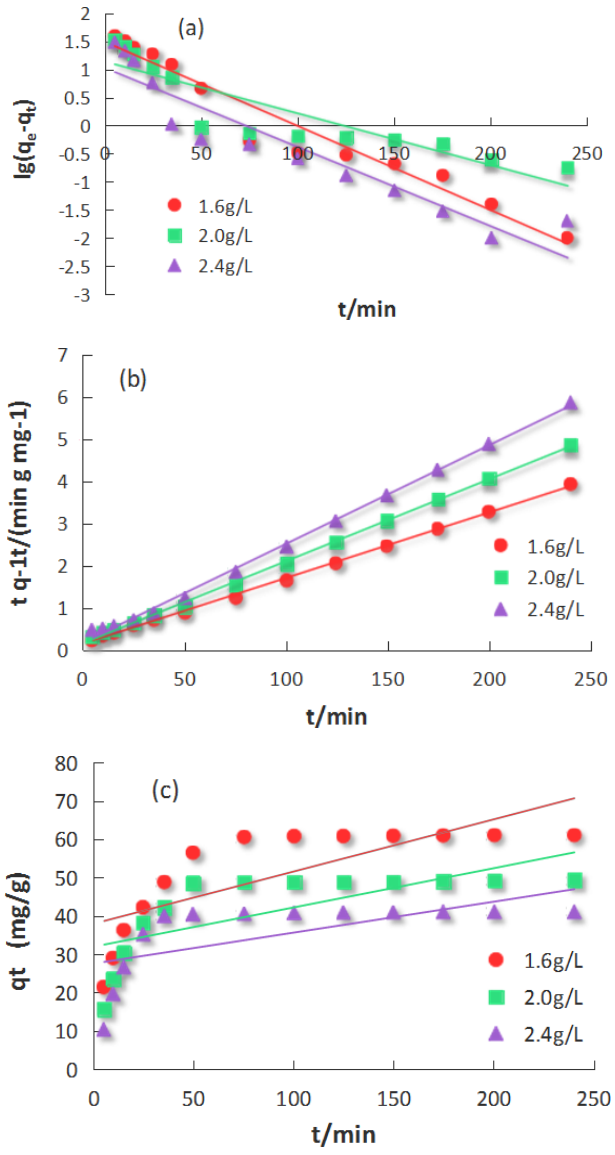


Fig. 4. Adsorption kinetic fitting curve of different dosage of adsorbent ((a) the pseudo-first-order equation, (b) the pseudo-second-order equation, (c) the intra particle diffusion equation).

Table 3
The fitting results of reaction rate equation under different dosage of adsorbent

m (g/L)	The pseudo-first-order equation		The pseudo-second-order equation			The intra particle diffusion equation		
	k_1 (min^{-1})	R^2	k_2 (g mg^{-1} min^{-1})	h (mg (g min)$^{-1}$)	R^2	k_p (mg g^{-1} $min^{-0.5}$)	C	R^2
1.6	0.009	0.798	0.023	85.78	0.998	0.136	38.18	0.587
2.0	0.015	0.951	0.019	46.27	0.999	0.102	32.18	0.502
2.4	0.014	0.891	0.016	26.91	0,999	0.081	27.78	0.403

can describe the whole adsorption process accurately. For instance: external liquid film diffusion, surface adsorption, intra particle diffusion and so on [22].

From the fitting curve of intra particle diffusion equation (Fig. 4c and Table 3), it can be seen that the value of R^2 of the curve was low so that the fitting degree was poor. At the same time, the adsorption rate constant decreased with the increase of the dosage, which indicated that the adsorption process was controlled by intra particle diffusion. The fitting curve was linear, but the curve was not through the origin. Therefore, it can be seen that the adsorption intra particle diffusion process of reactive brilliant red K-2BP particles in soybean straw with carbonization was the controlling step of adsorption rate, but it was not the sole rate controlling step. In addition, the adsorption rate was controlled by the diffusion process of particles (such as surface adsorption and liquid film diffusion) [23].

The variance R of the three kinds of curves (the results take up to three decimal places) from large to small were pseudo-second-order equation, pseudo-first-order equation and intra particle diffusion equation. Therefore, the fitting degree of the pseudo-second-order equation was the highest, the pseudo-first-order equation was the second, and the intra particle diffusion equation was the lowest. At the same time, according to calculation of equation curve, the maximum adsorption capacity of actual measurement $q_{e,exp}$ was closed to the maximum adsorption capacity q_{e2} calculated by pseudo-second-order equation. Therefore, the above results showed that the adsorption of modified soybean straw on reactive brilliant red K-2BP conformed to the pseudo-second-order model.

3.2.3. Effect of oscillation speed on adsorption rate

Modified soybean straw at different oscillation speed (100 rpm, 150 rpm, 200 rpm) was used for the adsorption of reactive brilliant red K-2BP and the adsorption rate curve is shown in Fig. 5, and the reaction conditions were as follows: the temperature was 25°C, pH was 4, the dosage of adsorbent was 2.0 g/L. With the increase of oscillation speed from 100 rpm to 200 rpm, the adsorption capacity of adsorbent increased, but the final equilibrium adsorption amount was not changed too much. That is because reactor was oscillated in a short period of time, the migration velocity of the solid particles in the solution was accelerated, which increased the concentration of dye molecules on the surface of solid particles so that the adsorption capacity increased [19]. However, with the increase of vibration time, the vibration velocity was not the main factor affecting the adsorption amount. Therefore, the final equilibrium adsorption amount was not changed too much.

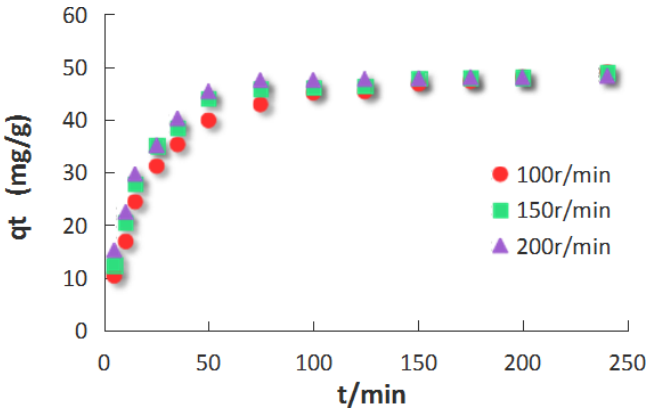


Fig. 5. Adsorption kinetics curves with different oscillation velocities.

Three kinetic equations were used to fitting the adsorption kinetic curve about the modified soybean straw and reactive brilliant red K-2BP with different oscillation speed, and the fitting parameters are shown in Table 4. The results showed that the pseudo-second-order equation can describe the adsorption process of adsorbent to reactive brilliant red K-2BP well, and the correlation coefficient was 0.999.

3.2.4. Effect of temperature on adsorption rate

Modified soybean straw was used for the adsorption of reactive brilliant red K-2BP at different temperature (20°C, 25°C, 30°C) [24] and the adsorption rate curve was shown in Fig. 6. It can be seen from the figure that the equilibrium adsorption capacity increased slightly with the increase of temperature, but the growth rate was small, which showed that temperature had little influence on adsorption effect of reactive brilliant red. Therefore, it was suitable to use under the condition of normal temperature.

Three kinetic equations were used to fitting the adsorption kinetic curve about the modified soybean straw and reactive brilliant red K-2BP with different temperature, and the fitting parameters are shown in Table 5. The results show that the pseudo-second-order equation can describe the adsorption process of adsorbent to reactive brilliant red K-2BP well, and the correlation coefficient was 0.998.

3.3. Thermodynamic parameter analysis of adsorption activation state

It assumed that the change of activation entropy ΔS^* and the change of activation enthalpy ΔH^* in the adsorp-

Table 4
Fitting results of reaction rate equation with different oscillation velocity

n_0 (rpm)	Pseudo-first-order equation		Pseudo-second-order equation			Intraparticle diffusion equation		
	k_1 (min^{-1})	R^2	k_2 (g mg^{-1} min^{-1})	h (mg (g min)$^{-1}$)	R^2	k_p (mg g^{-1} min$^{-0.5}$)	C	R^2
100	0.009	0.912	0.019	45.67	0.999	0.133	24.80	0.674
150	0.007	0.910	0.020	47.86	0.999	0.115	28.46	0.586
200	0.010	0.898	0.019	48.25	0.999	0.105	30.49	0.547

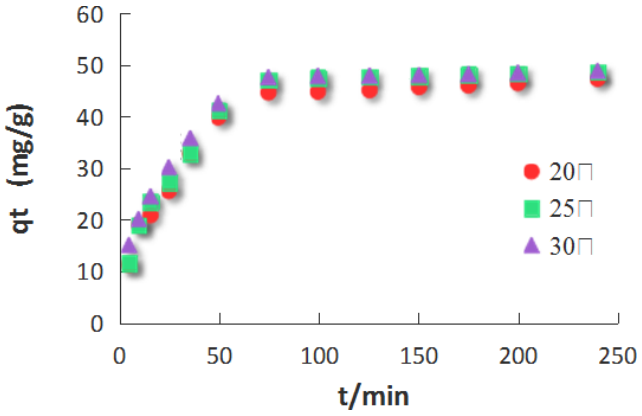


Fig. 6. Adsorption kinetics curves with different temperatures.

tion process were less affected by the temperature, which can be neglected, and it can be obtained according to the Arrhenius equation: [25,26].

$$In\,K = In\,A - \frac{E_a}{RT} \tag{7}$$

where k is constant of action, pseudo-second-order adsorption rate constant is k_2. Ea is the activation energy of the adsorption process (KJ/mol). R is molar gas constant, which usually takes R = 8.314. A is pre-exponential factor. The drawing about $-ln\,k$ and $1/T$ is shown in Fig. 7.

From the Arrhenius equation it can be seen that E_a/R is the slope of the trend line. Therefore, the activation energy of the reaction can be obtained E_a = 16.6 KJ/mol (5~20 KJ/mol). It is indicated that the adsorption process is a diffusion controlled process, which belongs to physical adsorption and its adsorption rate is very fast.

Gibbs free energy, activation enthalpy and activation entropy can be calculated from Eyring equation:

$$In\frac{K}{T} = In\frac{k_B}{h} + \frac{\Delta S^*}{R} - \frac{\Delta H^*}{RT} \tag{8}$$

where k_B is Boltzmann constant, h is Plank constant, R is Gas constant, k is Reaction rate constant. The pseudo-second-order adsorption rate constant is k_2.

The drawing about $-ln(k/T)$ and $1/T$ is shown in Fig. 8.

From the Eyring equation it can be seen that $\Delta H^*/R$ is the slope of the trend line, $-ln(k_B/h)-\Delta S^*/R$ is the intercept of the trend line. Therefore, the activation enthalpy ΔH^* and activation entropy ΔS^* of the reaction can be obtained.

The ΔG^* is activation Gibbs free energy [27], which is obtained by:

$$\Delta G^* = \Delta H - T\Delta S^* \tag{9}$$

The thermodynamic parameters of the adsorption activation states are shown in Table 6.

$\Delta H^* > 0$, it indicated that the reaction was endothermic. Therefore, increasing the temperature was conducive for reactive brilliant red molecules to overcome the steric hindrance in activated carbon, which accelerated the adsorption process. $\Delta S^* < 0$, it suggested that the degree of confusion in the molecule was reduced in the adsorption process which reactive brilliant red molecules from the solution to the activated carbon, and the ordered performance of the whole system was increasing. $\Delta G^* > 0$, it indicated that the adsorption of modified soybean straw to reactive brilliant red was non spontaneous reaction. Therefore, it is necessary to add energy to promote the reaction, and this study used oscillations to achieve this goal.

4. Conclusions

In this experiment, soybean straw was used as raw material, and activated carbon was prepared by $ZnCl_2$ activation method, which can effectively absorb reactive brilliant red K-2BP. Moreover, the removal efficiency decreased with the increase of pH value, and the adsorption effect could reach the best and the decolorization rate was as high as 95.6% when pH was 4. At the same time, the adsorption effect was mainly affected by the dosage of adsorbent, and it was less affected by temperature and oscillation speed.

The pseudo-second-order equation could provide good description to the adsorption process which was about the adsorption of adsorbent to reactive brilliant red K-2BP, and the correlation coefficient R^2 could reach at 0.999. The intra particle diffusion was the main control step of the adsorption rate, but it was not the only rate controlling step. In addition, the adsorption rate was controlled by the diffusion process of particles (such as surface adsorption and liquid film diffusion). Therefore, the adsorption process was the result of a variety of kinetic mechanisms.

Thermodynamic analysis suggested the adsorption activation energy of adsorption reaction related modified soybean straw and reactive brilliant red K-2BP was small and its value was 16.6 KJ/mol, which indicated that the adsorption process mainly was physical adsorption and its adsorption rate was fast. When the temperature was 20°C, 25°C or 30°C, the ΔG^* was greater than 0, and $\Delta H^* > 0 \Delta S^* < 0$, which indicated that the adsorption was a non-spontaneous reaction and it required additional energy to promote the reaction.

Table 5
Fitting results of reaction rate equation with different temperatures

T_0 °C	Pseudo-first-order equation		Pseudo-second-order equation			Intra particle diffusion equation		
	k_1 (min^{-1})	R^2	k_2 (g mg^{-1} min^{-1})	h (mg (g min)$^{-1}$)	R^2	k_p (mg g^{-1} min$^{-0.5}$)	C	R^2
20	0.011	0.879	0.016	44.80	0.998	0.131	23.92	0.671
25	0.013	0.939	0.019	44.88	0.997	0.136	25.12	0.658
30	0.011	0.912	0.020	45.43	0.999	0.125	27.33	0.640

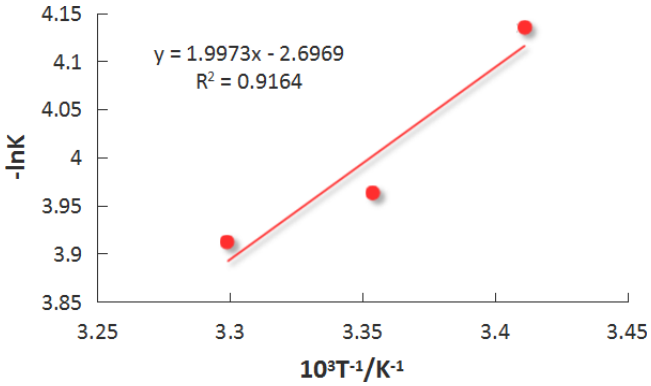


Fig. 7. Curve of *–lnk~1/T*.

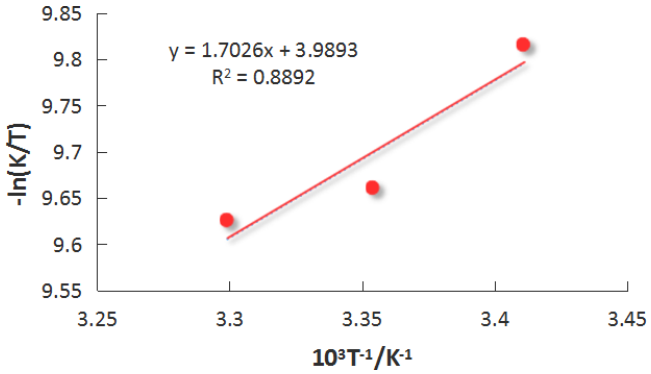


Fig. 8. Curve of *–ln(k/T)~1/T*.

Table 6
Thermodynamic parameters of the adsorption activation states

T(K)	Ea (kJ/mol)	ΔG^* (kJ/mol)	ΔH^* (kJ/mol)	ΔS^* (J/mol·K)
293.15	16.6	81.38	14.2	–230.7
298.15		82.98		
313.15		84.14		

Acknowledgements

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