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RESEARCH ARTICLE

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Adsorption of reactive brilliant red K-2BP on activated carbon developed from sewage sludge

Abstract Activated carbon was prepared from the sewage sludge of municipal wastewater treatment plant by chemical activation (activation reagent is $ZnCl_2$) and was used for the adsorption of dye (reactive brilliant red K-2BP). The impact of adsorbent amount, adsorption time and pH value on adsorption effect, the adsorption kinetics, and the adsorption thermodynamics were discussed according to batch adsorption tests. The results indicated that the activated carbon developed from sewage sludge (ACSS), which was mesoporous, possessed opened porous structures. The iodine number of the ACSS was 326 $mg \cdot g^{-1}$. The rate of achievement was 51.31%. The BET surface area was 298 $m^2 \cdot g^{-1}$ and the contents of heavy metals in the leachate didn't exceed the contents limit. The adsorption kinetics of reactive brilliant red K-2BP on the ACSS was accorded with the two-step kinetics rate equation and pseudo-second-order kinetics equation. Compared to the Freundlich isotherm equation, the Langmuir isotherm equation showed better applicability for the adsorption. The adsorption which was favorable was an endothermic (enthalpy $\Delta H > 0$) and spontaneous (free energy $\Delta G < 0$) process and was accompanied by an increase in entropy ($\Delta S > 0$).

Keywords activated carbon developed from sewage sludge (ACSS), reactive brilliant red K-2BP, dye adsorption, adsorption kinetics, adsorption thermodynamics

1 Introduction

Sewage sludge is the unavoidable byproduct of wastewater treatment and the sludge production rate is too high to be disregarded. Without proper treatment and disposal, it will cause a secondary pollution problem for the environment [1]. Activated carbon is a highly effective adsorbent that is extensively used for air and drinking water purification and is increasingly applied in industrial wastewater treatment. However, activated carbon adsorption remains an expensive treatment process due to the non-renewable and relatively expensive starting materials for activated carbon [2].

Many researchers have recognized sewage sludge as a resource with much potential for beneficial reuse other than direct disposal. Taking into account factors such as the high carbon content of the sewage sludge and the presence of volatile components, we considered that sewage sludge is suitable for the production of activated carbon. This measure allows sludge producers to offset their increasing sludge disposal cost against the cost of activated carbon production while saving non-renewable natural resources, disposing sewage sludge and producing a valuable product with potential applications in pollution control [3].

Therefore, in this study, the activated carbon was prepared from sewage sludge of the municipal wastewater treatment plant by chemical activation (activation reagent is $ZnCl_2$). Moreover, the activated carbon developed from sewage sludge (ACSS) was used for adsorption of dye (reactive brilliant red K-2BP). Based on the evaluation of basic characteristics of the ACSS, the emphasis of this study was placed on the adsorption properties (including kinetics and thermodynamics) of the reactive brilliant red K-2BP on the ACSS.

2 Experimental

2.1 Main experimental materials

The dewatered sewage sludge with total solid of 24.97% was obtained from Jinan Sewage Treatment Plant in Shandong Province, China. It contained 22.63% of total organic carbon, 52.27% of volatile matter and thermal energy of 14.842 $MJ \cdot kg^{-1}$ on a dry basis, respectively. It was found that the sewage sludge possessed a high value for reuse.

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Reactive brilliant red K-2BP was the dye used as an adsorbate. The molecular structure, molecular formula, molecular weight and maximum absorbance wavelength (λ_{max}) of reactive brilliant red K-2BP are listed in Table 1.

2.2 Preparation of activated carbon from sewage sludge [4,5,6]

The sludge sample was first dried at 105°C for 24 h in an oven, crushed, and sieved through a 100-mesh sieve (particle diameter < 150 μm). Then, the sample was impregnated into a $ZnCl_2$ solution of 25 % (mass fraction) with the mass ratio of 1:2 (sludge sample: $ZnCl_2$ solution), stirred thoroughly until well mixed, and left to stand for 24 h at room temperature. After the supernatant liquid was removed, the sample was dried at 105°C for 24 h. The chemically loaded sample was subsequently pyrolyzed in a tubular furnace in N_2 atmosphere at 550°C for 15 min. After cooling in an N_2 atmosphere, the product was acid-washed with HCl solution to reclaim $ZnCl_2$ and then washed repeatedly with distilled water to remove all traces of the acid. Finally, the activated carbon was dried at 105°C for 24 h, crushed, and sieved through a 100-mesh sieve for use.

2.3 Characterization of activated carbon developed from sewage sludge

The iodine number of the ACSS was estimated according to the "Standard test method for granular activated carbon from coal — Determination of iodine number" (GB/T 7702.7-1997). The specific surface area and the pore structure of the ACSS were determined by an Automated Surface Area and Pore Size Analyzer (Quantachrome Quadrasorb SI) based on nitrogen adsorption principle at 77.3 K. The surface physical morphology of the ACSS was visualized by Scanning Electron Microscopy (Hitachi S-520 SEM).

To measure the contents of heavy metals leached from the ACSS, the leachate of the ACSS was obtained according to the "Test method standard for leaching toxicity of solid wastes — Horizontal vibration extraction procedure" (GB 5086.2-1997). The contents of seven selected heavy metals in the leachate were examined by Inductively Coupled Plasma Atomic Emission Spectroscopy (Thermo Electron IRIS Intrepid II XSP ICP-AES, for Pb, As, Cu and Zn) and Atomic Absorption Spectrophotometer (Hitachi 180-80 polarized zeeman AAS, for Cr, Cd and Ni).

2.4 Adsorption experiments

A certain weight of ACSS was added to 50 mL of reactive brilliant red K-2BP dye solution at a certain dye concentration in a 100 mL conical flask. The mixture was agitated in a thermostatic mechanical shaker with the frequency of 140 $r \cdot min^{-1}$ for a specific time at a certain temperature. After the adsorption period, the mixture was separated through a centrifuge. The separated solution was analyzed for the residual dye concentration using a spectrophotometer (Spectrum WFZ75 UV/Vis) by measuring the light absorbance at the maximum absorbance wavelength of dye. The efficiency of decoloration and the amount of dye adsorbed were calculated using the Eq. (1) and Eq. (2) respectively:

$$\eta = \left(1 - \frac{c_e}{c_0}\right) \times 100\% \quad (1)$$

$$q = \frac{(c_0 - c_e)V}{m} \quad (2)$$

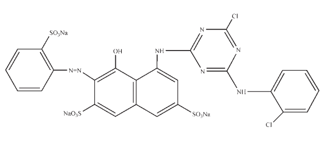
where η is the efficiency of decoloration; q is the amount of dye absorbed ($mg \cdot g^{-1}$); c_0 is the initial dye concentration ($mg \cdot L^{-1}$); c_e is the final (or equilibrium) dye concentration ($mg \cdot L^{-1}$); V is the volume of dye solution (L); m is the weight of ACSS (g).

3 Results and discussion

3.1 Characteristics of activated carbon developed from sewage sludge

The iodine number (I), rate of achievement (r), BET specific surface area (S_B) were based on the Brunauer-Emmet-Teller equation. The micropore surface area (S_m) was based on the t-method. The micropore volume (V_m) was based on the t-method. These, as well as the total pore

Table 1 The properties of reactive brilliant red K-2BP

dye	molecular structure	molecular formula	molecular weight/$g \cdot mol^{-1}$	λ_{max}/nm
reactive brilliant red K-2BP		$C_{25}H_{14}Cl_2N_7Na_3O_{10}S_3$	808.48	533

volume (V_t) for pores with diameter (D) less than 501.64 nm, and average pore diameter (D_p) of the ACSS, are presented in Table 2. Figs. 1 and 2 show the SEM photographs of the ACSS in 500 and 12 000 times magnification, respectively. Table 3 shows the contents of seven selected heavy metals in the leachate of the ACSS.

Table 2 The characteristics of ACSS

I/ mg·g^{-1}	r/%	S_B/ m^2·g^{-1}	S_m/ m^2·g^{-1}	V_m/ cm^3·g^{-1}	V_t/ cm^3·g^{-1}	D_p/nm
326	51.31	298.0	164.1	0.058	0.173	2.318

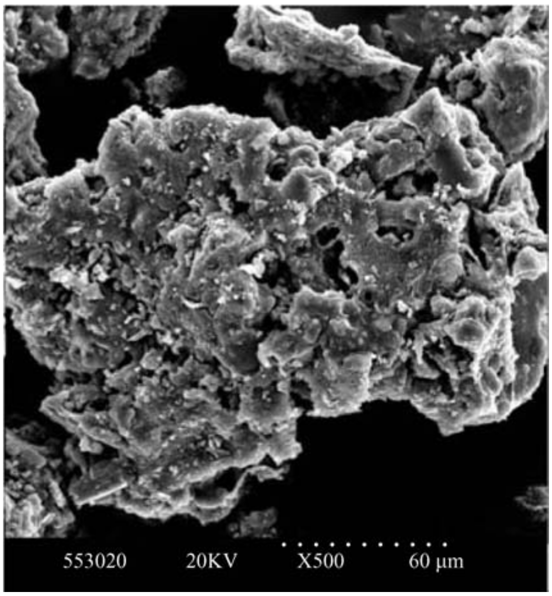


Fig. 1 SEM photograph of the ACSS (× 500)

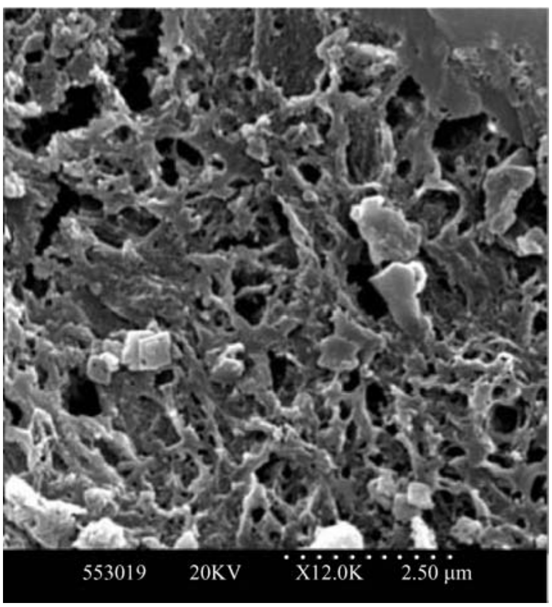


Fig. 2 SEM photograph of the ACSS (× 12000)

Table 3 The contents of heavy metals in the leachate of the ACSS

metals	contents measured/ (mg·L^{-1})	maximum contents allowed/(mg·L^{-1}) (GB 5085.3-1996)
Pb	0.0371	3.00
Cd	0.0000	0.30
Cr	0.0000	10.0
Cu	0.0418	50.0
Zn	21.300	50.0
Ni	0.3100	10.0
As	0.1478	1.50

As seen from Table 2 and Figs. 1 and 2, the ACSS possessed a relatively remarkable iodine number and BET surface area, and it did show rough areas on the surface, on which pores of different sizes and different shapes could be observed. These results highlighted the remarkable adsorption capability of the ACSS. Furthermore, the micropores ($D < 2$ nm) only contributed 55% of the surface area and 34 % of the pore volume to the ACSS . The residual surface area and pore volume were contributed by the mesopores (2 nm $< D <$ 50 nm) and the macropores ($D >$ 50 nm). It indicated that the ACSS had a more porous structure than the commercial activated carbon which was generally microporous. Further, the average pore diameter was 2.318 which shows the mesoporous property of the ACSS. Therefore, the ACSS, which had a more porous structure, is more suitable for the adsorption of large molecular weight compounds, such as dyes, than the commercial activated carbon which mainly has a microporous structure.

The possible release of hazardous heavy metals during use is a major concern with the ACSS. As seen from Table 3, the contents of seven heavy metals in the leachate were all less than the maximum contents allowed in the "Identification standard for hazardous wastes — Identification for extraction procedure toxicity" (GB 5085.3-1996). This result indicated that the ACSS was safe in use for water treatment. It was also found that the content of Zn was high, undoubtedly because of the inadequate reclamation of $ZnCl_2$ in the acid-washing procedure. Therefore, it is suggested that the acid-washing duration should be further increased to remove the residual Zn adequately.

3.2 Effect of adsorbent amount

At room temperature, for 50 mg·L^{-1} of reactive brilliant red K-2BP dye solution, the initial pH value of the solution and the 120 min adsorption time were fixed. The effect of adsorbent (ACSS) amount on the efficiency of decoloration was studied (Fig. 3). As seen from Fig. 3, the efficiency of decoloration increased with the increase of adsorbent amount. The efficiency of decoloration reached 90 % at 4 g·L^{-1}.

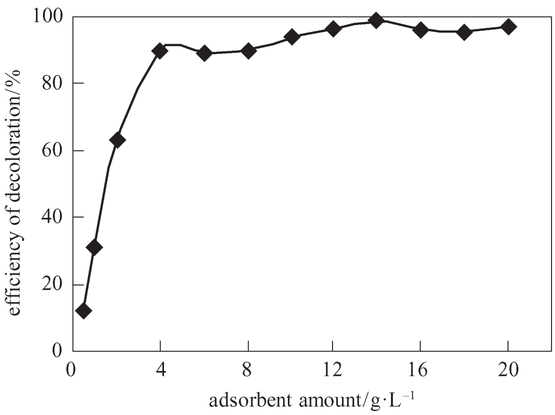


Fig. 3 The effect of adsorbent amount on efficiency of decoloration for adsorption of reactive brilliant red K-2BP on the ACSS

3.3 Effect of adsorption time

At room temperature, for every 50 mg·L^{-1} of reactive brilliant red K-2BP dye solution, the initial pH value of the solution and 4 g·L^{-1} of adsorbent amount were fixed and the effect of adsorption time on the efficiency of decoloration was studied (Fig. 4). As seen from Fig. 4, the efficiency of decoloration increased with an increase in adsorption time. Adsorption reached equilibrium and the efficiency of decoloration reached 89% at 60 min.

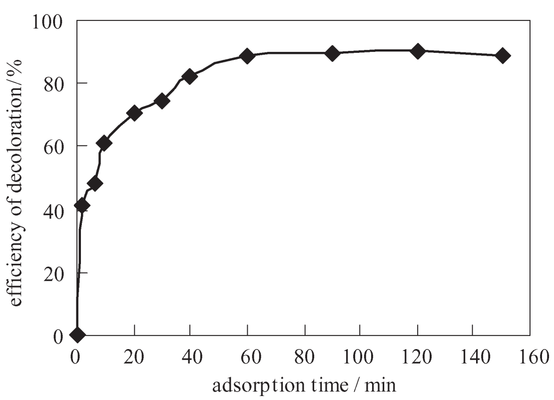


Fig. 4 The effect of adsorption time on efficiency of decoloration for adsorption of reactive brilliant red K-2BP on the ACSS

3.4 Effect of pH value

At room temperature, for every 50 mg·L^{-1} of reactive brilliant red K-2BP dye solution, 4 g·L^{-1} of adsorbent amount and 60 min of adsorption time were fixed and the effect of pH value on the efficiency of decoloration was studied (Fig. 5). As seen from Fig. 5, in the range of 2–10, the effect of pH value on the efficiency of decoloration was negligible, and the efficiency of decoloration decreased slightly with the increase of pH value. However, the efficiency of decoloration decreased sharply with increasing pH value from 10.

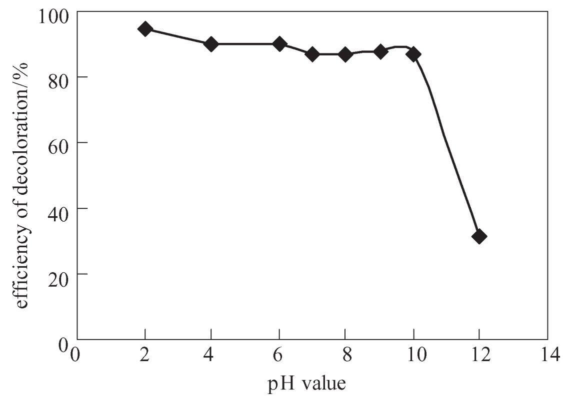


Fig. 5 The effect of pH value on efficiency of decoloration for adsorption of reactive brilliant red K-2BP on the ACSS

3.5 Adsorption kinetics

Based on the data of Fig. 4 (efficiency of decoloration vs. adsorption time), a two-step kinetics rate equation and a pseudo-second-order kinetics equation were selected to predict the adsorption kinetics of reactive brilliant red K-2BP on the ACSS.

3.5.1 Two-step kinetics rate equation [7]

Two-step kinetics rate equation is described as:

$$q_t = q_e - A_1 e^{-k_1 t} - A_2 e^{-k_2 t} \quad (3)$$

where q_t and q_e are the amounts of dye absorbed at time t and equilibrium respectively (mg·g^{-1}); k_1 and k_2 are the kinetics rate constants associated with two different adsorption kinetics steps; A_1 and A_2 are their pre-exponential amplitude terms at two steps.

Figure 6 displays the plot of the two-step kinetics rate equation by non-linear simulation for adsorption of the Reactive Brilliant Red K-2BP on the ACSS. The derived kinetic parameters are given in Table 4. It could be seen that, according to correlation coefficient R^2 values, the two-step kinetics rate equation generated a good fit to the adsorption process.

It could also be observed from Table 4 that the rate constant k_2 was much smaller than k_1. At the first step, the Reactive Brilliant Red K-2BP dye molecules were rapidly adsorbed on the active spots of the ACSS surface with random orientation. The surface of the ACSS became asymmetrically crowded with dye molecules. Then at the second step, the crowded dye molecules tended to be directionally arrayed. Such orientation change could result in the creation of more vacant spaces for further adsorption of dye molecules from the solution

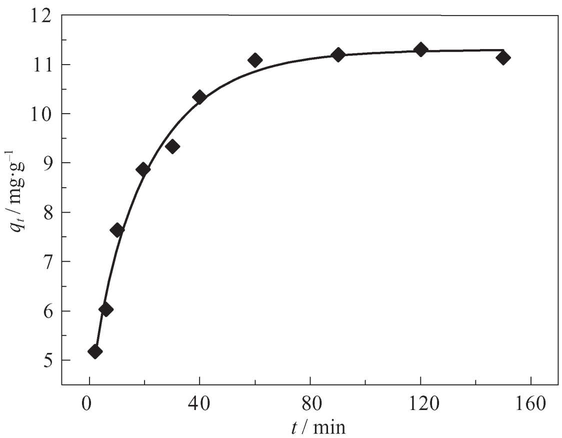


Fig. 6 The plot of two-step kinetics rate equation for adsorption of reactive brilliant red K-2BP on the ACSS

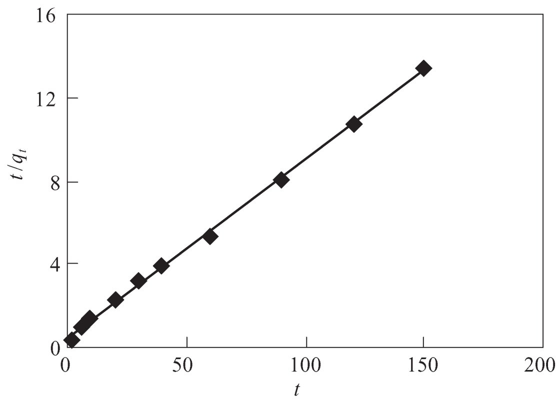


Fig. 7 The plot of pseudo-second-order kinetics equation for adsorption of reactive brilliant red K-2BP on the ACSS

to the surface. However, compared with the first step, the adsorption process at the second step was relatively much slower indeed.

3.5.2 Pseudo-second-order kinetics equation [8,9]

Pseudo-second-order kinetics equation is described as:

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

where k is the pseudo-second-order rate constant (g·mg^{-1}min^{-1}).

Figure 7 shows the plot of the pseudo-second-order kinetics equation by linear simulation for adsorption of reactive brilliant red K-2BP on the ACSS. The derived kinetic parameters are given in Table 4. As seen from Table 4, according to the correlation coefficient R^2 values, pseudo-second-order kinetics equation did show good applicability for the adsorption process.

3.6 Adsorption thermodynamics

3.6.1 Adsorption isotherm

The adsorption of reactive brilliant red K-2BP on the ACSS was studied at 20°C, 30°C, 40°C to determine the adsorption isotherms (Fig. 8). As seen in Fig. 8, the three isotherms all belonged to I [10]. Furthermore, the Langmuir isotherm equation and the Freundlich isotherm equation were selected to describe the adsorption isotherm.

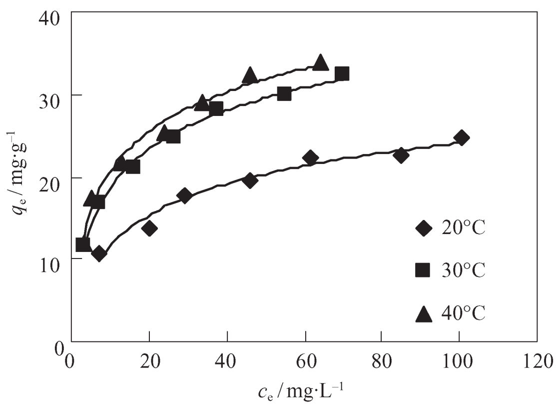


Fig. 8 Adsorption isotherms of reactive brilliant red K-2BP on the ACSS

Langmuir isotherm equation is represented as follows:

$$\frac{c_e}{q_e} = \frac{1}{bQ_m} + \frac{c_e}{Q_m} \tag{5}$$

where b is the Langmuir constant related to the affinity of binding sites; Q_m is the maximum adsorption capacity (mg·g^{-1}).

The Freundlich isotherm equation is represented as follows:

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

where K and n are all Freundlich constants. K is roughly a measure of the adsorption capacity and $1/n$ is an indication of adsorption intensity.

The Langmuir and Freundlich isotherms for adsorption of reactive brilliant red K-2BP on the ACSS in terms

Table 4 Kinetic parameters for adsorption of reactive brilliant red K-2BP on the ACSS

two-step kinetics rate equation					pseudo-second-order kinetics equation			
equation	q_e/mg·g^{-1}	k_1	k_2	R^2	equation	q_e/mg·g^{-1}	k	R^2
$q_t = 11.30193 - 1.04329e^{-0.18637t} - 5.97621e^{-0.04341t}$	11.3019	0.1864	0.0434	0.9902	$t/q_t = 0.0859t + 0.424$	11.6414	0.0174	0.9989

of Eq. (5) and Eq. (6) are shown in Figs. 9 and 10 and the values of Q_m, b, K, n, and the corresponding correlation coefficient R^2 are summarized in Tables 5 and 6. Based on the correlation coefficient R^2 values, the experimental data yielded good linear plots with both the Langmuir isotherm and the Freundlich isotherm. The Langmuir isotherm did show better applicability for the adsorption, comparatively. In addition, the maximum adsorption capacity (Q_m), and K which was roughly a measure of the adsorption capacity all increased with increasing temperature indicating that the adsorption of reactive brilliant red K-2BP on the ACSS was an endothermic process and that enhancing temperature was beneficial to the adsorption. Furthermore, the values of n were in the range of 1 to 10 which meant that good adsorption or favorable adsorption occurred.

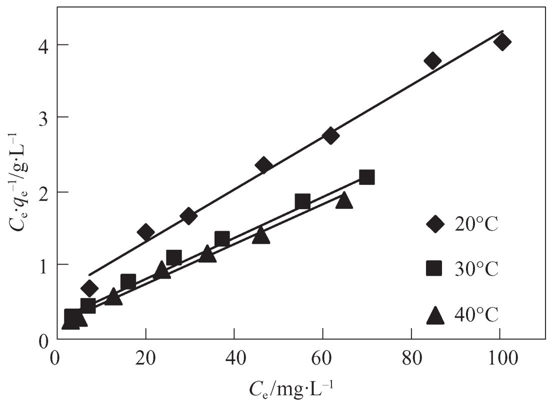


Fig. 9 Langmuir adsorption isotherms of reactive brilliant red K-2BP on the ACSS

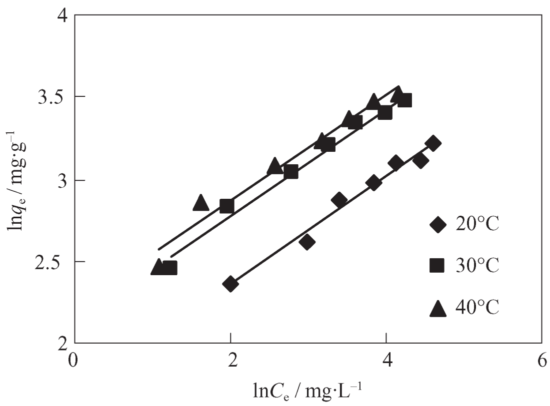


Fig. 10 Freundlich adsorption isotherms of reactive brilliant red K-2BP on the ACSS

Table 5 Langmuir adsorption isotherm parameters of reactive brilliant red K-2BP on the ACSS

T/°C	equation	Q_m/mg·g^{-1}	b	R^2
20	$c_e/q_e = 0.0356c_e + 0.6101$	28.0899	0.0584	0.9887
30	$c_e/q_e = 0.0280c_e + 0.2652$	35.7143	0.1056	0.9933
40	$c_e/q_e = 0.0268c_e + 0.2151$	37.3134	0.1246	0.9916

Table 6 Freundlich adsorption isotherm parameters of reactive brilliant red K-2BP on the ACSS

T/°C	equation	K	n	R^2
20	$\ln q_e = 0.3267\ln c_e + 1.7112$	5.5356	3.0609	0.9784
30	$\ln q_e = 0.3282\ln c_e + 2.1145$	8.2854	3.0469	0.9847
40	$\ln q_e = 0.3221\ln c_e + 2.2242$	9.2461	3.1046	0.9674

3.6.2 Thermodynamic parameters

The enthalpy changes (ΔH) can be estimated by the Clausius-Clapeyron equation [11]:

$$\ln c_e = \frac{\Delta H}{RT} + c \quad (7)$$

where ΔH is the enthalpy of adsorption (kJ·mol^{-1}); R is the gas constant; T is temperature (K); c is constant. The plots of lnc_e-1/T at different amounts of dye adsorbed (10 mg·g^{-1}, 20 mg·g^{-1} and 30 mg·g^{-1}) are shown in Fig. 11. The ΔH values are determined from the slope of each linear plot.

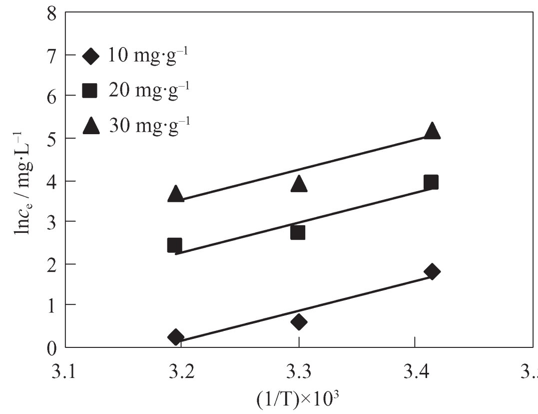


Fig. 11 The plots of lnc_e-1/T for adsorption of reactive brilliant red K-2BP on the ACSS

Since the Freundlich isotherm did show good applicability for the adsorption of reactive brilliant red K-2BP on the ACSS, the free energy of adsorption (ΔG) can be obtained from the following equation [12]:

$$\Delta G = -nRT \quad (8)$$

where ΔG is the free energy of adsorption (kJ·mol^{-1}); n is the Freundlich constant.

Thus the entropy changes (ΔS) can be calculated from the Gibbs-Helmholtz equation:

$$\Delta S = (\Delta H - \Delta G)/T \quad (9)$$

where ΔS is the entropy of adsorption (J·mol^{-1}·K^{-1}).

The values of ΔH, ΔG and ΔS at different amounts of dye adsorbed (10 mg·g^{-1}, 20 mg·g^{-1} and 30 mg·g^{-1})

obtained from Eqs. (7), (8) and (9) are given in Table 7. It was found that the values of ΔH were all positive, which conformed to the endothermic nature of the adsorption process of reactive brilliant red K-2BP on the ACSS. In a liquid solution, the thermodynamic parameters estimated from adsorption isotherms are the total values of two separate processes, i.e., the adsorption of the adsorbate and the desorption of the solvent. Furthermore, the adsorption of the adsorbate is generally exothermic, whereas the desorption of solvent is generally endothermic. In the ACSS-dye system, when a dye molecule was adsorbed on the ACSS surface, a large number of water molecules were desorbed simultaneously since the molecular size of dye was quite larger than that of water. Accordingly, the heat absorbed for the desorption of the abundant water molecules was higher than that released for adsorption of a few dye molecules. As a result, the entire process became endothermic.

Table 7 Thermodynamic parameters for adsorption of reactive brilliant red K-2BP on the ACSS

q/ mg·g^{-1}	ΔH/ kJ mol^{-1}	ΔG/kJ mol^{-1}			ΔS/J mol^{-1} K^{-1}		
		293 K	303 K	313 K	293 K	303 K	313 K
10	60.15	−7.46	−7.68	−8.08	230.76	223.86	218.00
20	59.02				226.89	220.13	214.38
30	58.36				224.63	217.94	212.27

As also seen from Table 7, ΔH decreased slightly with increasing amount of dye adsorbed (q). At low q, the interaction between the adsorbate and the adsorbent was mainly the immediate interaction. With the increase of q, the vacant space on the surface of the ACSS decreased. Meanwhile, the interaction between the adsorbate and the adsorbent was gradually replaced by the interaction between the adsorbate adsorbed on the ACSS and the adsorbate in the solution. As a result, ΔH decreased with the increase of q.

The values of ΔS were all positive. The adsorption of dye molecules was accompanied by a decrease in entropy as the dye molecules from the disturbed liquid state found a structured arrangement on the ACSS surface. Whereas, the desorption of water molecules brought on an increase in entropy as the water molecules were released from the regular arrangement on the ACSS surface to a chaotic state in the aqueous solution. As above, the increase in entropy for desorption of the abundant water molecules got ahead of the decrease in entropy for the adsorption of a few dye molecules. Consequently, the entropy changes of entire process became positive.

The values of ΔG were all negative which indicated the spontaneity of the adsorption process of reactive brilliant red K-2BP on the ACSS.

4 Conclusion

The results of this work can be summarized as follows:

1) The iodine number of the ACSS was 326 mg·g^{-1}. The rate of achievement was 51.31%. The BET surface area was 298 m^2·g^{-1}, and the contents of heavy metals in the leachate didn't exceed the limit. The ACSS, which was mesoporous, possessed an opened porous structure.

2) The adsorption kinetics of reactive brilliant red K-2BP on the ACSS was consistent with the two-step kinetics rate equation and pseudo-second-order kinetics equation.

3) The adsorption was favorable. Experimental data yielded good linear plots with both the Langmuir isotherm equation and the Freundlich isotherm equation. Comparatively, the Langmuir isotherm equation did show better applicability for adsorption.

4) The adsorption was an endothermic (enthalpy $\Delta H > 0$) and spontaneous (free energy $\Delta G < 0$) process, accompanied by an increase in entropy ($\Delta S > 0$). Increased temperature was beneficial to the adsorption.

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