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Analysis of adsorption characteristics of 2,4-dichlorophenol from aqueous solutions by activated carbon fiber

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Abstract

In this study experiments were conducted to investigate the adsorption of 2,4-dichlorophenol (2,4-DCP) by activated carbon fiber (ACF) activated by static air. With the results of batch experiments at various temperatures, the adsorption isotherms, kinetics and thermodynamics of this adsorption process were evaluated. Four adsorption isotherm models, Langmuir, Freundlich, Redlich-Peterson and Toth equations, were used to fit the experimental data and the results reveal that the adsorption isotherm models fitted the data in the order of: Langmuir > Redlich-Peterson > Toth > Freundlich isotherms. A pseudo second-order adsorption model was better to describe the adsorption data than the pseudo first-order model and the Bangham model at the temperatures tested. The activation energy was calculated to be 40.90 kJ/mol, while the thermodynamic parameters ΔH and ΔS were estimated to be -5.82 kJ/mol and 0.07 kJ/(mol K), respectively.

Keywords: 2,4-DCP; ACF; Adsorption; Isotherm; Kinetics; Thermodynamics

1. Introduction

Chlorophenols are mainly produced in chemical industries, such as petroleum refineries, plastics, pharmaceuticals, pesticide, and wood preservation. As one type of the most hazardous materials [1,2], they are carcinogenic, mutagenic and resistant to biodegradation, and thus have to be decomposed before discharging into receiving waters, in order to avoid the biomagnified toxicity to aquatic flora and fauna through various food chains. Many efforts have been made for the physicochemical and/or biological treatments of chlorophenol-rich wastewaters. For instance, they could be removed effectively through adsorption process by using a variety of adsorbents. Among various adsorbents, activated carbon is most commonly used in wastewater treatment, attributed to its vast surface area and great affinity for organics [3]. Activated carbon fiber (ACF) is a new form of activated carbon and has been developed in recent years following powdered activated carbon (PAC) and granular activated carbon (GAC). Because it is used in the form of fabric, the handling of ACF is more facilitated than that of PAC [4]. On

0304-3894/\$ - see front matter © 2006 Elsevier B.V. All rights reserved. doi:10.1016/j.jhazmat.2006.10.003 the other hand, compared with conventional GAC, it has the advantages of greater adsorption–desorption rate, much vaster surface area, faster equilibrium rate and higher fluid permeability [5–9].

A large number of studies have been carried out to investigate the adsorption of chlorophenols by activated carbon [10–14]. Carrott et al. [11] studied the adsorption equilibrium of phenol, 4-nitrophenol, 4-chlorophenol and 2-chlorophenol onto commercial activated carbon before and after the oxidation of activated carbon at various pHs. Khan et al. [14] investigated the adsorption isotherms of phenol, *p*-chlorophenol, and *p*-nitrophenol from aqueous solutions using activated carbon at different temperatures. They also compared the influences of different adsorbents, sorbate concentration and solution pH on the adsorption efficiency. However, at the present time little information is available concerning the adsorption of chlorophenols onto ACF, a new excellent adsorbent.

Therefore, the objective of this study was to investigate the adsorption of 2,4-dichlorophenol (2,4-DCP), a typical chlorophenol, onto ACF from aqueous solutions, and to evaluate the adsorption efficiency of ACF as a new type of adsorbent. In this work, four isotherm models, Langmuir, Freundlich, Redlich-Peterson and Toth equations, were compared for describing the adsorption isotherms. In addition, the

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adsorption kinetics was explored using three kinetic models. With the experimental data of adsorption isotherms and kinetics, the thermodynamics of the adsorption process was also analyzed.

2. Experimental

2.1. Adsorbent

The ACF used in this study was prepared at a yield of 33.0% using a spinning polyvinyl alcohol (PVA) as precursor. The preparation process was described in our previous paper [15]. The ACF used in this work was activated by static air at 900 °C for 1 h. The textual properties of ACF are given in Table 1. In addition to the micropores, there were many mesopores with a diameter around 3.90 nm in the ACF, which was beneficial to facilitating the transportation of 2,4-DCP in the adsorption process. The ACF was washed in boiling water for three times to remove impurities and then dried at 120 °C for 2 h prior to adsorption.

2.2. Chemicals

Continuous filament yarns of raw wet spinning PVA fiber was purchased from Hunan Xiangwei Co., China. The chemicals used in this study, i.e., diammonium phosphate and 2,4-dichlorophenol (2,4-DCP), were purchased from Shanghai Chemical Reagent Co., China. All the reagents were of analytical reagent grade and were used without further purification.

Table 1

Textural properties of the ACFs used in this work

Item	Value
Surface area (m ² /g)	702
Pore volume (cm^3/g)	0.280
Average micropore diameter (nm)	0.70
Average mesopore diatmeter (nm)	3.90
Adsorption capacity for iodine (mg/g)	1678
Adsorption capacity for methylene blue (mg/g)	184

2.3. Adsorption experiments

Adsorption experiments were conducted in flasks by allowing an accurately weighted amount of ACF to reach equilibrium with 100-mL 2,4-DCP solution at temperatures of 293, 303, 313 and 323 K. These flasks were then agitated in a temperaturecontrolled shaker at 150 rpm. At equilibrium, the supernatant were sampled for analysis. The experiments of adsorption kinetics were carried out at 283, 293, 303, 313 and 323 K. Samples were analyzed at given time intervals. Each run of the experiments was replicated at least three times.

2.4. Analysis

The concentration of 2,4-DCP was determined using a UV–Vis spectrophotometer (UV751GD, Shanghai Analytical Instrument Co., China) at an absorbance wavelength of 280 nm. The surface area and pore structure of the ACF were determined by the N₂-BET method. The measurements were performed at 77.4 K with a volumetric adsorption analyzer (OMNISORP 100 CX, Coulter, USA) in relative pressures of 10^{-6} to 1. All samples were degassed for 3 h at 573 K prior to the vacuum volumetric analysis.

3. Results and discussion

3.1. Adsorption isotherms

In order to understand the adsorption mechanisms of 2,4-DCP onto ACF, four adsorption isotherm models, Langmuir, Freundlich, Redlich-Peterson and Toth, were used to fit the adsorption experimental results. The Langmuir model is usually used with an ideal assumption of an entirely homogeneous adsorption surface, whereas the Freundlich model is appropriate for a heterogeneous surface. The Redlich-Peterson and Toth isotherm models, the combinations of the Langmuir and Freundlich, are applicable to describing microporous adsorption. These isotherm equations are given in Table 2.

The Langmuir and Freundlich isotherms, which have two parameters, could be respectively linearized as follows:

$$\frac{C_{\rm eq}}{q_{\rm eq}} = \frac{1}{k_{\rm L}Q_{\rm max}} + \frac{C_{\rm eq}}{Q_{\rm max}} \tag{1}$$

Table 2	2
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Isotherm models adopted in this work and their parameters

T	NC 11	
Isotherm	Model	Parameters
Langmuir	$q_{\rm e} = \frac{Q_{\rm max}k_{\rm L}C_{\rm e}}{1 + k_{\rm L}C_{\rm e}}$	C_{e} : equilibrium liquid phase concentration (mg/L); Q_{e} : equilibrium adsorption capacity; k_{L} : constant of Langmuir (L/mg); Q_{max} : the maximum adsorption capacity (mg/g)
Freundlich	$q_{\rm e} = k_{\rm F} C_{\rm e}^{1/n}$	$k_{\rm F}$: constant of Freundlich (mg ^{1-1/n} g ^{1/n} L ^{1/n}); <i>n</i> : constant of Freundlich
Redlich-Peterson	$q_{\rm e} = \frac{k_{\rm RP}C_{\rm e}}{1 + p_{\rm e}C_{\rm e}^{\rm g}}$	k_{RP} : constant of Redlich-Peterson (L/g); p_e : constant of Redlich-Peterson (L/mg) ^g ; g: constant of Redlich-Peterson
Toth	$q_{\rm e} = \frac{AC_{\rm e}}{\left(B + C_{\rm e}^D\right)^{1/D}}$	A: constant of Toth (L/g); B: constant of Toth $(mg/L)^D$; D: constant of Toth



Fig. 1. Adsorption isotherms of 2,4-DCP by ACF. Scatters: experimental data at (•) 293 K, (()) 303 K, (**(**)) 313 K, and (()) 323 K. Lines: model fit.

$$\ln q_{\rm eq} = \ln k_{\rm F} + \frac{1}{n} \ln C_{\rm eq} \tag{2}$$

The parameters in the equations above could be graphically determined through linear regression. However, the constants of the Redlich-Peterson and Toth isotherm models, which have three parameters, could only be estimated using non-linear regression. Different adsorption isotherms obtained at various temperatures are illustrated in Fig. 1, and the values of the parameters estimated from the plots along with the correlation coefficients are listed in Table 3. The high regression correlation coefficients were obtained, suggesting that all these adsorption isotherm models were applicable to describing the 2,4-DCP adsorption onto ACF.

As shown in Table 3, the constants Q_{max} and k_{L} in the Langmuir equation decreased with an increase in temperature, indicating that, at lower temperatures, the adsorption density was higher and the sorption energy was lower. This result was in consistent with the experimental observation. The magnitude of the exponent 1/n in the Freundlich model usually gives an indication of the favorability and capacity of the adsorbent/adsorbate system. In these experiments, the *n* values greater than 1 suggest that the 2,4-DCP could be readily adsorbed by ACF at all the temperatures studied. In addition, the values of *n* were all in the range of 1–10, showing that the adsorption was beneficial for 2,4-DCP. As shown in Table 3, the isotherm models fitted to the experimental data in the order of: Langmuir > Redlich-Peterson > Toth > Freundlich.

3.2. Adsorption kinetics

Three kinetic models, pseudo first-order, pseudo secondorder and Bangham models, were used to evaluate the 2,4-DCP adsorption kinetics by ACF at various temperatures, and are presented in Table 4. The linearized form of the pseudo first-order model could be expressed as following:

$$\ln(q_{\rm eq} - q) = \ln q_{\rm eq} - k_{1,\rm ad}t \tag{3}$$

However, to fit Eq. (3) to experimental data, the value of q_{eq} (equilibrium adsorption capacity) had to be pre-estimated by extrapolating the experimental data to $t = \infty$. In addition, in most cases, the first-order rate equation of the Lagergren model is usually applicable over the initial 30–50 min of the adsorption process [16]. The plots of $\ln(q_{eq} - q)$ as a function of adsorption time are shown in Fig. 2a. The linear relationships were observed only for the initial 30-min adsorption, and after this short period the experimental data deviated considerably from the theoretical ones (not shown). The rate constants $k_{1,ad}$ and theoretical values of q_{eq} calculated from the slope and intercept of the linear plots are summarized in Table 5, along with the corresponding correlation coefficients.

The linearized form of the pseudo second-order model could be given as:

$$\frac{t}{q} = \frac{1}{k_{2,\rm ad}q_{\rm eq}^2} + \frac{t}{q_{\rm eq}}$$
(4)

Table 3
Isotherm parameters for 2.4-DCP adsorption by ACF at various temperatures

Isotherm model	Temperature (K)	Мо	Model parameter		R^2
		$k_{\rm L}$		Q_{\max}	
Langmuir	293	0.1	70	372	0.999
•	303	0.1	56	365	0.998
	313	0.14	48	355	0.998
	323	0.12	35	342	0.997
Isotherm model	Temperature (K)	Мо	del parameter		R^2
		$k_{ m F}$		n	
Freundlich	293	55.	1	2.01	0.905
	303	61.4	4	2.22	0.899
	313	60.1	3	2.38	0.897
	323	62.5	8	2.54	0.896
Isotherm model	Temperature (K)	Model parameter		R^2	
		k _{RP}	pe	g	
Redlich-Peterson	293	50.3	0.086	1.10	0.993
	303	42.1	0.071	1.10	0.992
	313	34.6	0.052	1.12	0.981
	323	33.9	0.052	1.13	0.993
Isotherm model	Temperature (K)	Model parame	ter		R^2
		Ā	В	С	
Toth	293	354	22.0	1.48	0.994
	303	357	14.8	1.26	0.990
	313	348	20.3	1.31	0.978
	323	330	32.0	1.48	0.990

By plotting t/q against t for different temperatures, slight lines were obtained (Fig. 2b). The values of the second-order rate constants $k_{2,ad}$ and q_{eq} , determined from the slopes and intercepts of the plots in Fig. 2b, are given in Table 5.

Fig. 2c presents the fitting of the adsorption data to the Bangham equation. The values of the Bangham rate constant k_b , and constant *a* are also shown in Table 5. As shown in Table 5, the second-order rate constant $k_{2,ad}$ increased with increasing temperature. For all the cases, the correlation coefficients for the second-order kinetic model were close to 1.0, and the theoretical values of q_{eq} were consistent with the experimental data. The correlation coefficients for the pseudo first-order kinetics were lower than those of the pseudo second-order one.

The adsorption of adsorbate onto the adsorbent surface from the solution phase occurs in several steps, such as film or external diffusion, pore diffusion, surface diffusion and adsorption on the pore surface [17]. The overall adsorption process may be controlled by one or more steps. The possibility of pore diffusion was explored using the Bangham equation. If the experimental data could be fitted well by this equation, the pore diffusion should be the only rate-controlling step in the adsorption process [17]. As shown in Fig. 2c, the experimental data did not give a good fit to the Bangham model. This implies that the diffusion of 2,4-DCP into the pores of ACF was not the sole rate-controlling step. These results also show that the pseudo second-order adsorption model was better to describe the adsorption data than the pseudo first-order model and Bangham model.

Table 4				
Kinetic models and	their equa	ations use	d in thi	s study

Model	Equation	Parameters
Pseudo first-order	$\frac{\mathrm{d}q}{\mathrm{d}t} = k_{1,\mathrm{ad}}(q_{\mathrm{eq}} - q)$	<i>t</i> : time (min); <i>q</i> : adsorption capacity at time <i>t</i> (mg/g); q_{eq} : equilibrium adsorption capacity; $k_{1,ad}$: rate constant (min ⁻¹)
Pseudo second-order	$\frac{\mathrm{d}q}{\mathrm{d}t} = k_{2,\mathrm{ad}}(q_{\mathrm{eq}} - q)^2$	q_{eq} : equilibrium adsorption capacity; $k_{2,ad}$: rate constant (g/(mg min))
Bangham	$\ln \ln \frac{C_0}{C_0 - qm} = \ln \frac{k_{\rm b}m}{V} + a \ln t$	C_0 : initial concentration of adsorbates (mg/L); <i>m</i> : adsorbent mass per liter of solution (g/L); k_b : constant in Bangham's equation; <i>V</i> : volume of solution (L); <i>a</i> : Bangham constant (<1)



Fig. 2. Adsorption kinetics of 2,4-DCP by ACF (initial DCP concentration: 2 mM). Scatters: experimental data at (\bullet) 293 K, (\bigcirc) 303 K, (\blacktriangle) 313 K, and (\triangle) 323 K. Lines: simulation with (a) pseudo first-order model, (b) pseudo second-order model, and (c) Bangham model.

Table 5
Kinetic parameters for the adsorption of 2,4-DCP by ACF at various temperatures

Kinetic model	Temperature (K)	Kinetic parameters		R^2
		$\overline{k_1}$	$q_{ m eq}$	
Pseudo first-order	283	0.020	318	0.964
	293	0.017	262	0.697
	303	0.032	311	0.951
	313	0.041	280	0.950
	323	0.047	280	0.970
Kinetic model	Temperature (K)	Kinetic parameters		R^2
		$\overline{k_2}$	$q_{ m eq}$	
Pesudo second-order	283	3.92×10^{-5}	338	0.999
	293	7.35×10^{-5}	332	0.999
	303	1.05×10^{-4}	328	0.999
	313	2.20×10^{-4}	328	0.999
	323	3.33×10^{-4}	324	0.999
Kinetic model	Temperature (K)	Kinetic parameters		R^2
		kb	а	
Bangham's equation	283	0.0316	0.293	0.921
	293	0.0455	0.258	0.943
	303	0.0365	0.307	0.828
	313	0.0590	0.259	0.841
	323	0.0554	0.280	0.831



Fig. 3. Arrhenius plot for adsorption of 2,4-DCP by ACF.

3.3. Thermodynamic analysis

The pseudo second-order rate constant can be expressed as a function of temperature by the Arrhenius equation and the activation energy (E_a) can be calculated as follows:

$$k_{2,\mathrm{ad}} = k_0 \exp\left(-\frac{E_a}{RT}\right) \tag{5}$$

$$\ln k_{2,ad} = \ln k_0 - \frac{E_a}{RT}$$
(6)

Fig. 3 shows the linear plot of $\ln k_{2,ad}$ as a function of $10^3/T$ for 2,4-DCP adsorption onto ACF at 283–323 K. The apparent activation energy calculated from the slope of plot was estimated to be 40.90 kJ/mol.

The Gibbs free energy change ΔG , indicating the degree of the spontaneity of the adsorption process, is defined as:

$$\Delta G = -RT\ln K \tag{7}$$

Thermodynamic parameters for the adsorption such as enthalpy change (ΔH) and the entropy change (ΔS) were respectively calculated using Eq. (7) and van't Hoff equation:

$$\ln K = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \tag{8}$$

The *K* value can be obtained from the linearized form of the Langmuir equation. The values of ΔG at different temperatures are listed in Table 6. A plot of ln *K* as a function of 1/T yielded a straight line (Fig. 4). The values of ΔH and ΔS , obtained from the slope and intercept of the plot, are summarized in Table 6.

Table 6 Thermodynamic parameters of 2,4-DCP adsorbed by ACF

T (K)	ΔG (kJ/mol)	ΔH (kJ/mol)	$\Delta S \left(\text{kJ/(mol K)} \right)$	E _a (kJ/mol)
293	-24.92			
303	-25.55			
313	-26.26			
323	-26.85			
		-5.82	0.07	40.90



Fig. 4. van't Hoff plot for the adsorption of 2,4-DCP by ACF.

The negative ΔG values indicate that the adsorption process led to a decrease in Gibbs free energy and that the adsorption process was feasible and spontaneous. The change of Gibbs free energy for the physical adsorption is generally in the range of nil to -20 kJ/mol, and that for the chemical adsorption is in the range of -80 to -400 kJ/mol [18]. The values of ΔG at different temperatures in this study were in the range between those of physical and chemical adsorptions. Thus, the adsorption of 2,4-DCP onto ACF could be considered as a physical adsorption enhanced by the electrostatic effect. On the other hand, the adsorption enthalpy for the physical adsorption is usually in the range of nil to -42 kJ/mol, and that for the chemical adsorption is in the range of -42 to -125 kJ/mol. Hence, based on a ΔH value of -5.82 kJ/mol in this study, the adsorption of 2,4-DCP onto ACF could also be considered as a physical adsorption. In addition, the negative ΔH value implies that the adsorption process was exothermic, which was in good agreement with the experimental observations. The positive value of ΔS suggests that the randomness increased in the liquid/solid interface in the adsorption of 2,4-DCP onto ACF. The adsorbed water molecules, which were displaced by the 2.4-DCP species, gained more translational entropy than that which the 2,4-DCP molecules lost, thus allowing the prevalence of randomness in the system. The increase in entropy of 2,4-DCP adsorption process might be associated with the configuration changes in 2,4-DCP in adsorption process and/or the changes of ACF surfaces caused by adsorption.

3.4. Evaluation of ACF as an adsorbent for 2,4-DCP

Comparisons between the 2,4-DCP adsorption results in this work and those in other relevant studies are summarized in Table 7. In this study, the values of Q_{max} in the Langmuir model, which is an indicator of adsorption capacity, and *n* in Freundlich model, which is an indicator of adsorption intensity, are comparable to those in most of other relevant studies, in which GAC or PAC were used as the adsorbents. On the other hand, compared with GAC and PAC, the diffusion distance in ACF is significantly smaller than that in GAC, and accordingly the overall adsorption rate is usually higher in ACF than in GAC; ACF is usually much

Table 7				
Comparison of 2,4-DCP	adsorption by ACF	in this work and by	GAC or PAC in	other studies

Adsorbents	Equilibrium models	$Q_{\rm max} \ ({\rm mg/g})$	n	Reference
ACF	Langmuir and Freundlich	372 ^a	2.01	This study
PAC	Langmuir	19.1	_b	[19]
R1	Freundlich	_	1.83	[20]
R2	Freundlich	_	1.96	[20]
R0.8	Freundlich	_	2.29	[20]
CG	Freundlich	_	1.86	[20]
PAC	Langmuir	19.2	1.90 ^c	[21]
GAC	Langmuir	157	_	[22]
AP-32	Langmuir	224	_	[22]
AP-35	Langmuir	420	_	[22]
AP-42	Langmuir	339	1.50	[22]
AP-44	Langmuir	320	_	[22]
AP-45	Langmuir	595	_	[22]
AP-55	Langmuir	533	_	[22]
0.5 (KOH/char)	Langmuir and Freundlich	217.8	9.71	[23]
1.0 (KOH/char)	Langmuir and Freundlich	288.7	13.3	[23]
2.0 (KOH/char)	Langmuir and Freundlich	552.5	6.54	[23]
3.0 (KOH/char)	Langmuir and Freundlich	728.3	2.29	[23]
Steam	Langmuir and Freundlich	314.7	4.13	[23]

^a At temperature of 293 K.

^b Not available.

^c At concentration of 10 mg/L.

easier to be handled and prepared than PAC, because of its fabric form [4]. The experimental results above demonstrate that the ACF activated by air was a highly efficient and cost-effective adsorbent for removal of phenolic compounds from wastewater.

4. Conclusions

The PVA-ACF activated by static air was used to adsorb 2,4-DCP from aqueous solution. Four isotherm equations of Langmuir, Freundlich, Redlich-Peterson and Toth models were used to fit the experimental data, and they were found to fit the data in the order of: Langmuir > Redlich-Peterson > Toth > Freundlich. The adsorption kinetics was found to follow the pseudo secondorder. The apparent activation energy was calculated to be 40.90 kJ/mol. The negative values of ΔG indicate the feasibility and spontaneity of the adsorption process. The thermodynamic parameters, ΔH and ΔS , were estimated to be -5.82 kJ/mol and 0.07 kJ/(mol K), respectively, suggesting that the adsorption was exothermic. The positive value of the entropy implies the increased randomness at the solid/solution interface in the adsorption of 2,4-DCP onto ACF.

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