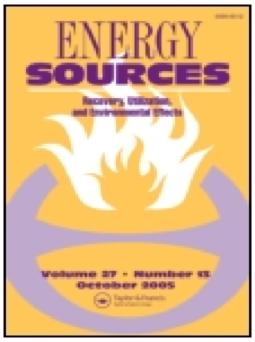

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Utilization of pyrolytic char derived from bamboo chips for furfural removal: Kinetics, isotherm, and thermodynamics

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ABSTRACT

Bamboo charcoal obtained from the pyrolysis of bamboo chips was used to remove furfural, a representative fermentation inhibitor in hydrolyzates or pyrolysis oil. Kinetics, isotherm, and thermodynamic calculations were performed. The adsorption process can be well depicted by Ho's pseudo-second-order model. The particle diffusion of homogeneous particle diffusion model (HPDM) and shrinking core model (SCM) was found to be the controlling step. Isotherm analysis indicated the adsorption feature took place by a nonideal adsorption. Thermodynamic calculation suggested that $\Delta H^\circ > 0$, $\Delta G^\circ < 0$, $\Delta S^\circ > 0$, and E_a value was 2.02 kJ/mol. The comparison results demonstrate bamboo charcoal is a promising adsorbent for furfural removal.

KEYWORDS

Adsorption kinetic; furfural; pyrolytic char; thermodynamic

1. Introduction

Bioethanol, a renewable biofuel, has drawn great attentions nowadays because of the shortage of petroleum, the increasing global energy demand, and the large amounts greenhouse gas emission (Balat, 2009). The lignocellulosic biomass can be used as a potential feedstock for bioethanol production (Demirbas, 2006), because it mainly consists of hemicellulose and cellulose. The hemicellulose and cellulose are the structures of polymers of five/six carbon chain sugar (Ranjan et al., 2009). These polysaccharides could be decomposed into monosaccharide by enzymatic/acid hydrolysis or by the pyrolysis process (Li and Zhang, 2004). The monosaccharide can be used as the carbon sources for microbial fermentation. However, many kinds of inhibitor compounds will be generated in the transformation process of biomass, including aldehydes, acids, and so on (Parawira and Tekere, 2011), and furfural is one of the representative fermentation inhibitors that strongly inhibits the growth of microorganisms (Xiros et al., 2011). Thus, furfural must be removed first before bioethanol production.

Several attempts have been made for the removal of furfural along with some other fermentation inhibitors from the pyrolysis oil or hydrolyzates, and the adsorption method has proved to be a reliable technology (Ranjan et al., 2009). In the authors' previous study, a cheap pyrolytic char derived from bamboo chips (named bamboo charcoal) was used to remove furfural, and it was found that charcoal could selectively adsorb furfural despite the presence of sugars due to the π-π dispersion interaction between furfural and the bamboo charcoal (Li et al., 2013). However, the adsorption kinetics and isotherm along with the adsorption thermodynamic process of furfural in detail by this pyrolytic char were still not comprehensive. The kinetics, isotherm, and thermodynamic parameters are very important for the adsorption design, as some of them may provide an insight into understanding the mechanism of the adsorption process. Benamor et al. (Benamor et al., 2008) studied the kinetic process of cadmium ions onto XAD-7 and found the rate-limiting step was particle diffusion. He also quantitatively calculated the

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diffusion coefficient of the adsorption. Hameed and Rahman (Hameed and Rahman, 2008) deduced that the adsorption of phenol on activated carbon was a monolayer adsorption based on the fitting result of the isotherm. Qiu et al. (Qiu et al., 2012) investigated the thermodynamic phenomenon of the adsorption of *p*-xylene onto activated carbon, and found the process was endothermic, spontaneous, and physics. Because of the specific structure of pyrolytic char derived from bamboo chips that was quite different from activated carbon or polymers, the kinetic, isotherm, and thermodynamic mechanisms on this pyrolytic char still need to be studied. Furthermore, the accurate adsorption capacity of furfural onto this charcoal remains unveiled.

In this paper, the adsorption kinetics and isotherm of bamboo charcoal for the adsorptive removal of furfural are investigated. The rate-limiting step is determined by the homogeneous particle diffusion model (HPDM) as well as the shrinking core model (SCM). Thermodynamic analysis is also performed. The basic data obtained will be very important for further adsorption design. In addition, the results are compared with those of the commercial activated carbons and other adsorbents to further shed some light on the possibility of using bamboo charcoal as the adsorbent for furfural removal, which may partially replace the commercial activated carbon at a relatively much lower cost and for better waste reutilization.

2. Materials and methods

2.1. Adsorbent and adsorbate

The bamboo charcoal used here was prepared at 823 ± 20 K in a fluidized bed reactor using bamboo chips as the feedstock. The physical/chemical properties of the bamboo charcoal have been provided in Ref. (Li et al., 2013). The charcoal of 0.25–0.83 mm particle size was collected for later usage.

The representative fermentation inhibitor solution was prepared by dissolving a precise quantity of furfural (analytical grade; >99% purity) into deionized water. The concentrations of furfural were 5–20 g/L, similar to the inhibitor concentration in pyrolysis oil. Furfural was purchased from Jin Shan Ting Xin Chemical Ind. Ltd. (Shanghai, China) and it was used without any further purification.

2.2. Adsorption experiments

The adsorption experiments were performed as follows. Furfural solution (100 ml) with known initial concentration (C = 5–20 g/L) and a known adsorbent dose (m = 75 g/L) was added into a 200 ml conical flask. The mixture was then agitated at 100 rpm in a temperature-controlled (T = 298–328 K) shaker. The bamboo charcoal was then separated from the solution at different time intervals by filtering. A Lambda 35 UV-spectrophotometer (PerkinElmer, USA) was used to analyze the residual furfural concentration. For adsorption isotherms, different concentrations (C = 5, 8, 10, 15, 20 g/L) of furfural solution were agitated at a sorbent dose of 75 g/L until the equilibrium was reached. The blank experiment was also conducted by agitating furfural solution but without adding bamboo charcoals. Preexperiment showed that furfural was stable with time. The repeatability of all the results was greater than 95%.

2.3. Kinetic study

The instantaneous behaviors of the “bamboo charcoal-furfural” system were modeled by Lagergren’s pseudo-first-order equation and Ho’s pseudo-second-order equation. The linear forms of these two models are $\lg(q_e\text{-}q_t) = \lg q_e\text{-}k_1t/2.303$ and $t/q_t = 1/(k_2q_e^2) + t/q_e$, respectively (Allen et al., 2005). Here, k_1 and k_2 stand for the rate constant of Lagergren’s model (min^{-1}) and Ho’s model (g/mg min), respectively; q_e and q_t stand for the quantity of furfural adsorbed at equilibrium and at time t (mg/g), respectively. The applicability of the kinetic models could be checked by their linearity.

2.4. *Rate-controlling steps determination*

The adsorption of furfural onto bamboo charcoal can be described by three sequential processes: diffusion of furfural through the liquid film, through the particle, and then chemical reaction with bamboo charcoal. One step that offers much larger resistance than the other two is believed to be the rate-limiting step (Dana and Wheelock, 1974). Two main kinetic models (HPDM and SCM) are selected to determine which mechanism controls the furfural uptake. For HPDM, if the adsorption process is controlled by the liquid film, equation of $-\ln(1-X(t)) = K_{li}\,t$ (labeled as Eq. (1)) can be used (Valderrama et al., 2008); if it is controlled by particle diffusion, $-\ln(1-X^2(t)) = 2Kt$ (labeled as Eq. (2)) can be used. Here, $X(t)$ is defined as "q_t/q_e", and K_{li} and K are the rate constants for HPDM and SCM, respectively (Valderrama et al., 2010). For SCM, equations of $X(t) = 3C_{Ao}K_{mA}t/arC_{so}$ (Eq. (3)), $1-3(1-X(t))^{2/3} + 2(1-X(t)) = 6D_eC_{Ao}t/ar^2C_{so}$ (Eq. (4)), and $1-(1-X(t))^{1/3} = K_sC_{Ao}t/r$ (Eq. (5)) can describe the process if the adsorption is controlled by liquid film, particle diffusion, and chemical reaction, respectively. Here, D_e is the effective diffusion coefficient. The definitions of the other kinetic parameters have been provided in Ref. (Valderrama et al., 2010; Valderrama et al., 2008). The target of this kinetic study was to determine the rate-controlling steps of furfural onto bamboo charcoal and obtain more kinetic parameters for representative fermentation inhibitor removal.

2.5. *Adsorption isotherms*

The adsorption isotherms are fundamental in the description of the interactive reaction between adsorbent and solutes. Two widely used isotherms (Langmuir/Freundlich isotherm) are selected to evaluate the interactive behavior. The Langmuir isotherm usually supposes a monolayer sorption on the surface of identical sites and the Freundlich isotherm is usually used in a nonideal adsorption system. The linear forms of these two isotherms are $C_e/q_e = C_e/q_m + 1/(q_mK_L)$ and $\lg q_e = \lg C_e/n + \lg K_F$, respectively (Pehlivan and Arslan, 2006). Here, q_m is the maximum adsorption capacity of furfural onto bamboo charcoal (mg/g), and n stands for the adsorption intensity. A value of $1/n < 1$ means a favorable adsorption process.

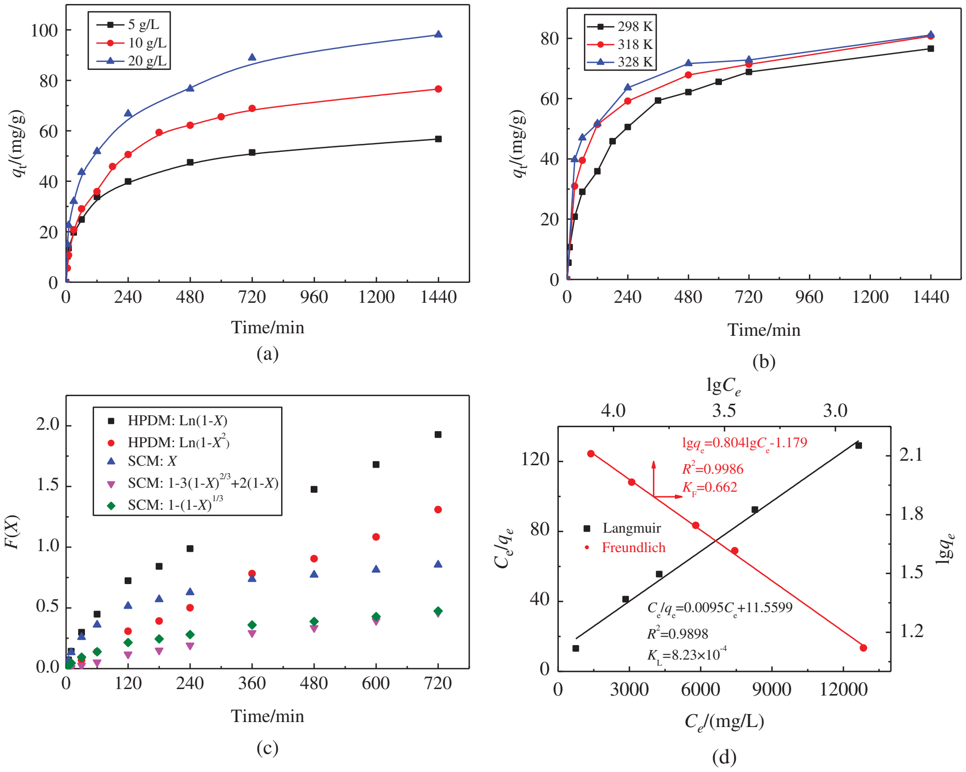
3. Results and discussion

3.1. *Adsorption kinetics for furfural*

The evolvement of furfural uptake q_t (mg/g) with time at different initial concentrations and temperatures is presented in Figure 1a and 1b. It can be seen that the equilibrium of adsorption is achieved within 24 h. The quantity of adsorbed furfural increases with the increase of the initial concentration and temperatures. However, the effect of temperature on the adsorption of furfural is not very significant compared to the influence of initial concentrations. The kinetic parameters obtained from the fitting of Lagergren's and Ho's models are listed in Table 1. It can be seen that Ho's model agrees with the experimental data well, and is better than Lagergren's model for the whole sorption stage based on its high correlation coefficients (R^2). The values of $q_{e,cal}$ calculated by Ho's model are much consistent with the experimental data than that of Lagergren's model. The k_2 values increase with the increase of temperatures, indicating an increasing adsorptive rate. However, it was not observed with the increase of initial concentrations and a similar trend can be found in other research (Sahu et al., 2008).

3.2. *Kinetic mechanisms*

To realize the adsorption process from a microscopic point of view, kinetic mechanisms mentioned in section 2.4 were evaluated. The experimental data with 10 g/L of furfural were analyzed using the kinetic models defined by Eqs. (1)–(5), and the results are shown in Figure 1c, in which $F(X)$ is the function of $X(t)$ (Valderrama et al., 2008). It can be observed that neither the liquid film diffusion model nor the chemical



(a) (b) (c) (d)

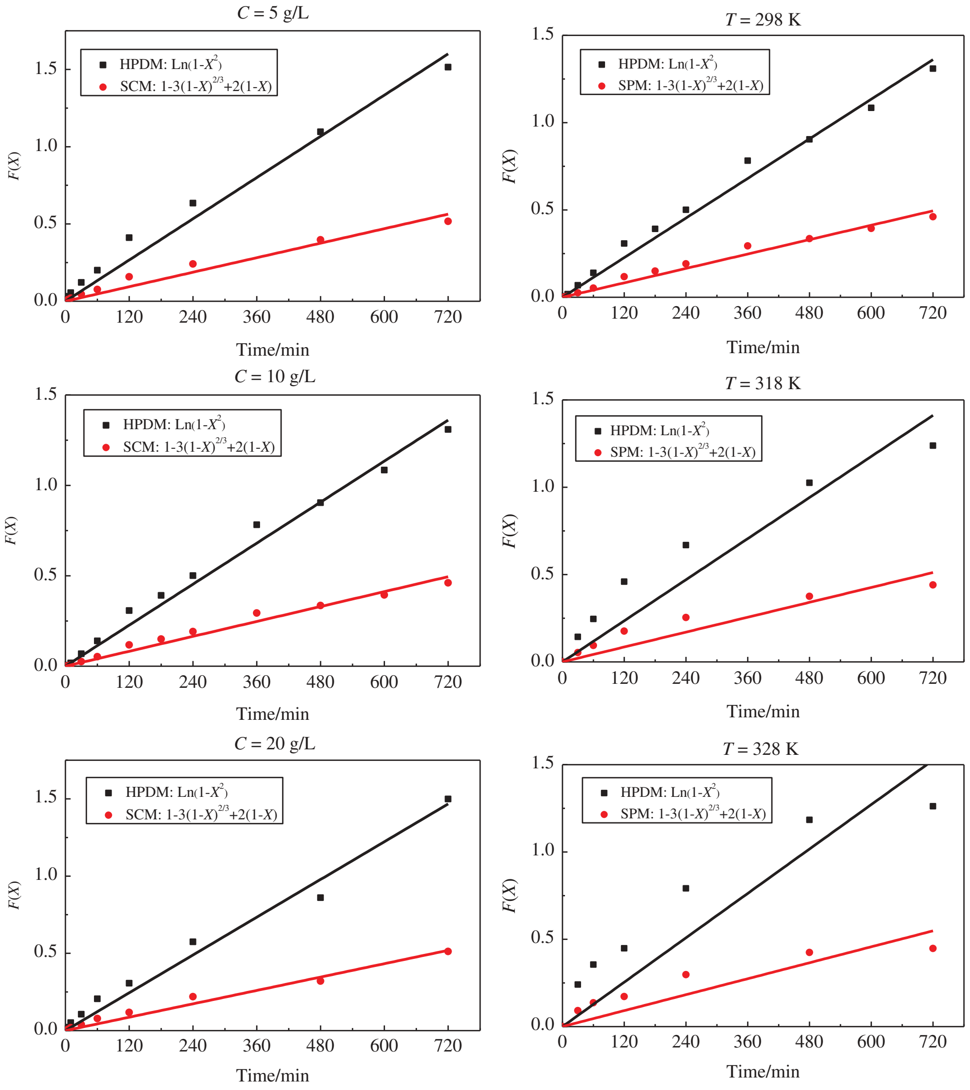
Figure 1. Adsorption characteristics of furfural onto bamboo charcoal. (a) Effect of initial concentrations on the adsorption capacity of furfural, T = 298 K, m = 75 g/L; (b) effect of temperature on the adsorption capacity of furfural, C = 10 g/L, m = 75 g/L; (c) test of the kinetic models defined by Eqs. (1)–(5) for the homogeneous particle diffusion and shrinking core models. m = 75 g/L, T = 298 K, C = 10 g/L; (d) linear Langmuir and Freundlich plots for the adsorption isotherm of furfural.

reaction model gives straight lines. In general, if it is controlled by film diffusion, the conditions mainly comprise small particle size, low solution concentrations, and low degree of agitation (Dana and Wheelock, 1974). The adsorption process in this study does not meet the point. Simultaneously, the chemical reaction is usually considered to be too quick to influence the whole adsorption process (Saha et al., 2004). Thus film diffusion and chemical reaction could be excluded as the controlling step. Serarols (Serarols et al., 2001) investigated the adsorption of Zn(II) onto XAD2, a macroporous resin. They found the dominant controlling-step of the adsorption is the liquid film mass transfer at low concentrations while it is the intra-particle diffusion for high concentrations. From Figure 1c it can be seen that both HPDM and SCM controlled by particle diffusion agree with the data well for the overall process. In order to examine the general applicability, the HPDM and SCM models controlled by particle diffusion were further performed to fit the other adsorption data. The results are shown in Figure 2 for different initial concentrations (5–20 g/L) and temperatures (298–328 K). The linear regression analysis of the parameters is listed in Table 2.

From Figure 2 and Table 2, it is suggested that the particle diffusion control is the dominant transport rate for furfural adsorption onto bamboo charcoal since all of them produce straight lines and have high correlation coefficients, specifically at the concentration of 5–20 g/L of furfural at 298 K. Thus, it can be deduced that the furfural removal efficiency will be enhanced by increasing the force of interparticle mass transfer. However, with the increasing temperatures, a slight deviation from linearity was observed using these two kinetic models. Kinetic models defined by Eqs. (1), (3), and (5) were also used to fit the data and none of them gave a straight line (not shown here). That is to say, neither the liquid film diffusion nor the chemical reaction is the rate-limiting step when the adsorption occurs at a high temperature. Shakir and

Table 1. Parameters of kinetic models of furfural adsorption onto the bamboo charcoal.

		Pseudo-first-order model			Pseudo-second-order model		
Adsorption condition	$q_{e,exp}$ (mg/g)	$q_{e,cal}$ (mg/g)	k_1 (min^{-1})	R^2	$q_{e,cal}$ (mg/g)	k_2 (g/(mg min))	R^2
Initial concentration (g/L)							
5	56.71	41.11	2.42E-03	0.9671	2.44E-04	0.9946	
10	76.57	59.30	2.08E-03	0.9431	1.09E-04	0.9937	
20	98.01	74.85	2.35E-03	0.9755	1.21E-04	0.9909	
Temperature (K)							
298	76.57	59.30	2.08E-03	0.9431	1.09E-04	0.9937	
318	80.72	51.77	2.07E-03	0.9393	1.44E-04	0.9961	
328	81.11	45.79	2.20E-03	0.9208	1.89E-04	0.9970	



(a)

(b)

Figure 2. Particle diffusion models of furfural defined by Eqs. (2) and (4). (a) With different initial concentrations, $T = 298$ K, $m = 75$ g/L. (b) At different initial temperatures, $C = 10$ g/L, $m = 75$ g/L.

Shakir and Beheir, (1980) qualitatively demonstrated the adsorption of UO_2^{2+} with di-(2-ehtylhexyl) phosphoric acid (DEHPA) was controlled by united chemical reaction and particle diffusion. Thus, coupling of kinetic mechanisms may play a critical role in this study.

Since the HPDM and SCM models controlled by particle diffusion give better fitting than the other two models, the diffusion coefficients for furfural can be calculated by Eqs. (2) and (4) through the slope values. The obtained diffusion coefficients are actually a measurement of the mean of the furfural molecules related to the adsorption process (Valderrama et al., 2010). The results are also shown in Table 2. For HPDM, the values of diffusion coefficient (D_e) decrease first, and then increase with the increase in furfural concentration. However, the diffusion coefficients counted by the SCM decrease with the increase of the initial furfural concentration. It may be caused by the accumulation of the product from the reacted layer that blocks the progress of furfural within the layer. The diffusion coefficients

Table 2. Linear regression analysis for HPDM and SCM.

	HPDM			SCM		
Condition	$F(X) = f(t)$ slope (min^{-1})	R^2	D_e (m^2/s)	$F(X) = f(t)$ slope (min^{-1})	R^2	D_e (m^2/s)
C = 5 g/L	2.22E-03	0.99	8.21E-12	7.83E-04	0.98	7.50E-11
C = 10 g/L	1.89E-03	0.99	6.99E-12	6.88E-04	0.99	2.11E-11
C = 20 g/L	2.04E-03	0.99	7.54E-12	7.22E-04	0.99	1.41E-11
T = 298 K	1.89E-03	0.99	6.99E-12	6.88E-04	0.99	2.11E-11
T = 318 K	1.96E-03	0.95	7.25E-12	7.11E-04	0.93	2.37E-11
T = 328 K	2.12E-03	0.91	7.84E-12	7.62E-04	0.90	2.61E-11

calculated by both models increase slightly with increasing temperatures, indicating that the diffusion resistance decreases with the increase of temperatures. Furthermore, the diffusion coefficient predicted by the HPDM was in the order of 10^{-12} m^2/s, that agrees very well with previous studies that obtained from the resins (Benamor et al., 2008; Cortina and Miralles, 1997), but it was lower than that of the SCM, which had an order of 10^{-11} m^2/s. Benamor et al. also found the diffusion coefficients of HPDM predicted from cadmium were in the order of 10^{-12} m^2/s, smaller than those of SCM, which had an order of 10^{-7} m^2/s. The diffusion coefficients predicted by HPDM are lower than those of SCM, which might be attributed to the existence of chemical reaction between the furfural and the functional groups on the bamboo charcoal surface (Saha et al., 2004).

3.3. *Adsorption isotherm*

The adsorption data of furfural were examined by the Langmuir and Freundlich equations and the results are shown in Figure 1d. As can be seen, the adsorption data can be well described by both the Langmuir and Freundlich isotherm equations. Based on the value of correlation coefficient (R^2), the Freundlich isotherm provides a better fitting. It indicated that the adsorption feature of bamboo charcoal takes place by a nonideal adsorption on the heterogeneous surface. In addition, the value of $1/n$ (0.804) is less than 1, which demonstrates that the adsorption condition is favorable.

3.4. *Adsorption thermodynamic*

To fully recognize the nature adsorption process of bamboo charcoal, a thermodynamic study was performed. The Gibbs free energy can be calculated by the Van't Hoff equation: $\Delta G° = -RT \ln K_D$, where $\Delta G°$ is the change of Gibbs free energy (kJ/mol). K_D is defined as a linear or single point adsorption distribution coefficient, and it is equal to q_e/C_e (Sahu et al., 2008). The change in Gibbs free energy is still associated with the heat of adsorption and entropy change at constant temperature by $\Delta G° = \Delta H° - T\Delta S°$ (Sahu et al., 2008), where $\Delta H°$ and $\Delta S°$ are the change of enthalpy (kJ/mol) and entropy (J/K), respectively. Combining the two equations above gives $\ln K_D = -\Delta G°/(RT) = \Delta S°/R - \Delta H°/(RT)$. The value of $\Delta H°$ can be obtained from the slope of the equation plot, and the intercept gives the value of $\Delta S°$.

As mentioned in Section 3.1, Ho's model could fit well the adsorption process. Actually, the rate constants (k_2) of Ho's model can be used to calculate the activation energy of adsorption from the Arrhenius equation (Li et al., 2010): $\ln k_2 = \ln A - E_a/(RT)$. A and E_a are the Arrhenius factor and activation energy, respectively. The activation energy could be calculated from the slope of $\ln k_2$ versus $1/T$.

All of the thermodynamic parameters calculated from the above equations are listed as follows: the K_D values are 20.37 L/kg, 23.24 L/kg, and 24.21 L/kg at 298 K, 318 K, and 328 K, respectively. However for $\Delta G°$, it is −7.47 kJ/mol, −8.32 kJ/mol, and −8.69 kJ/mol, respectively. The $\Delta H°$, $\Delta S°$, and E_a values are 4.77 kJ/mol, 0.44 J/K, and 2.02 kJ/mol, respectively. Generally, the free energy change is from −20 to 0 kJ/mol for physical adsorption, while it is from −400 to −80 kJ/mol for chemical adsorption (Qiu et al., 2012). From the value of $\Delta G°$, it can be seen that the adsorption of furfural on bamboo charcoal is negative, and is in the range of −20 ~ 0 kJ/mol, which is consistent with that of other studies (Li et al., 2010; Qiu et al., 2012). It implies the adsorption of furfural is a spontaneous

Table 3. Comparison of adsorption capacities of different adsorbents for furfural.

Adsorbent	Adsorption capacity (mg/g)	Temperature (K)	Reference
Bamboo charcoal	105.15	298	This work
Polymeric resin: XAD-4	74.55	303	(Gupta et al., 2001)
Bagasse Fly Ash	81.97	293	(Sahu et al., 2007)
Activated Carbon: Coconut-based	55	293	(Sahu et al., 2008)
Activated Carbon: supplied by Unicarbon, Italian	374	303	(Sulaymon & Ahmed, 2007)
Nano-porous adsorbent	196.1	308	(Anbia & Mohammadi, 2009)

process of physical adsorption. Moreover, when the temperature increases from 298 K to 328 K, $\Delta G°$ changes from −7.47 to −8.69 kJ/mol; thus it is suggested the adsorption is even more spontaneous at higher temperature and similar results can also be found in the literature (Li et al., 2010). The value of $\Delta H°$ provides a measurement of the bonding strength between the adsorbent and adsorbate surface (Suzuki and Fujii, 2004). The $\Delta H°$ with a positive value confirms the endothermic property of the overall adsorption process, and it is supported by the phenomenon of higher temperature resulting in higher adsorption capacity of furfural. Moreover, the positive value of $\Delta S°$ indicates the randomness increased at the solid–liquid interface and the degree of freedom of the absorbed species was also increased in the adsorption process. In addition, the activation energy (E_a) is 2.02 kJ/mol in this study, which further confirms that the adsorption process is mainly physical. However, further increase of temperature is harmful for furfural removal, as previously discovered (Li et al., 2013).

3.5. *Comparison of different adsorbents*

Table 3 lists a comparison of furfural adsorption capacity by different types of adsorbents. Owing to the variety of parameters and conditions, direct comparison was impossible. However, from Table 3, the adsorption capacity (q_m, calculated by the Langmuir isotherm equation) of furfural onto the bamboo charcoal was comparable to or much better than most of the other adsorbents reported in the literature, which further revealed that bamboo charcoal is feasible to be used as an adsorbent for furfural removal.

4. Conclusions

Owing to the challenges of the fermentation inhibitor removal for bioethanol production, the bamboo charcoal derived from the pyrolysis process was introduced for this application. The kinetics, isotherm, and thermodynamic characterizations on the adsorption of furfural were investigated. The adsorption process of furfural onto bamboo charcoal obeys Ho's pseudo-second-order kinetic model. Both HPDM and SCM can be used for the description of furfural removal onto bamboo charcoal. The particle diffusion of the HPDM and SCM mechanisms shows a good approach to the adsorption data of furfural onto bamboo charcoal, indicating the rate-limiting step is the adsorbent phase diffusion. Thus, increasing the force of interparticle mass transfer will be helpful for furfural removal. The furfural diffusion coefficient predicted by HPDM was in the order of 10^{-12} m^2/s, similar to that reported in the literature, but it was smaller than that calculated by SCM, which had an order of 10^{-11} m^2/s. The Freundlich equation provided a better fitting to the isotherm data compared to the Langmuir equation, implying that the adsorption feature of the bamboo charcoal takes place by a nonideal adsorption on heterogeneous surfaces. Thermodynamic analysis suggested $\Delta H° > 0$, $\Delta G° < 0$, $\Delta S° > 0$, and $E_a = 2.02$ kJ/mol, indicating the adsorption of furfural onto bamboo charcoal is a spontaneous physical and endothermic process. Compared with other adsorbents, bamboo charcoal proved to be a promising sorbent for furfural removal. This preliminary research may invite further investigation on the property improvement of bamboo charcoal for its utilization in the removal of fermentation inhibitors by the adsorption method.

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