Isotherm, Kinetic and Thermodynamic Studies for Adsorption of Cd(II) from Aqueous Solution onto Mesoporous Bamboo Activated Carbon

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Summary
In this study, activated carbons were prepared from Moso bamboo by N2 carbonization under N2 gas atmosphere, using H3PO4 as activator and oxidized by (NH4)2S2O8 for the removal of Cd(II). The physicochemical properties of the prepared activated carbons (BAP) and commercially available bead-shaped activated carbon (BAC) were compared. The characterization results showed that BAP had higher surface area and mesopore volume compared with BAC, and the oxidized sample BAP1 had more surface functional groups. Adsorption equilibrium data were analyzed by the Langmuir and the Freundlich models. The adsorption equilibrium data were well described by the Langmuir model and the maximum adsorption capacity of BAP1-Na was found to be 0.721 mmol/g, which was greater than that of BAC1-Na. In the adsorption thermodynamics parameters study, the adsorption was spontaneous, favorable and exothermic. The experimental kinetic data were best fitted to pseudo-second-order model, and adsorption rate of BAP1-Na was faster than that of BAC1-Na. The prepared BAC1-Na showed better adsorption capacity for Cd(II) adsorption, and pH, oxidation time, adsorbent dosage and temperature also affected adsorption capacity of Cd(II).

Key words: Bamboo, Activated carbons, Toxic metal, Isotherm, Kinetic

INTRODUCTION
Toxic metal wastewater is mainly discharged from mining, metal smelting, metal surface treatment, ceramics, pulp, electroplating, medicine, paint, battery manufacturing and other industries1). Toxic metals can be hardly biodegraded. When toxic metal wastewater is discharged into the water, a small portion of the toxic metal is absorbed by fish, and most of the toxic metals are adsorbed by colloid and particulate matter and then deposited at the bottom of water. Cadmium is a common toxic metal and can cause several diseases, including lung dysfunction, renal dysfunction, cancer, disease and high blood pressure2). According to WHO regulations, the maximum allowable concentration of cadmium in drinking water is 0.003 mg/L3). Therefore, the removal and elimination of cadmium from contaminated water and wastewater are necessary to protect the environment and the human health.

The adsorption method mainly uses the intermolecular physicochemical action to remove the heavy metal ions in the wastewater. Widely used adsorbents generally have a large specific surface area, such as activated carbon4),5), zeolites4), and molecular sieves5), of which the application of activated carbon is the most common. Activated carbon is a black porous material that has been activated by physical or chemical methods6). It has the characteristics of internal porosity development, large specific surface area and high adsorption capacity. Activated carbon is stable nature, high acid and alkali resistance and heat tolerance, insoluble in water or organic solvents and easy to regenerate, so it is widely used as an environment-friendly adsorbent.

Bamboo is abundant, especially in Asia, and possesses the rapid growth ability compared to other plants because it can mature at only 3–5 years of age, so bamboo could be regarded as cheaply available material7). Surface physical structure modification for activated carbon refers to the process by physical or chemical activation methods to increase the specific surface area8), to change the pore volume and structure, and to adjust the pore size distribution, thus changing the physical adsorption properties of activated carbon. In chemical activation method, raw material is crushed first, and the activator and the crushed raw materials are mixed with a desired percentage. According to the different activators, raw materials were selectively heated under inert atmosphere, to complete carbonization and activa-
tion of the materials. The main activator used is ZnCl₂, H₃PO₄, alkali such as KOH and NaOH, and then washed in a Soxhlet extractor for 24 h to remove chloride ions and other impurities, and the samples were referred to as BAPx-Na(x = 1, 2, 3) and BAC1-Na, respectively.

Characterization of activated carbon

Specific surface area (Sₘₐₓ), mesopore volume (Vₘₐₓ), and micropore volume (Vₘᵢₙ) for the prepared activated carbons were analyzed by N₂ adsorption-desorption at −196°C with a surface area and pore size analyzer. The amounts of surface functional groups and basic sites for the prepared activated carbon were quantified by Boehm titration method. About 0.05 g of the prepared activated carbon was added into 25 mL of 0.05 M NaClO₃, 0.1 M NaOH, 0.1 M HCl or 0.1 M NaHCO₃ solution in flasks. The flask were shaken at 100 rpm for 48 h at 25°C. NaHCO₃ neutralizes only the carboxy group on the surface of the carbon, and NaClO₃ neutralizes the carboxy, lactone and phenolic hydroxyl groups. The amount of the corresponding functional group can be calculated from the difference in alkali consumption.

Adsorption isotherm

In the adsorption equilibrium experiment, 0.05 g activated carbons were added into 100 mL conical flasks containing 25 mL of different initial concentrations (50–1000 mg/L) of Cd(II) solution. The flasks were agitated in a thermostatic shaker at 25°C. The Langmuir isothermal adsorption model is a theoretical model assuming that the adsorbate is adsorbed on the monolayer of surface of the adsorbent. The linear form of Langmuir equation can be given as

\[ \frac{C_e}{Q_e} = \frac{1}{K_L X_m} + \frac{1}{X_m} C_e \]

where \( Q_e \) (mmol/g) is the adsorptive amount of Cd(II) at equilibrium, \( X_m \) (mmol/g) is the monolayer adsorption capacity and \( K_L \) (L/mmol) is the Langmuir constant related to adsorption energy. The linear equation of the Langmuir isothermal adsorption model defines \( R_L \) as the separation factor, which is represented as equation (2):

\[ R_L = \frac{1}{1 + K_L C_i} \]

where \( C_i \) is the initial Cd(II) concentration in the solution. \( R_L \) is the adsorption equilibrium parameter. There are four possibilities for the \( R_L \) value\(^{26}\): 0 < \( R_L \) < 1, favorable adsorption; \( R_L = 1 \), unfavorable linear adsorption; and \( R_L = 0 \), irreversible adsorption.

The linear form of Freundlich equation is:

\[ \ln Q_e = \ln K_F + \frac{1}{n} \ln C_i \]

where \( K_F \) is the Freundlich constant and \( 1/n \) is the heterogeneity factor. The greater the value of \( n \), the better the adsorption performance.

RESULTS AND DISCUSSION

Characterization of activated carbon

The textural and surface properties of the prepared activated carbons are shown in Table 1. The data showed that the highest surface
area and the highest mesopore volume were obtained for BAP. In the oxidized samples, the amount of acidic functional groups was larger than that for the non-oxidized sample, and the surface area and pore volume were significantly decreased. BAP was introduced with more acidic functional groups than BAC under the same period of oxidation.

Adsorption studies of Cd(II) in aqueous solutions

Effect of equilibrium pH

Equilibrium solution pH plays a significant role in the removal efficiency of Cd(II) from aqueous solution. The amount of Cd(II) adsorbed at equilibrium increased gradually in the equilibrium pH range from 1.5 to 5.5.

In the process of Cd(II) adsorption on activated carbon, Cd(II) ions were exchanged with hydrogen ions on the surface carboxy group or other functional groups of the oxidized activated carbon, causing the decrease in the solution pH. It is also obvious from Fig. 1 that the decrease in the pH would reduce the adsorption capacity. In the experiments of adsorption isotherms and adsorption kinetics, Na-intercalated activated carbons were used.

Table 1: Textural and surface properties of each prepared activated carbon

| Sample | $S_{\text{BET}}$ [m$^2$/g] | $V_{\text{micro}}$ [cm$^3$/g] | $V_{\text{meso}}$ [cm$^3$/g] | Surface functional groups of each sample [meq/g] |
|-------|----------------|----------------|----------------|------------------------------------------|
|       |                |                |                | Carboxy | Lactone | Hydroxy | Basic |
| BAC   | 1380           | 0.57           | 0.03           | 0.00    | 0.16    | 0.10    | 0.60   |
| BAP   | 1470           | 0.32           | 1.64           | 0.00    | 0.00    | 0.19    | 0.06   |
| BAC1  | 940            | 0.34           | 0.12           | 2.40    | 0.23    | 1.48    | 0.00   |
| BAP1  | 580            | 0.14           | 0.24           | 3.71    | 0.26    | 3.65    | 0.00   |

Adsorption equilibrium experiments

1) Adsorption isotherms for different adsorbent

Adsorption isotherms of Cd(II) for BAC1-Na and BAP1-Na are shown in Fig. 2. Adsorption isotherms using Langmuir and Freundlich models were applied to investigate the adsorption process of Cd(II) at 25°C and the different isotherm constants determined were presented in Table 2.

Through the Langmuir and Freundlich data fitting of adsorption isotherms of Cd(II) for BAP1-Na, the $R^2$ values were greater than 0.999, indicating that the two models can be a good description of Cd(II) adsorption behavior. In the Langmuir isothermal adsorption model, $R_L = 0.008$ (0 ≤ $R_L$ ≤ 1), indicating favorable adsorption of Cd(II) on the AC samples.

In the Freundlich isothermal adsorption model, $n = 12.804$, also draw the same conclusion. Langmuir model fitting experiment data are slightly better than Freundlich adsorption model, indicating that Cd(II) adsorption onto modified activated carbon tended to be monolayer adsorption. Similar trends could be drawn from the adsorption isotherms experiments data of BAC1-Na. In comparison with the maximum adsorption capacity ($X_m$) of two adsorbents in adsorption

Fig. 1: The Cd(II) adsorption capacity of BAP1 under different equilibrium pH values
Amount of adsorbent: 50 mg. Solution volume: 25 mL.

Fig. 2: Adsorption isotherms of Cd(II) for 48 h at 25°C
Amount of adsorbent: 50 mg. Solution volume: 25 mL.
isotherm experiment, the $X_a$ value for BAP1-Na was larger than that for BAC-AFS-Na, implying that the adsorption capacity of BAP1-Na was better than that of BAC1-Na. This may be because BAP has a greater specific surface area than BAC, as shown in Table 1. And adsorption capacity of Cd(II) was higher than that reported by Mohan et al.\textsuperscript{30} (0.34 mmol/L), Lu et al.\textsuperscript{29} (0.11 mmol/L) and Moreno-Tovar et al.\textsuperscript{29} (0.39 mmol/L).

\textbf{ii) Adsorption thermodynamics parameters}

Adsorption isotherms of Cd(II) for BAP1-Na at the temperature of 298, 308, 318K are shown in Fig. 3. The thermodynamic parameters Gibbs free energy change $\Delta G^\circ$ (KJ/mol), enthalpy change $\Delta H^\circ$ (KJ/mol) and $\Delta S^\circ$ (entropy change, J/mol K) are calculated by Eqs. (4), (5) and (6).

\begin{eqnarray}
\Delta G^\circ &=& -RT\ln K_d \\
\Delta G^\circ &=& \Delta H^\circ - T\Delta S^\circ \\
\ln K_d &=& \frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R}
\end{eqnarray}

where $R$ (8.314 J/mol K) is a general gas constant, $T$ (K) is the thermodynamic temperature, and $K_d$ (L/mg) is the standard thermodynamic equilibrium constant defined by $q_e/C_e$, which is from the Langmuir equation ($K_d$) or Freundlich equation $K_f$. The correlation coefficients ($R^2$) are given in Table 3. Because Cd(II) adsorption data on the BAP1-Na fit better with the Langmuir adsorption isothermal equation in the study, the Langmuir adsorption isothermal constant was used. The slopes and intercepts of the plots of $\ln K_d$ vs. $1/T$ were shown in Fig. 4. The values of $\Delta H^\circ$, $\Delta S^\circ$ and $\Delta G^\circ$ for Cd(II) adsorption on BAP1-Na are given in Table 4. The negative values of $\Delta H^\circ$ indicated an exothermic process of adsorption, and the negative values of $\Delta S^\circ$ showed that the system of Cd(II) adsorption disorder decreased at the solid/solution interface during adsorption. The negative value of $\Delta G^\circ$ indicates that all adsorption processes were viable and spontaneous, similar to that reported by Mohan et al.\textsuperscript{30} for the adsorption of Cd(II).

\textbf{Cd(II) adsorption kinetics of the prepared AC}

In the adsorption experiment, 0.1 g of the prepared activated carbons were mixed with 100 mL of 500 mg/L Cd(II) solution and agitated at 100 rpm at 25°C and the solution pH was not adjusted.

\textbf{i) Effect of adsorbent type}

The effects of adsorbent type on Cd(II) adsorption were compared. The amount of Cd(II) adsorbed on the AC was studied as a function of shaking time at different adsorbent types (BAP1-Na and BAC1-Na). The relationship between adsorption time and adsorption capacity is shown in Fig. 5.

![Fig. 3 Adsorption isotherms of Cd(II) for 48 h at 298 K, 308 K and 318 K](image)

It is evident from these results that the adsorptivity of Cd(II) was enhanced with the increase in contact time from 0 to 20 min, and was almost saturated after 20 min for BAC1-Na. The adsorption rate of BAP1-Na was significantly faster than BAC1-Na, and was almost saturated after 10 min. Under the same experimental conditions, the adsorption capacity of Cd(II) for BAP1-Na was higher than that of BAC1-Na.

\textbf{ii) Effect of oxidation time}

Fig. 6 shows the relationship between the amount of Cd(II) adsorbed and oxidation time of activated carbon (1 d, 2 d or 3 d).

With increasing the oxidation time, the adsorption capacity of Cd(II) was also increased. This trend would be due to the increased oxidation time after the activated carbon was loaded with more acidic functional groups. Mena Aguilar et al.\textsuperscript{30} had the same conclusion in studying the Pb(II) adsorption onto oxidized activated carbon fiber.

\textbf{iii) Effect of adsorbent dosage}

The influence of adsorbent dosage on Cd(II) adsorption was studied by varying the amount of adsorbent (BAP1-Na) from 1.0 to 3.0 g/L. Fig. 7 depicts the adsorption capacity of Cd(II) with different adsorbent dosages. With the increase in the amount of adsorbent dosage, the accumulation of particles causes the aggregation and overlap of the adsorption sites, resulting in a decrease in the available surface area of the adsorbents and an increase in the length of the diffusion path.

\textbf{Kinetic analysis}

The experimental data in Figs. 5–7 are fitted with pseudo-first-order model and pseudo-second-order model when BAP1-Na was used.
Table 3  Langmuir and Freundlich parameters of Cd(II) at different temperature

| Sample   | Temperature [K] | Xₐ [mmol/g] | K_L [L/mmol] | R² | K_f [(mmol/g)(mmol/L)⁰] | R² |
|----------|-----------------|-------------|--------------|----|--------------------------|----|
|          | 298             | 0.721       | 16.381       | 0.008 | 0.999 | 12.804 | 0.634 | 0.930 |
| BAP1-Na  | 308             | 0.670       | 13.613       | 0.009 | 0.998 | 17.391 | 0.597 | 0.899 |
|          | 318             | 0.610       | 12.371       | 0.010 | 0.996 | 28.818 | 0.563 | 0.584 |

Table 4  Adsorption thermodynamic parameters for Cd(II) adsorbed on BAP1-Na

|                       | ΔS° [J/molK] | ΔH° [KJ/mol] | ΔG° (298 K) [KJ/mol] | ΔG° (308 K) [KJ/mol] | ΔG° (318 K) [KJ/mol] |
|-----------------------|-------------|--------------|---------------------|---------------------|---------------------|
|                       | -14.098     | -11.096      | -6.885              | -6.744              | -6.603              |

Fig. 4  Plot of ln K_d vs. 1/T for Cd(II) adsorption on BAP1

Fig. 5  Effect of adsorbent type on Cd(II) adsorption rate

Fig. 6  Effect of oxidation time on Cd(II) adsorption rate

Fig. 7  Effect of adsorbent dosage on Cd(II) adsorption rate
The adsorption rate constants for the pseudo-first-order model was given by Langergen and Svenska\textsuperscript{30}. The linear form of the pseudo-first-order equation can be expressed as Eq. (7)

$$\ln(Q_e - Q_t) = \ln Q_e - k_1 t,$$

where \(Q_t\) is the amount of Cd(II) adsorbed at equilibrium (mmol/g), \(Q_e\) is the amount of Cd(II) adsorbed (mmol/g) at time \(t\) (min), and \(k_1\) is the rate constant of the pseudo-first-order model (1/min).

The linear form of the pseudo-second-order model equation is given as Eq. (8)

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e},$$

where \(k_2\) is the rate constant of the pseudo-second-order model (g/mmol min). The results of the kinetic analysis are shown in Tables 5–7.

The pseudo-first-order model has lower correlation coefficient \((R^2)\) when compared to the pseudo-second-order models, and all \(R^2\) values are greater than 0.99 for pseudo-second-order models. The prepared BAP-1-Na displayed higher adsorption rate than BAC-1-Na. This may be due to the higher surface area and larger mesopore volumes for BAP than BAC, which causes higher adsorption rate.

The adsorption rate for Cd(II) decreased with the increase in oxidation time. With the increase of the oxidation time and the number of acidic functional groups on the activated carbon surface and the adsorption capacity was also increased, and the adsorption rate was decreased. With the increase of the activated carbon dosage, the time for adsorption equilibrium was decreased. It may be due to the enhanced number of active sites for adsorption.

### CONCLUSIONS

Moso bamboo was used as a precursor for the production of mesoporous activated carbon by H\textsubscript{3}PO\textsubscript{4} activation, and it was indicated that the prepared activated carbon BAP had high surface area (1470 m\textsuperscript{2}/g) and mesopore volume (1.64 cm\textsuperscript{3}/g). The initial solution pH played an important role in the removal of Cd(II) from aqueous phase, and the decrease in pH resulted in the decrease in the amount of cadmium adsorbed by activated carbon. The Na-intercalated oxidized activated carbon BAP-1-Na could keep the pH of the solution constant during the adsorption process, and it was very effective for the removal of Cd(II). The adsorption equilibrium was well explained by the Langmuir isotherm and the pseudo-second-order kinetics. BAP-1-Na showed higher adsorption capacity and adsorption rate than those of BAC-1-Na. According to the adsorption thermodynamics parameters study, the \(\Delta H^0\) value was negative, suggesting an exothermic process and the negative value of \(\Delta S^0\) showed that the system of Cd(II) adsorption disorder decreased at the solid/solution interface during adsorption. Oxidation time, adsorbent dosage and temperature could affect the adsorption capacity and kinetics of Cd(II).

Table 5  Kinetic parameters obtained for Cd(II) adsorption using different adsorbents

| Sample     | Pseudo-first-order model | Pseudo-second-order model |
|------------|--------------------------|---------------------------|
|            | \(k_1\) [1/h] | \(Q_{c1} (\text{cal})\) [mmol/g] | \(R^2\) | \(k_2\) [g/mmol h] | \(Q_{c2} (\text{cal})\) [mmol/g] | \(R^2\) |
| BAC1-Na    | 0.107 | 1.097 | 0.977 | 0.253 | 0.797 | 0.994 |
| BAP1-Na    | 0.110 | 4.432 | 0.832 | 2.343 | 0.833 | 0.999 |

Table 6  Effect of oxidation time on kinetic parameters obtained for Cd(II) adsorption

| Sample     | Pseudo-first-order model | Pseudo-second-order model |
|------------|--------------------------|---------------------------|
|            | \(k_1\) [1/h] | \(Q_{c1} (\text{cal})\) [mmol/g] | \(R^2\) | \(k_2\) [g/mmol h] | \(Q_{c2} (\text{cal})\) [mmol/g] | \(R^2\) |
| BAP1-Na    | 0.110 | 4.432 | 0.832 | 2.343 | 0.833 | 0.999 |
| BAP2-Na    | 0.414 | 0.088 | 0.811 | 1.267 | 0.906 | 0.999 |
| BAP3-Na    | 0.119 | 0.887 | 0.959 | 0.703 | 1.244 | 0.999 |

Table 7  Effect of adsorbent dosage on kinetic parameters obtained for Cd(II) adsorption

| Sample     | Adsorbent dosage [g/L] | Pseudo-first-order model | Pseudo-second-order model |
|------------|-------------------------|--------------------------|---------------------------|
|            | \(k_1\) [1/h] | \(Q_{c1} (\text{cal})\) [mmol/g] | \(R^2\) | \(k_2\) [g/mmol h] | \(Q_{c2} (\text{cal})\) [mmol/g] | \(R^2\) |
| BAP1-Na    | 0.5 | 0.088 | 2.340 | 0.332 | 0.596 | 1.163 | 0.999 |
|            | 1.0 | 0.110 | 4.432 | 0.832 | 2.343 | 0.833 | 0.999 |
|            | 2.0 | 0.069 | 7.250 | 0.544 | 3.299 | 0.730 | 0.999 |
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