Comparative evaluation for the sorption capacity of four carbonaceous sorbents to phenol

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ABSTRACT
Sorption kinetics and isotherms of phenol by four carbonaceous sorbents (activated carbon (AC), mesoporous carbon (MPC), bamboo biochar (BBC) and oak wood biochar (OBC)) were compared in this study. MPC has the fastest sorption rate and initial sorption potential, which were indicated by sorption rate constants and initial sorption rate \( h \) in a pseudo-second-order kinetic model. The ordered and straight pore structure of MPC facilitated the accessibility of phenol. The AC showed the greatest sorption capacity towards phenol with maximum sorption of 123 mg/g as calculated by the Langmuir model. High surface area, complexity of pore structure, and the strong binding force of the \( \pi - \pi \) electron-donor-acceptor interaction between phenol molecules and AC were the main mechanisms. The BBC and OBC had much slower sorption and lower sorption capacity (33.04 and 29.86 mg/g, respectively), compared to MPC (73.00 mg/g) and AC, indicating an ineffective potential for phenol removal from water.

Introduction
The rapid growth of the population and economies all over the world lead to a massive discharge of anthropogenic organic contaminants (AOCs) from industry, agriculture, and domestic into water,\cite{1} which consequently harms human health both directly and indirectly via water and food consumption. Removal of AOCs from wastewater is urgent to protect human health and eco-environment. However, conventional municipal wastewater treatment processes are not sufficient to effectively remove AOCs from water, particularly some trace contaminants.\cite{2,3} Therefore, cost-effective approaches are needed to remove AOCs before their discharge into the environment.

Among the techniques for water and wastewater treatment of AOCs, adsorption is considered as one of the most promising approaches, because it is simple to design and operate, and requires low investment for both initial cost and land requirements.\cite{4} Carbonaceous materials have been widely used as sorbents for water/waste water treatment.\cite{5–8} Activated carbon (AC) is one of the most frequently investigated sorbents for both organic and inorganic contaminants in water due to large surface area and well developed pore structure, as well as surface functional groups, all of which are effective for adsorption of contaminants.\cite{7–9} However, the high cost of commercial AC has limited its application.

Biochar, a solid material obtained from the carbonization of biomass,\cite{10} has been applied as soil amendment for centuries. In recent decades, immobilization of anthropogenic contaminants by biochar in soil and water has become a new research interest due to its cost-effectiveness.\cite{6,11–14} Compared to AC, biochar is less expensive because of the availability of cheap biomass (such as agricultural wastes, sewage sludge, and animal litter) and simple means of production.\cite{13,15}

Besides natural biomass based AC and biochar, newly synthesized carbon material, such as mesoporous carbon (MPC), has been developed for different purposes. MPC has attracted more attention in recent years since its discovery in 1992 \cite{16} as an effective sorbent in separation processes because of its ordered pore structures, high pore volumes, high specific surface areas and tuneable pore diameters.\cite{17}

In order to evaluate the comparative sorption capability of these carbonaceous materials, phenol was selected as a model contaminant for adsorption in this study. Phenolic compounds, especially phenol, are considered as priority pollutants since they are harmful to plants, animals, and humans.\cite{4} Phenolic compounds can affect the taste and odor of fish and drinking water, even at low concentrations.\cite{13} Furthermore, phenol in source water is the precursor of some more toxic organic disinfection
byproducts, such as haloacetic acids, by chlorination during drinking water treatment.[18] As a result, removal of phenols from both wastewater and natural water sources is important to ensure safe drinking water and promote human health. Therefore, adsorption kinetics and isotherms of phenol by four carbonaceous materials (AC, MPC, and two biochars) were studied and the adsorption rates and adsorption capacities were compared.

**Material and methods**

**Materials**

AC, bamboo biochar (BBC), and oak wood biochar (OBC) were commercially purchased. The BBC and OBC were produced under temperature of 400 °C. MPC was synthesized using solvent evaporation induced self-assembly with amphiphilic triblock copolymers according to Lu et al. [19] and Meng et al. [20]. The AC, OBC and BBC were further grounded using agate grinding bowl and then passed through a 200-mesh sieve. The MPC was directly passed through a 200-mesh sieve due to its fine particle size. All prepared sorbents were dried in an oven drier at 65 °C overnight and then kept in a drying container for further use. Phenol was purchased from J&K Chemicals (China) with purity of 99.5%.

**BET analysis**

The surface areas and pore volumes of all carbonaceous sorbents were analyzed with Micromeritics ASAP 2020 using BET-N2-adsorption–desorption method after degassing at temperature of 300 °C for 3 h.

**Sorption kinetics and isotherms**

A batch sorption experiment was employed to study sorption kinetics and equilibrium sorption of phenol by four carbonaceous sorbents. In order to obtain a suitable removal rate (between 30 and 70%) for accurate sorption results in the kinetic and isotherm study, the solid-to-liquid ratio was 0.005 g:20 mL for AC and MPC and was 0.008 g:20 mL for BBC and OBC.

For adsorption kinetics, taking AC as an example, 0.005 g AC were weighed into 22 mL glass tubes and then filled with 20 mL 10 mg/L phenol containing 0.01 M NaCl, to maintain ion strength, and 0.02% NaN₃ to avoid any possible biodegradation during sorption. All tubes were tightly sealed with a Teflon-lined screw cap, and then were shaken in a shaker at 150 rpm for certain time intervals (0, 0.5, 1, 2, 4, 8, 12, 16, 24, 36, and 48 h). Duplicate samples were prepared for each time interval and two control samples without sorbents were prepared at the same time. Sorption kinetics of MPC, BBC, and OBC followed the same method as AC, with different solid-to-liquid ratio as described above.

For adsorption isotherms, 0.005 g AC, 0.005 g MPC, 0.008 g BBC, and 0.008 g OBC were separately weighed into different tubes for isotherm study. The concentration of phenol was prepared in the range of 2–100 mg/L containing 0.01 M NaCl to maintain ion strength and 0.02% NaN₃, to avoid any possible biodegradation during sorption. Tubes were shaken for 24 h after being mixed with different concentration of phenol. All samples were prepared in duplicate and control samples without sorbents were also included.

After shaking, all samples were centrifuged at 4000 rpm for 10 min. Supernatants were transferred to 2 mL HPLC vials for determination. Phenol was determined by high performance liquid chromatography (HPLC, Agilent 1260) equipped with a C18 reverse phase column (5 μm × 15 cm) and a UV detector at a wavelength of 254 nm using mixture of methanol and water (70:30/V/V) as mobile phase.

The sorption amount of phenol at each time intervals was calculated by the difference between samples and control samples (Equation (1)):

\[
q_t = \frac{(C_0 - C_t)V}{m}
\]

where \(q_t\) is the sorption amount of phenol at time \(t\). \(C_0\) is the initial concentration of phenol which was determined in control samples. \(C_t\) is phenol concentration remained in aqueous phase at time \(t\). \(V\) is the volume of phenol solution and \(m\) is the mass of sorbents. While sorption amount of phenol in the equilibrium study was calculated similarly as below:

\[
q_e = \frac{(C_0 - C_e)V}{m}
\]

where \(q_e\) is the equilibrium sorption amount of phenol on sorbents at equilibrium concentration \(C_e\).

**Results and discussion**

**Sorption kinetics**

Sorption kinetics of phenol on AC, MPC, BBC, and OBC were investigated at initial concentration of 10 mg/L and results are shown in Figure 1. A rapid phenol sorption on MPC was observed, and equilibrium was reached within 1 h of contact time. On the contrary, sorption of phenol on AC and two biochars, BBC and OBC, were much slower, and their equilibrium was reached at around 24 h. In order to quantitatively compare the sorption rate of phenol by different carbonaceous materials, the most typical and frequently used kinetic models, a pseudo-first-order kinetic model and a pseudo-second-order kinetic model, were used to simulate the adsorption kinetics in this study.

Lagergren [21] suggested a liquid-solid phase adsorption rate equation, which is the earliest first-order rate
equation to describe the adsorption rate based on adsorption capacity. Lagergren first-order rate equation was commonly called pseudo-first-order kinetic model, which can be described as equation (3):

\[ q_t = q_{e1}(1 - e^{-k_1t}) \]  

where \( q_t \) is the sorption amount of phenol on sorbent at time \( t \), \( q_{e1} \) is the maximum sorption capacity, and \( k_1 \) is the first order kinetic rate constant.

Ho et al. assumed that when chemisorption occurred, the sorption process may be second-order,[22,23] and suggested a pseudo-second-order kinetic sorption model as described below (Equation (4)):

\[ q_t = \frac{q_{e2}k_2t}{1 + q_{e2}k_2t} \]  

where \( k_2 \) is the second order kinetic rate constant, \( q_{e2} \) is the maximum sorption capacity, and \( k_2 \) is the second order kinetic rate constant.

Kinetic data was fitted with using Origin (version 8.0). It was obvious that the pseudo-second-order kinetic model fitted the experimental data better than pseudo-first-order model, indicating that chemisorption of phenol occurred on these carbonaceous materials. The corresponding kinetic sorption parameters are listed in Table 1. The correlation coefficient \( r^2 \) values of the pseudo-second-order model for all carbonaceous sorbents used in this study were higher that pseudo-first-order model, and the predicted sorption amount (\( q_{e2} \)) of pseudo-second-order model was also much closer with determined values (\( q_{e,exp} \)) than those predicted by pseudo-first-order model (\( q_{e1} \)), which indicated that the pseudo-second-order kinetic model fitted kinetic data in this study better.

### Table 1. Parameters of pseudo-first order and pseudo-second order sorption kinetics of phenol by four carbonaceous sorbent (initial phenol concentration: 10 mg/L).

| Sorbents | \( q_{e,exp} \) | \( q_{e1} \) | \( k_1 \) | \( r^2 \) | \( q_{e2} \) | \( k_2 \) | \( r^2 \) |
|----------|----------------|-------------|----------|--------|-------------|--------|--------|
| AC       | 66.17          | 59.60       | 0.02348  | 0.8925 | 63.66       | 5.1 \times 10^{-4} | 0.9544 |
| MPC      | 29.68          | 28.73       | 0.08752  | 0.9879 | 29.13       | 111.4 \times 10^{-4} | 0.9921 |
| BBC      | 17.30          | 15.29       | 0.01738  | 0.8969 | 16.49       | 14.1 \times 10^{-4} | 0.9637 |
| OBC      | 12.95          | 11.81       | 0.01871  | 0.9223 | 12.58       | 22.2 \times 10^{-4} | 0.9766 |

Notes: The \( q_{e,exp} \) is determined equilibrium sorption capacity of phenol at initial concentration of 10 mg/L, while \( q_{e1} \) and \( q_{e2} \) were the first-order and second-order kinetic model estimated equilibrium sorption capacity.
fракций, [12] которые были необъявлены и не так легко доступны. Поэтому скорость адсорбции BBC и OBC также была намного медленнее MPC.

Можно сделать вывод, основываясь на кинетических данных, что из четырех углеродосодержащих материалов, использованных в этом исследовании, MPC имеет наименьшую скорость адсорбции и наименьшую адсорбционную способность, оцененную кинетическими константами. Скорость адсорбции AC, MPC, BBC и OBC последовательно следовала в порядке MPC ≫ AC > BBC ≈ OBC.

Сорбционные изотермы

В целях дальнейшего изучения сорбционной способности четырех углеродосодержащих материалов, использованных в этом исследовании, были проведены равновесные сорбционные изотермы на широком диапазоне концентраций фенола. Результаты представлены на рисунке 4. Была отмечена быстрая адсорбция фенола при увеличении равновесной концентрации. Была установлена корреляция между скоростью адсорбции и концентрацией, оцененная с помощью скорости адсорбции и начальной скорости адсорбции в кинетических моделях. Начальная скорость адсорбции AC, MPC, BBC и OBC показана на рисунке 2. MPC имеет наименьшую начальную скорость адсорбции, что указывает на легкую доступность фенола к MPC в начальный период адсорбции. Этот результат согласуется с вышеуказанными кинетическими константами.

Скорость адсорбции фенола в MPC была выше, чем в AC, BBC и OBC, что можно объяснить более развитой структурой и пористостью MPC. Кроме того, MPC имеет более развитую пористую структуру и высокую поверхность (Таблица 2), что позволяет легко доступаться к внутренней поверхности микропор, не разделяя их так быстро, как в AC. BBC и OBC имеют менее развитые пористые структуры, чем MPC и AC, и даже существуют некarbonизированные фракции, которые были необъявлены и не так легко доступны. Поэтому, скорость адсорбции BBC и OBC были также намного меньше, чем MPC.

Из кинетических данных можно сделать вывод, что MPC имеет наименьшую скорость адсорбции и наименьшую адсорбционную способность, оцененную с помощью скорости адсорбции и начальной скорости адсорбции в кинетических моделях. Начальная скорость адсорбции AC, MPC, BBC и OBC последовательно следовала в порядке MPC ≫ AC > BBC ≈ OBC.

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Таблица 2.

| Углеродосодержащие материалы | Поверхность, (м²/г) | Поровое объем (см³/г) | Диаметр пор (нм) |
|-----------------------------|--------------------|----------------------|-----------------|
| Активированный углерод (АС) | 758.9              | 0.037                | 0.42            |
| Керамический углерод (МС)  | 816.1              | 0.776                | 3.54            |
| Бамбуковый активированный углерод (BBC) | 163.3 | 0.028 | 1.10 |
| Деревянный активированный углерод (ОК) | 75.33 | 0.021 | 1.85 |

Figure 2. Initial adsorption rate (h) of four carbonaceous sorbents.

Figure 3. TEM image of MPC.
of granular ACs, with maximum adsorption capacities of 74.07 and 166.6 mg/g, which were similar with the results in this study. The differences in sorption capacity among four carbonaceous materials in this study was mainly caused by their surface area, which has been listed in Table 2. The surface area of AC and MPC are much higher than BBC and OBC, and consequently the sorption capacity of AC and MPC are greater than BBC and OBC.

Although MPC had the fastest sorption rate and higher surface area, its sorption capacity towards phenol was much lower than AC, which is about 73 mg/g. Haque et al. [26] evaluated the adsorption of phenol by MPC CMK-3s with different textural properties, and found a fast and effective removal of phenol from aqueous solution. All MPCs employed in their study had better sorption capability than AC, and the maximum adsorption of CMK3-150 was 473 mg/g. However, Shi et al. [17] also observed a much lower phenol adsorption capacity (less than 50 mg/g) by MPC CMK-3s.

Much lower sorption capacity of phenol by BBC and OBC were observed, which were about 33.04 and 29.86 mg/g, respectively. However, higher adsorption of phenol by biochars from waste biomass was observed, and the maximum sorption amount of biochar derived from rice husk was up to 589 mg/g.[27] Although the surface area of rice husk biochar was not significantly larger than BBC and OBC in this study, it was believed that hydrogen bonding, Lewis acid-base reactions, and π-π stacking interactions between phenol molecules and biochar formed a multi-layer adsorption system, which strongly enhanced the sorption of phenol by rice husk biochar. However, no similar result was obtained in this study.

Table 3. Parameters of Langmuir and Freundlich adsorption model.

| Sorbent | Langmuir model | Freundlich model |
|---------|----------------|-----------------|
|         | $K_L$          | $q_m$ (mg/g)    | $r^2$ | $K_f$       | $1/n$ | $r^2$ |
| AC      | 0.3846         | 123.38          | 0.9553 | 40.32       | 0.2871 | 0.9741 |
| MPC     | 0.2034         | 73.00           | 0.9594 | 20.28       | 0.3083 | 0.9870 |
| BBC     | 0.1607         | 33.04           | 0.9631 | 7.247       | 0.3591 | 0.9912 |
| OBC     | 0.1418         | 29.86           | 0.9591 | 7.066       | 0.3338 | 0.9915 |

Figure 4. Sorption isotherms of phenol by four carbonaceous sorbents fitted by (a) Langmuir and (b) Freundlich model.

Figure 5. Surface area normalized sorption capacity of phenol by four carbonaceous sorbents.
The sorption coefficient of both models ($K_p$ and $K_f$) showed that AC > MPC > BBC = OBC, with the sorption affinity of phenol by AC was greatest. The $q_m$ values also followed the same order, indicating that AC had the best sorption capacity and affinity towards phenol. Surface area is one of the most important factors that influenced the sorption capacity of sorbents towards contaminants. Obviously, higher-surface-area sorbents (AC and MPC) had much higher sorption capacity than lower-surface-area sorbents (BBC and OBC). However, although the surface area of MPC was greater than AC, AC had a much higher sorption capacity for phenol. Surface-area-normalized sorption capacity ($q_{m,CA}$) of the four carbonaceous for phenol was shown in Figure 5. It can be concluded that surface area was not the only predominant factor affecting the sorption of phenol on carbonaceous materials.

The high surface area of AC provided abundant potential sorption sites for phenol. The reports of sorption of phenol on AC may imply electron donor-acceptor complex, or even a dispersion forces between $\pi$-electrons in phenol and $\pi$-electrons in AC.[28,29] Furthermore, the complexity of the micropore structures of AC formed multi-direction sorption force to phenol from the surface of narrow pore. However, due to the ordered and straight mesopores of MPC and the small molecule size of phenol, sorption force may only from one direction on the inner surfaces of the mesopores. Consequently, although the surface area of MPC was the highest among the four carbonaceous materials, the binding affinity seems not as strong as on AC, indicated by relatively lower sorption capability than AC. For BBC and OBC, their alkali nature caused the ionization of phenol to a negative charged ion, and the electrostatic repulsion between the negatively charged biochar surface and the negative phenol prohibited the sorption of phenol on the biochar surface. Along with the smallest surface area of BBC and OBC, their sorption capabilities for phenol were much lower than AC and MPC, and ineffective for the potential use to remove phenol in water.

Conclusion

Comparative evaluation of the sorption capacity of phenol by AC, MPC, BBC, and OBC was studied. MPC showed the fastest sorption rate, which was due to its ordered and straight pore structure. The AC showed the greatest sorption capability for phenol because of its high surface area, developed pore structure, and strong affinity between phenol molecules and AC. Two biochars, BBC and OBC, showed ineffective removal of phenol for water.

Disclosure statement

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