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Adsorption of cadmium by biochar derived from municipal sewage sludge: Impact factors and adsorption mechanism

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HIGHLIGHTS

- pH and biochar dosage are key factors for adsorption by sludge-derived biochar.
- Equilibrium temperature has weak adsorption effect.
- The mechanism of adsorption by biochar involves surface precipitation and ion exchange.

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ABSTRACT

Static equilibrium experiments were carried out to investigate the impact factors and the mechanism of cadmium adsorption on biochar derived from municipal sewage sludge. An appropriate dosage of biochar is sufficient; in the experiment, 0.2% is the optimal dosage for the largest removal capacity, while the removal capacity of biochar reduces with the increasing dosage. pH is another dominant factor of the adsorption process. The removal capacity of biochar is lower than 20 $mg \cdot g^{-1}$ when the solution initial pH is lower than 2 pH units, comparatively retaining more than 40 $mg \cdot g^{-1}$ at the solution initial pH higher than 3 pH units. Temperature has weak influence on the adsorptive performance. The main mechanism of the adsorption process of biochar for cadmium mainly involves (1) surface precipitation by forming insoluble cadmium compounds in alkaline condition, and (2) ion exchange for cadmium with exchangeable cations in the biochar, such as calcium ions.

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1. Introduction

Heavy metals are one category of the most significant contaminants, causing many environmental issues that threaten human health and ecological systems, such as the "itai–itai disease" and "minamata disease" (Taty-Costodes et al., 2003; Wang and Chen, 2009). Both industrial and agricultural effluents discharge large amount of heavy metals into surface water, as well contaminating ground water in trace amount by leaching from the earth surface after rainfall or snow (Di Natale et al., 2008; Kılıç et al., 2013). Cadmium, one of the most toxic heavy metals, has become a significant concern because of its solubility, mobility and biological accumulation (Sud et al., 2008; Belhalfaoui et al., 2009), which is ubiquitous throughout the world, and could lead to bone and kidney damage after prolonged exposure (Volesky and Holan, 1995; Sud et al., 2008).

Traditional treatment methods to remove heavy metals from aqueous solutions include coagulation, chemical precipitation, ion exchange and membrane separation processes. However, given the high cost and significant amount of residual sludge of these traditional methods, adsorption is considered a better choice, especially at low concentration (Wang et al., 2009; Kılıç et al., 2013). Despite the widespread use and perfect adsorption performance, alternative materials for activated carbon as the most popular adsorbent are still needed due to its expensive price (Babel and Kurniawan, 2003; Febrianto et al., 2009; Stavropoulos and Zabaniotou, 2009; Moreno-Barbosa et al., 2013). Biochar, a form of black carbon, is a potential low-price adsorbent with ideal adsorption efficiency, which is always produced as a by-product of biomass pyrolysis for energy recovery (Chun et al., 2004; Peng et al., 2012; Deveci and Kar, 2013; Wang et al., 2013). The mechanism of heavy metal adsorption onto biochar varies, which depends on the properties of both biochar and heavy metals,

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including: (1) surface adsorption via coordination to π electrons (C=C); (2) precipitation as insoluble matters such as hydroxide, phosphate and carbonate; (3) metal exchange with cations; (4) surface complexation with free carboxyl functional groups and hydroxyl functional groups; and (5) electrostatic interactions (Cao et al., 2009; Uchimiya et al., 2010; Harvey et al., 2011; Lu et al., 2012; Kılıç et al., 2013).

Pyrolysis is a promising technique that transforms biomass residue of different origin, among others, sludges into fuel (syngas and bio-oil) and emits less pollutants (Shinogi and Kanri, 2003; Yanik et al., 2007; Folgueras et al., 2013; Mašek et al., 2013). Biophysical drying process can reduce sludge moisture content with less energy input, and as well fine and loose particles form from raw sludge by microbial activity; due to the higher thermal conductivity, after the following fast pyrolysis, syngas with high heat value is obtained, which composes of 42.6 vol.% H_2 (0.0181 g $H_2 \cdot g^{-1}$ dried sludge) at 900 °C (Han et al., 2012). Our previous work also reported that biochar derived by the above two-step process has much higher adsorption ability for cadmium than commercial activated carbon, and 900 °C is the optimal temperature for both energy recovery and heavy metal removal (Chen et al., 2014).

Adsorbent dosage, solution pH and equilibrium temperature are important factors, that could strongly influence adsorption performance. The resolution of adsorption mechanism is the foundation of removal process controlling. Analyzing the impact factors and the adsorption mechanism is the prerequisite of biochar application in heavy metal removal from aqueous solutions.

2. Materials and methods

Analytical reagent (AR) grade chemicals and deionized water (DW) were used throughout this study. All the labware was soaked in dilute nitric acid at least overnight, thoroughly flushed with tap water, and washed three times with DW.

2.1. Biochar preparation

Municipal sewage sludge with an initial moisture content of 82.1% sampled from the Xiaojiahe Municipal Sewage Treatment Plant in Beijing, China, was treated via biophysical drying and fast pyrolysis as described by Han et al. (2012). The moisture content of the sludge after seven days biophysical drying decreased to approximately 25%, and then reduced to lower than 2.5% by air drying, denoting the residue as "dried sludge". The pyrolysis temperature was 900 °C, and the solid result biochar was ground through a 40 mesh sieve (0.45 mm) without further activation, abbreviated as "BC900". The main characteristics of BC900 are as following reported by Chen et al. (2014): ash content percentage 88.07 ± 0.56 wt.%; C element 15.92 ± 2.74 wt.%, H element 0.11 ± 0.11 wt.%, O element 2.439 ± 0.575 wt.%, N element 0.53 ± 0.07 wt.%; Ca element 69.56 $g \cdot kg^{-1}$, Mg element 17.52 $g \cdot kg^{-1}$; pH 12.15 (S/L = 1:10), pH_{PZC} 10.17, cation exchange capacity 247.51 ± 7.49 $cmol \cdot kg^{-1}$; surface area 67.603 $m^2 \cdot g^{-1}$ (Brunauer-Emmett-Teller [BET] model), average pore size 3.840 nm, pore volume 0.09855 $cm^3 \cdot g^{-1}$.

To remove exchangeable ions and soluble alkaline substances, 2 g of BC900 was soaked in 800 mL DW overnight followed by filtering with 0.45 μm polysulfone filter membrane and flushing with plenty of DW until the effluent pH reached between 6 and 7, and then oven dried at 60 °C to constant weight. The residue biochar was referred as "washed BC900". To exclude the adsorption distribution of organic matters, the BC900 residue after calcinating to constant weight during approximately 120 min at 650 °C was referred as "BC900 ash".

2.2. Adsorption equilibrium experiment

Cd^{2+} stock solution was prepared by dissolving $Cd(NO_3)_2 \cdot 4H_2O$ in DW, and the Cd^{2+} concentration in Cd^{2+} stock solution was 2000 $mg \cdot L^{-1}$. 20.00 mL nitric acid (~15 M) and 9.25 g $Ca(OH)_2$ was mixed and diluted with DW to 500 mL, and Ca^{2+} stock solution was desired at a Ca^{2+} concentration of 10000 $mg \cdot L^{-1}$. Afterwards, Cd^{2+}-bearing solutions were prepared by diluting the stock solutions to specific concentrations. In the pH influence experiments, the set pH level of Cd^{2+}-bearing solutions (initial Cd^{2+} concentration of ~200 $mg \cdot L^{-1}$) was adjusted by 1 M HNO_3 and NaOH solutions before adding the adsorbent; the initial pH of the Cd^{2+}-bearing solutions was not adjusted in other cases.

Approximately 50 mg of absorbents, such as BC900, dried sludge, washed BC900 and BC900 ash, was placed into a 40 mL glass bottle. Then, 25 mL of Cd^{2+}-bearing solution was added and intensively mixed using a vortex maker. After stirring using a thermostatic box overnight at specific temperature, the suspension was filtered with 0.45 μm polysulfone filter membrane. The residual Cd^{2+} and released Ca^{2+} and Mg^{2+} concentrations were determined by ICP–OES (IRIS Intrepid II XSP Spectrometer, ThermoFisher, USA). All adsorption experiments were run in triplicate, and the blank solution was measured for quality control. To investigate the influence of BC900 dosage, the BC900 mass placed into an equilibrium system was 25, 50, 125, 200 and 250 mg, with the S:L (the solid mass/the liquid volume) value of 0.1%, 0.2%, 0.5%, 0.8% and 1.0%, respectively.

The removal percentage and removal capacity of Cd^{2+} were calculated as follows:

$$R = (C_0 - C_e)/C_0 \times 100\% \quad (1)$$

$$Q = (C_0 - C_e)V/m \quad (2)$$

Where, R is the removal percentage of Cd^{2+} (%); C_0 and C_e are the initial and equilibrium concentrations of Cd^{2+} ($mg \cdot L^{-1}$); Q is the removal capacity of Cd^{2+} at equilibrium ($mg \cdot g^{-1}$); V is the volume of the solution (mL) and m is the weight of the absorbents (mg).

2.3. Other characterization

After adsorption equilibrium, BC900 was picked up onto a carbon-coated copper grid and then air dried. The micromorphology was observed by an S-5500 scanning electron microscope (SEM. Hitachi, Japan).

BC900 after equilibrium in Cd^{2+}-bearing solutions at different Cd^{2+} concentrations were dried at 60 °C. The samples were characterized by X-ray powder diffraction (XRD) on a D8 advance X-ray diffractometer (Bruker/AXS, Germany) at 40 kV and 40 mA for monochramatized Cu Kα (λ = 0.15418 nm) radiation with a scanning rate of 8°·min^{-1}.

Fourier-transformed infrared (FTIR) spectra were investigated in the 4000–400 cm^{-1} region under a 4 cm^{-1} resolution using a Spectrum GX spectrometer (Perkin Elmer, USA). The baseline of the raw data was adjusted and then the modified data were normalized, by OMNIC 8.0.342 software (Thermo Scientific, USA).

3. Results and discussion

3.1. Effect of dosage

Absorbent dosage is a significant impact factor of adsorption process, determining the adsorbent–adsorbate equilibrium of the system (Deveci and Kar, 2013). The removal percentage of Cd^{2+} enhances as the dosage increases from 0% to 0.5%, and then maintains at approximately 100% until the dosage reaches to 1.0%.

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