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# Sorption of heavy metal ions onto crayfish shell biochar: Effect of pyrolysis temperature, pH and ionic strength

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## ABSTRACT

In this study, crayfish shell was pyrolyzed at 300 (CS300), 450 (CS450) and 600 °C (CS600) to identify the optimum temperature for improving the binding capacity of the produced biochar. The removal efficiency of biochars was assessed following mono-element sorption of Pb(II) under batch sorption and fixed-bed column experiments. The effect of pH and ionic strength on Pb(II) sorption was further investigated on the most efficient biochar type to understand environmental factors affecting sorption onto crayfish biochar. Besides, this most efficient biochar was further investigated in multi-element sorption (Pb(II), Cu(II) and As(III)) trial to assess the effect of competitive sorption on Pb(II) bioavailability. Pyrolysis temperature had different effects on elemental composition, surface area and active functional groups of the produced biochar. CS600 showed the highest sorption capacity (190.7 mg/g) of Pb(II). Sorption mechanisms were identified following FTIR, SEM, XRD and ICP analyses suggested the participation of physisorption. Sorption of Pb(II) onto crayfish biochar was strongly pH- and ionic strength-dependent as the sorption capacity showed significant reductions at low pH and high ionic strength values. The competitive sorption of Pb(II) with other metal ions (Cu(II) and As(III)) showed its superiority to bind with active sorption sites of biochar as compared with other heavy metals.

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## 1. Introduction

Biochar is pyrogenic black carbon produced from pyrolysis of biomass under oxygen-limited conditions. In the last decade, attention has been paid to biochar as a low-cost biomaterial for heavy metal sorption. Heavy metal contaminants are non-biodegradable and show a high bioavailability to reach the food chain [1]. Following the fast urbanization, heavy metals ions released to the environment through various anthropogenic industrial activities including mining, refining ores, sewage sludge, pesticide, fertilizer, batteries, iron and steel industries [2]. Lead, arsenic and copper ions are among the most dangerous heavy metals ions released to the environment. Some areas in the world have been suffering from serious heavy metals contamination including lead [3]. Stringent contaminant limits thus have been put into practice to mitigate the potential hazardous effect associated with these metals release into the environment. According to the Food and Agriculture Organization of the United Nations (FAO), permissible concentration of arsenic,

copper and lead ions in irrigation water should not exceed 0.1, 0.2 and 5.0 mg/L, respectively [4].

There are various developed technologies for decontamination of heavy metal ions (e.g. electrochemical treatment, chemical redox reactions, chemical precipitation, ion exchange and membrane process), however, most of these technologies are expensive or inefficient at large quantities [5]. Biochar shows its effectiveness in wastewater decontamination due to its large surface area, microporous structure, abundance of active functional groups and minerals [6]. Biochar-based sorbents have recorded high sorption capacity of lead [7], copper [8] and arsenic ions [9].

Several waste feedstock materials have been applied in biochar production including coconut shell [10], sawdust [11], corncob [12], apricot stone [13], and pecan shell [14]. However, little has been reported on crustacean shell wastes as feedstock materials for biochar production. Crustaceans such as shrimp, crab and crayfish shells have received much research attention due to its high content of chitin [15]. The potential of utilizing chitin which is the second most abundant biomaterial on earth manufacture functionalized biochar [16]. Chitin and its derivatives (e.g. chitosan) have high ability to bind heavy metal ions through their active functional groups to form strong complexes with the metals [17]. Yet,

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no previous studies have investigated the potential application of shellfish as a feedstock for biochar production.

Since biochar is mainly produced by the thermal pyrolysis of feedstock, understanding the effect of peak pyrolysis temperature will be a key factor for assessing the applicability of shellfish to serve as feedstock for biochar engineering. Significant differences in sorption capacity of lead were observed among biochars produced at different pyrolysis temperatures (350, 450 and 600 °C) due to alterations in their physicochemical properties. It is thus necessary to determine the optimum pyrolysis temperature for biochar production to optimize its sorption ability.

Beside the pyrolysis temperature, there are other environmental conditions including solution pH and ionic strength affecting sorption process. According to Rahman and Islam [18], sorption of Cu(II) is significantly affected by solution pH as sorbent recorded very low sorption capacity (28.66%) under acidic conditions. This sorption capacity increased progressively to reach 83.25% with raising pH up to 7. Solution ionic strength also has vital role in controlling heavy metal sorption through its compressing effect to the electrostatic double layer of sorbent. Previous studies have suggested the significant effect of increasing ionic strength on the sorption of heavy metals including lead [19], uranium [20], and cadmium [21].

Despite the intensive research on mono-element sorption, the literature is insufficient concerning the competitive sorption of multiple metal ions [22]. Previous results obtained from batch sorption and column experiments have revealed that the multi-element sorption may lead to reduced sorption of the metals as compared with that of the mono-element solution [22]. According to Park et al. sorption capacities (mg/g) of heavy metals ions sorbed as a mono-metal ion onto sesame-straw biochar were in the order of Pb(II) (102) > Cd(II) (86) > Cr(II) (65) > Cu(II) (55) > Zn(II) (34). These sorption capacities, however, showed significant reduction as multi-elements with changed order of Pb(II) (88) > Cu(II) (40) > Cr(II) (21) > Zn(II) (7) > Cd(II) (5) [23].

The objectives of this research thus were as follows: (i) study the effect of different pyrolysis temperatures on physiochemical properties and Pb(II) sorption capacity of onto crayfish shell biochar, (ii) understand the responsible mechanisms for the sorption process, (iii) evaluate the effect of solution pH and ionic strength on sorption capacity of Pb(II) onto the biochar, and (v) evaluate the effect of competitive sorption on Pb(II) sorption/release.

## 2. Materials and methods

### 2.1. Materials

All chemicals and reagents used in this study were of analytical grades, and the chemical solutions were prepared using deionized (DI) water. Lead nitrate (Pb(NO<sub>3</sub>)<sub>2</sub>), sodium chloride (NaCl), nitric acid (HNO<sub>3</sub>) and sodium hydroxide (NaOH) were purchased from Sinopharm Chemical Reagent Co., Ltd. Crayfish shell was obtained from a commercial source (Wuhan, China).

### 2.2. Biochar production

The original feedstock (crayfish shell) was air-dried, placed into a quartz tube reactor and slow pyrolyzed using a muffle furnace under high purify nitrogen gas environment to provide oxygen-limited conditions (<0.5%). Slow pyrolysis was performed at three temperatures (300, 450, and 600 °C) for 2 h. The produced biochar was ground using a mortar and sieved to a uniform size fraction of ~0.9–1.2 mm. Samples were washed several times with DI water to remove impurities and oven dried at 105 °C. The produced

**Table 1**  
The content of C, H, N, S, P, and Ca in different biochars.

Category	C (%)	H (%)	N (%)	S (%)	P (%)	Ca (%)
CS300	23.45	0.92	1.85	0.29	1.645	20.47
CS450	20.63	0.31	1.28	0.21	1.748	22.56
CS600	21.17	0.22	1.26	0.23	1.961	16.08

From 300 °C to 600 °C, with the increase of temperature, protein decomposition, reduce the water content, content of C, H, O and S is reduced, so the Ca element content increased, from 450 °C to 600 °C in the process, some Ca compound decomposition, so reduce the Ca content.

**Table 2**  
The specific surface area.

Biochar categories	BET (m <sup>2</sup> /g)
CS300	32.67
CS450	25.46
CS600	63.79

biochar samples were labeled as CS300, CS450, and CS600, and stored for later experiments.

### 2.3. Biochar characterization

Using dry combustion technique, organic elements concentration (C, H, N and S) was analyzed using a CHNS Elemental Analyzer (Vario EL cube). Metal elements (P, Ca, Mg and Pb) concentration in different biochar samples was determined using inductively coupled plasma-atomic emission spectrometry (ICP-OES, Optima 4300DV, Perkin-Elmer SCIEX, USA) (Table 1). Surface area of biochar samples was measured by TriStar II Automatic surface area analyzer using Brunauer-Emmett-Teller (BET) method (Table 2). Surface functional groups of biochars were characterized by Fourier-transform infrared spectroscopy (FTIR, Thermo Nicolet, 6700, Madison, WI, USA). Surface morphology of the samples was determined using scanning electron microscopy (JEM-6700F, Japan). A computer-controlled X-ray diffractometer (XRD) (PANalytical, X'Pert Pro, Netherlands) equipped with a stepping motor and graphite crystal monochromator was used to investigate the crystalline element in biochar samples.

### 2.4. Sorption kinetics and isotherms

#### 2.4.1. Sorption kinetics

Sorption kinetics of Pb(II) (50 mg/L) onto biochar were performed by adding 0.1 g of different engineered biochars into 50 mL of the sorbate solution in centrifuge tubes at 25 ± 0.5 °C. In triplicates, sorbent-sorbate mixtures were shaken at 120 rpm till sampling at 0.5, 1, 2, 5, 10, 15, 20, 24, and 48 h. The suspension was immediately filtered through 0.22 μm pore size membrane filters before Pb(II) determination by inductively coupled plasma-atomic emission spectrometry (ICP-OES, Optima 4300DV, Perkin-Elmer SCIEX, USA). Amounts of Pb(II) sorbed onto the engineered biochar were calculated based as the differences between initial and final aqueous concentrations.

Adsorption dynamics is the relationship between adsorption quantity and time in a continuous period of time after the experiment begins to achieve equilibrium. The transformation of the adsorption quantity and the time of adsorption equilibrium are obtained by the dynamic curve. Pseudo-first-order, pseudo-second-order, and Elovich models were used to describe the sorption kinetic data. The governing equations of these models can be written as:

$$\text{First - order: } q_t = q_e(1 - e^{-k_1 t}) \quad (1)$$

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