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Study on adsorption characteristics of biochar on heavy metals in soil

Hong Wang, Wen Xia, and Ping Lu†

School of Energy & Mechanical Engineering, Nanjing Normal University, Nanjing 210042, China

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Abstract−Three kinds of biochars (called poplar branch biochar (PBC), water hyacinth biochar (WHC), and corn straw biochar (CSC)) were prepared in a fixed-bed pyrolyzer at different pyrolysis temperature of 300-700 °C. The effects of biochar species, pyrolysis temperature and biochar addition on adsorption characteristics of typical heavy metals (HMs) of Pb and Zn in vegetable soil (collected from lead-zinc-silver mining area, Nanjing, China) were investigated. The obtained results indicate that WHC presents the best adsorption ability at the same experimental conditions, whose adsorption efficiency on HMs of Zn and Pb is 21.83% and 44.57%, and the relative adsorption capacity of Zn and Pb is 227.65 μg/g and 363.76 μg/g, respectively. The adsorption efficiency of biochar on HMs of Zn and Pb in soil increases gradually with the increasing of pyrolysis temperature. The increasing of biochar addition is beneficial to increase adsorption efficiency of soil HMs, but unhelpful for adsorption capacity.

Keywords: Biochar, Soil, Heavy metals, Adsorption Characteristics

INTRODUCTION

Soil contamination by heavy metals (HMs, e.g. Pb, Zn, Cd, etc.) has become more and more serious, which not only makes the soil fertility degradation, crop yield and quality reduction, but also ultimately endangers human health through food chain [1,2]. Applying soil conditioners to remedy heavy metals contaminated soil is a promising way, and has been paid more attention around the world. The common soil conditioners involve lime, phosphate and silicate, and so on. However, these soil conditioners all have issues that the fixation effect of HMs is unstable or new HMs are introduced [3]. Therefore, it is necessary to develop a new soil conditioner which has characteristics of strong remediation ability, high stability and environmental friendliness. Biochar is a solid material produced by biomass thermochemical process under the condition of lack of oxygen (gasification) or oxygen free (pyrolysis), which possesses good pore structure, large specific surface area and a variety of surface oxygen-containing functional groups. This excellent physico-chemical characteristics of biochar is helpful for adsorption and immobilization of HMs in soil, therefore it could be a kind of soil conditioner with great application prospect [3-5].

Biochar characteristics depend on biomass species and preparation conditions (such as pyrolysis temperature, residence time, pyrolysis atmosphere), which directly affect the remediation effect on soil heavy metals with biochar. Xu and Zhao [6] investigated the adsorption performance of crop straw biochars on Cu(II), Pb(II) and Cd(II) in three variable charge soils from southern China, and found that the peanut straw biochar had better adsorption ability on three metals than canola straw biochar. Lu et al. [7] made a pot experiment to investigate the effects of biochars derived from bamboo and rice straw on bioavailability and plant growth in a paddy soil naturally co-contaminated with Cd, Cu, Pb and Zn, and found the solubility of Cd, Cu, Pb, and Zn as measured by Toxicity Characteristic Leaching Procedure (TCLP) was significantly lower ($p<0.05$) in the biochar-amended soils than in the control soil. They also concluded that the influence of biochar on heavy metals bioavailability varied not only with the feedstock and application rate of biochars, but also with the metal species. Ding et al. [8] studied the effect of the bagasse biochar on the adsorption of Pb in aqueous solution, and concluded that Pb adsorption ability of bagasse biochar pyrolyzed at 250 °C and 400 °C was better than that of bagasse biochar pyrolyzed at 500 °C and 600 °C. Li et al. [9] investigated the effect of biochars prepared at pyrolysis temperature of 300-600 °C on Cd speciation in soil, and found the content of available Cd in soil decreased first and then increased a little with increasing pyrolysis temperature, and concluded 400 °C was the best pyrolysis temperature for biochar preparation. Biochar addition is also an important factor that affects the adsorption of soil HMs, and a little biochar addition can't effectively reduce the bioavailability of HMs, while much more biochar addition will not only increase the cost, but also lead to a reduction in crop production and animal and plant poisoning [10,11]. Liu et al. [12] found the concentration of available Cd, Cu and Zn in soil decreased significantly ($p<0.05$) with the increasing of the biochar addition based on a one-year incubation experiment through adding biochar with different particle sizes (<1 mm and <0.25 mm) and addition amount (0, 1%, 5%) into a sandy loam paddy soil. Jiang et al. [13] carried out a 30 d of soil incubation experiment with rice straw biochar, and concluded that the adsorption capacity of rice straw biochar on Pb (II) gradually increased with the increasing of the biochar addition. To our knowledge, there are many literatures about the effect of crop straw biochars on adsorption characteristic of heavy

†To whom correspondence should be addressed.
E-mail: luping@njnu.edu.cn
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Table 1. Proximate analysis and ultimate analysis of the biomass

Biomass	Proximate analysis wt/%				Ultimate analysis wt/%					$Q_{net.ad}$
	M_{ad}	A_{ad}	V_{ad}	FC_{ad}	C_{ad}	H_{ad}	O_{ad}^{*}	N_{ad}	S_{ad}	MJ/kg
PB	8.22	1.50	77.19	13.19	45.00	4.11	39.99	0.60	0.58	16.81
WH	10.79	27.47	57.66	4.08	30.99	4.84	23.26	2.43	0.22	16.77
CS	3.79	4.60	70.11	21.50	40.73	6.22	44.57	-	0.09	15.68

*Obtained by subtraction

metals in soil, however, the reports are contradictory depending on biomass source materials, preparation temperature, biochar addition and the individual metals concerned [14-17], it is worth to carry out further research.

In this study, biochars were prepared from typical biomass materials including poplar branch (PB), water hyacinth (WH) and corn straw (CS) in a fixed-bed pyrolyzer at pyrolysis temperatures of 300-700 °C. The objective is to study the effects of biochar species, pyrolysis temperature and biochar addition on adsorption characteristics of heavy metals of Pb and Zn in contaminated soil.

MATERIALS AND METHOD

1. Sample Preparation and Analysis

The proximate analysis and ultimate analysis on air dry basis (ad) of three biomass called poplar branch (PB), water hyacinth (WH) and corn straw (CS) are listed in Table 1. In which, M, A, V and FC represent the weight percentage of moisture, ash, volatile matter and fixed carbon in raw biomass, respectively; C, H, O, N and S represent the weight percentage of elements of carbon, hydrogen, oxygen, nitrogen and sulfur in raw biomass, respectively; and $Q_{net.ad}$ is the lower heating value of test biomass. Biomass samples was air-dried and sieved to the particle sizes less than 10 mm. The biochars were prepared in a fixed-bed pyrolyzer at conditions of pyrolysis temperature of 300, 500 and 700 °C, residence time of 30 min and continuous supplying of N_2. The biochars prepared from different biomass and pyrolysis temperature were called as PBC300, WHC500, CSC700, etc., in which, PBC, WHC and CSC represent PB biochar, WH biochar and CS biochar, respectively; 300, 500 and 700 denote the pyrolysis temperature. The biochars were ground to the particle size of less than 0.177 mm and dried 2 h at 45 °C for adsorption test.

The N_2-adsorption isotherms of biochars at different pyrolysis temperatures was evaluated at 77 K with relative pressures (P/P_0) up to 0.995 by using a high-speed surface area and pore size analyzer (Nova-1000e, Quantachrome). The biochar samples were outgassed at 393 K for 24 h before the N_2 adsorption. The specific surface areas (S_{BET}) were determined at P/P_0=0.05-0.30 according to the Brunauer-Emmett-Teller (BET) method. The total pore volume (V_T) was directly measured at P/P_0=0.995 and average pore width (D_a) was calculated based on the total pore volume and BET surface area [18,19]. Mineral compositions of the biochars were identified by X-ray diffraction (XRD, D/Max-2500, Rigaku) with Cu Kα radiation operated at 40 kV and 200 mA. The surface functional groups on biomass and their biochars was characterized by Fourier transform infrared spectroscopy (FTIR), and the spectra of samples were determined from 400 to 4,000 cm^{-1} wave number by KBr method on a Nicolet NEXUS 670 FTIR spectrometer (Thermo Electron Corporation, USA) [8].

The soil sample was collected from vegetable garden at lead-zinc-silver mining area, Nanjing China. The soil sampling depth is 0-20 cm. Soil sample was air-dried, crushed and ground to the particle size less than 0.105 mm. Before adsorption test, each soil sample was dried at 45 °C for 2 h. The concentrations of heavy metals in soil samples were analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES, Prodigy, Leeman Labs) [20]. The soil samples were digested in a series of solutions of $HCl/HNO_3/HF/HClO_4$ prior to determination of heavy metals. The background concentrations of heavy metals of Pb, Zn, Cu, As and Cd in the soil sample are 434, 353, 50, 447 and 1.2 μg/g, which is 8.68, 1.41, 0.50, 17.88 and 3.0 times higher than that of the second level criterion regulated by the China standard of Environment Quality Standard for Soil (GB 15618-2008) [21], respectively. The leaching experiment of original soil sample in 0.01 mol/L HCl solution shows the mobility of Cu, As and Cd is very lower, whose leaching rates are 16.0%, 0.12% and 15.8%. Therefore, HMs of Pb and Zn were selected to evaluate the adsorption characteristics of biochars on heavy metals in the soil sample.

2. Adsorption Experiment Procedure and Evaluation Method

Fig. 1 shows the flow diagram for soil HMs adsorption experiments. The adsorption experiment procedure is briefly described as follows: (1) Weigh 5 g soil sample and a certain amount of bio-

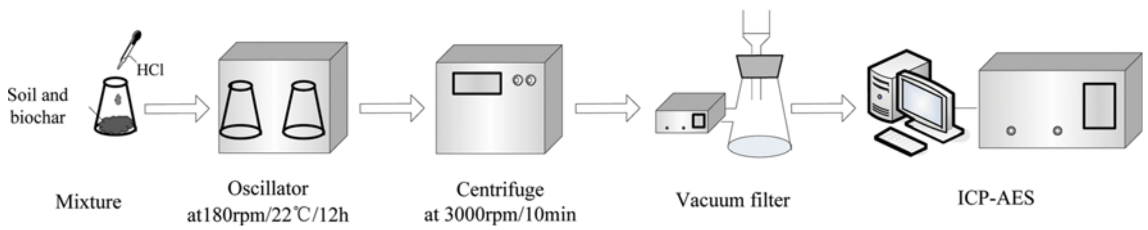

Fig. 1. The experimental flow diagram for adsorption of heavy metals in soil.

char (mass fraction of biochar to soil is 1%, 5% and 10%, respectively), and thoroughly mix them with 50 mL HCl (0.01 mol/L) in a 200 mL oscillation flask; (2) move the mixture to a 50 mL centrifuge tube, and put it into the thermostatic oscillator (ZHWY-100B, Shanghai Zhicheng Co., China) to shake at 22 °C and 180 rpm for 12 h; (3) take out the tube and centrifuge it in a table centrifuge (TDL-40B, Shanghai Leigu Co., China) at 3,000 rpm for 10 min; (4) obtain the leachate through vacuum filtration (membrane pore size is 0.45 μm) and analyze the concentrations of Pb and Zn by ICP-AES. Blank Experiments of soil sample without adding biochar were also carried out following the same procedure. The residue after adsorption is also digested and analyzed by ICP-AES. Mass balance of HMs (Pb and Zn) was calculated based on concentration of HMs in the soil sample, the leachate and the residue. The data for mass balance of HMs of Zn and Pb were in the range of 80%-120%, which indicates the results are believable.

Each treatment was repeated three times and the average values were obtained for the final analysis. The adsorption properties of biochar on soil HMs are characterized by adsorption efficiency (η_s, %) and the adsorption capacity (Q_s, μg/g). The adsorption efficiency can be calculated by Eq. (1), and the adsorption capacity is defined as the total amount of HMs adsorbed by unit biochar and is calculated by Eq. (2).

$$\eta_s = \frac{C_0 - C_t}{C_0} \times 100\% \tag{1}$$

$$Q_s = (C_0 - C_t) \times m_0 / m_t \tag{2}$$

Where C_0 and C_t are the concentration of each HMs in soil leachate at the condition of without or with biochar addition, respectively, μg/g. m_0 is the amount of the soil in each experiment, g. m_t is the biochar addition in each experiment, g.

RESULTS AND DISCUSSION

1. Effect of Biochar Species on the Adsorption of HMs in Soils

Fig. 2 shows the effect of biochar species on the adsorption characteristics of soil HMs, in which, the pyrolysis temperature is 500 °C, and the biochar addition amount is 5%. It shows in Fig. 2(a) that three biochars can reduce the concentrations of Zn and Pb in soil leachate. WHC addition obtains the best HMs fixation effect, whose concentrations of Zn and Pb in soil leachate decrease from 83.30 μg/g and 25.54 μg/g (without biochar addition) to 65.11 μg/g and 14.16 μg/g, respectively. As shown in Fig. 2(b)-(c), WHC and CSC have basically the same adsorption efficiency on Zn in soil, which is about 20%, and the corresponding adsorption capacity is about 360 μg/g; however, PBC has the lowest adsorption efficiency on Zn in soil, which is about 13%, and the corresponding adsorption capacity is about 216.94 μg/g. WHC has the highest adsorption efficiency on Pb in soil, which is about 44.57%, and the corresponding adsorption capacity is about 227.65 μg/g.

The experimental results indicate that three biochars can effectively adsorb Zn in soil and the adsorption abilities of WHC and CSC are better than that of PBC. Meanwhile, WHC gets the best adsorption capacity on Pb in soil, however PBC and CSC have almost no adsorption capacity on Pb in soil. The phenomena agree with the results of Xu and Zhao [4], who found that the adsorption efficiency of crop straw char on Cd (II) was much larger than that on Cu (II) and Pb (II), and the adsorption capacity of crop straw char on Cd (II) was significantly greater than that of rape straw char. This suggests that biochar has a strong selective adsorption on soil HMs in natural multi-metal environments [22]. Chen et al. [23] reported that heavy metals rarely occur alone, and their associations and interactions with each other as well as other components in natural environments are known to influence their transport and fate between aqueous solutions and solid surfaces. In this test, the concentration of Zn (83.31 μg/g) is pretty higher than that of Pb (25.54 μg/g) in original soil sample. The high mobility of Zn is much more helpful to adsorb by biochars, which resulting in the high adsorption efficiency and adsorption capacity for three biochars. There-

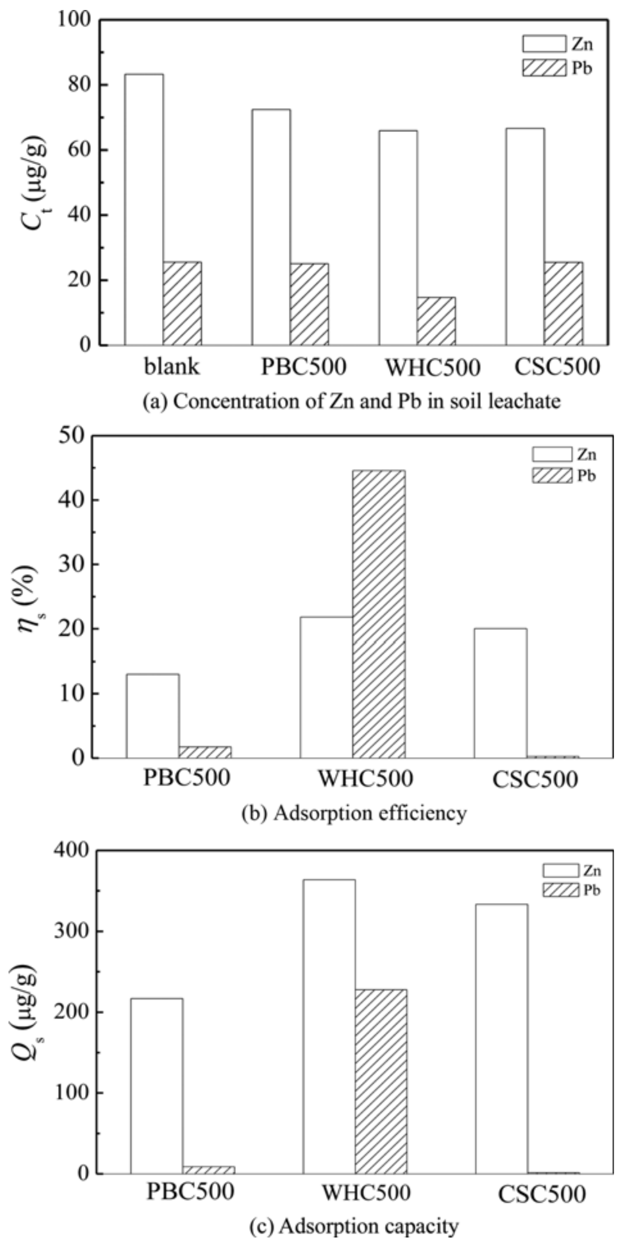

Fig. 2. Effect of biochar species on the adsorption of HMs in soils.

fore, PBC and CSC present low adsorption ability on Zn in soil due to dual influences of competition of Zn and low mobility of Pb in soil.

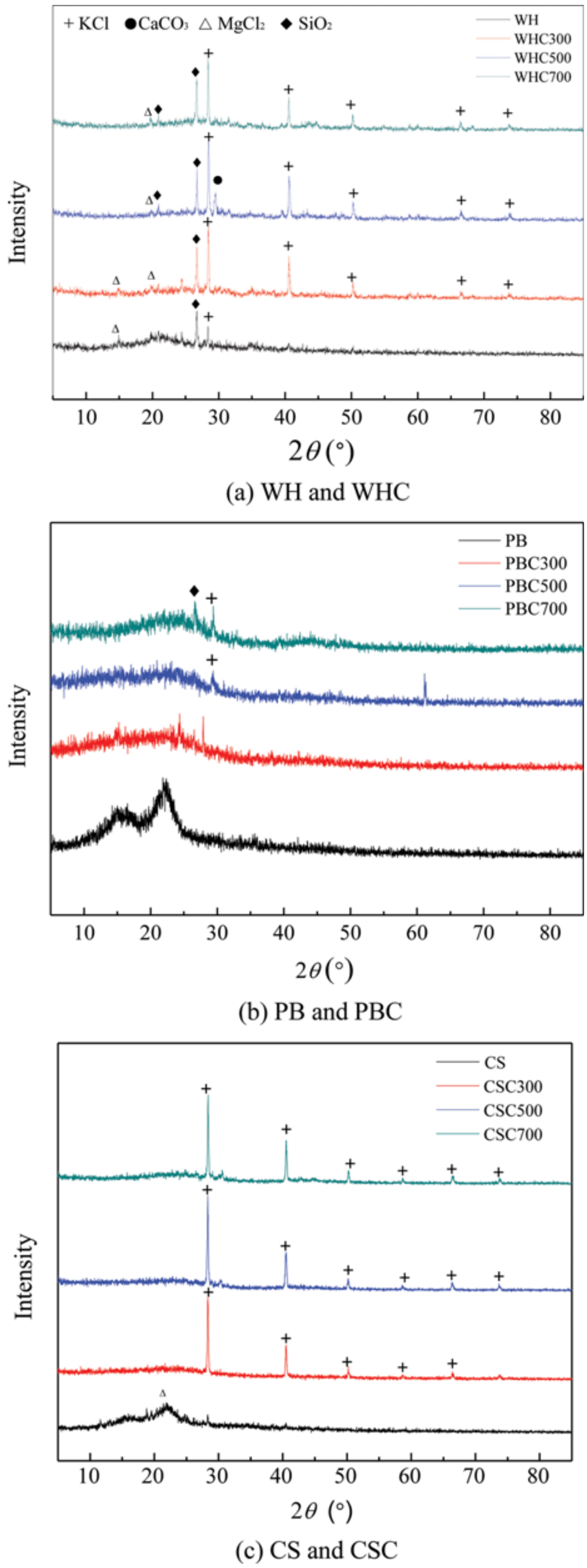

Fig. 3. XRD curves of the biomass and their biochars.

Ahmad et al. [24] postulated that ion exchange, electrostatic interaction (anionic metal attraction, cationic metal attraction) and precipitation are prevailing mechanisms for the remediation of inorganic contaminants by biochar, and pointed out biochar is efficient for inorganic contaminants due to the presence of more O-containing functional groups and the greater release of cations. Fig. 3

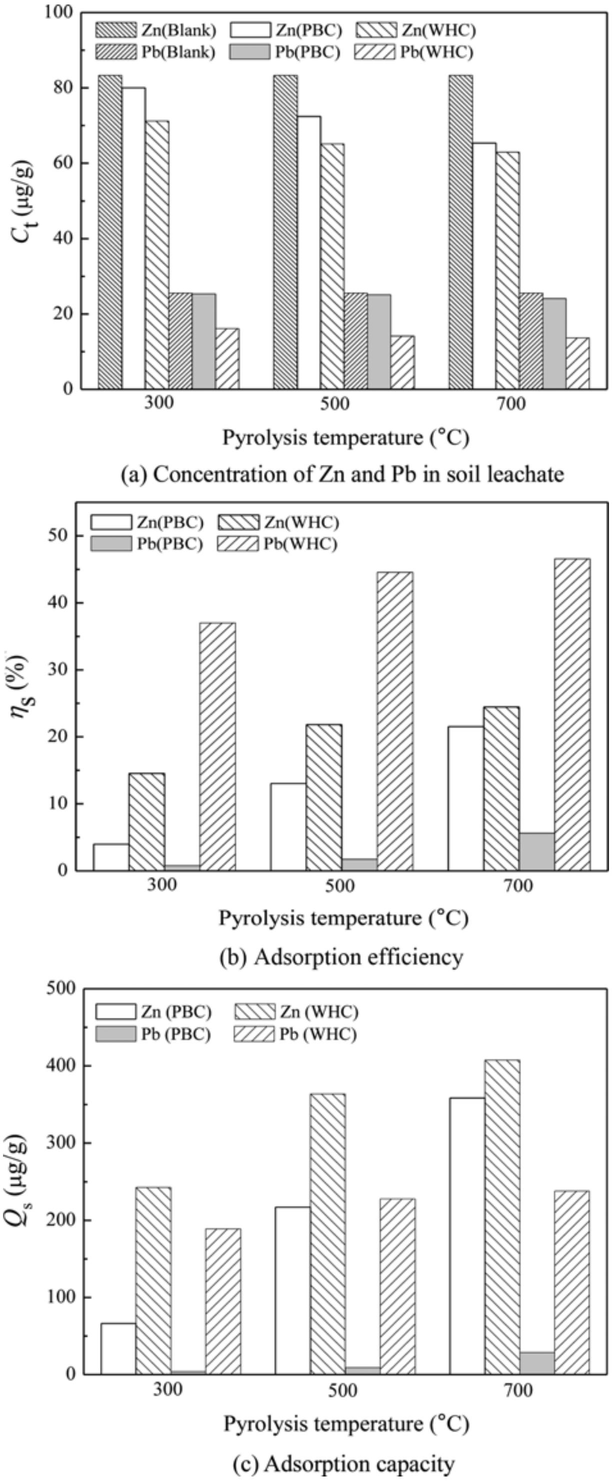

Fig. 4. Effect of pyrolysis temperature on the adsorption of HMs in soils.

shows XRD curves of three biomass and their biochars pyrolyzed at 300, 500 and 700 °C. It shows that biochars contain KCl, $CaCO_3$, $MgCl_2$ and other substances, and the quantity of elements of K, Ca and Mg in WH chars is much higher than that in other two biochars, which can provide amounts of cations to promote ion exchange, and lead to strong adsorption ability on heavy metals in soil.

2. Effect of Pyrolysis Temperature on the Adsorption of HMs in Soils

Fig. 4 shows the effect of pyrolysis temperature on the adsorption characteristics of HMs in soil, in which, the biochars are PBC and WHC, pyrolysis temperatures are 300, 500 and 700 °C, and biochar addition amount is 5%. It shows in Fig. 4(a) that the concentrations of Zn and Pb in soil leachate decrease while adding PBC and WHC pyrolyzed at different pyrolysis temperature into the soil, and the minimum values of Zn and Pb in soil leachate are 62.29 μg/g and 13.65 μg/g, respectively. As shown in Fig. 4(b)-(c), the adsorption efficiency and adsorption capacity of biochars on Zn and Pb in soil increase gradually with increasing pyrolysis temperature. The maximum adsorption efficiency and adsorption capacity for Zn in soil are 24.46% and 407.58 μg/g, and the maximum adsorption efficiency and adsorption capacity for Pb in soil are 46.56% and 237.79 μg/g, respectively. The experimental results indicate that pyrolysis temperature has a great influence on the adsorption properties of biochars on HMs of Zn and Pb in soil. This is consistent with the results of Ding et al. [25], who applied biochars pyrolyzed at temperatures of 300, 500 and 700 °C into the soil samples contaminated by Pb and Cd, and concluded the pyrolysis temperature for reducing bioavailability of target heavy metals by biochars amendments is in the order of 700 °C>500 °C>300 °C. Furthermore, WHC prepared at temperatures of 500 and 700 °C on adsorption of Zn and Pb in soil almost obtains the same adsorption ability, which indicates that the moderate pyrolysis may be a good choice for HMs adsorption by WHC.

The pore structure parameters of some biochars are listed in Table 2. It can be seen that biomass pyrolyzed at high temperature normally behaves the high specific surface area (S_{BET}), the large total pore volume (V_T) and the small average pore width (D_a), which

Table 2. Pore structure parameters of the biomass and their biochars

Biochar	S_{BET}	V_T	D_a
	m^2/g	mm^3/g	nm
CS	1.88	3.44	7.32
CSC300	8.81	9.33	6.25
CSC500	102.43	67.28	2.63
CSC700	8.38	11.01	5.25
WH	3.46	10.77	12.45
WHC300	4.77	20.05	16.83
WHC500	7.34	22.95	12.50
WHC700	36.16	47.10	5.21
PB	2.17	5.32	9.83
PBC300	-	-	-
PBC500	219.38	114.8	2.09
PBC700	256.86	131.80	2.05

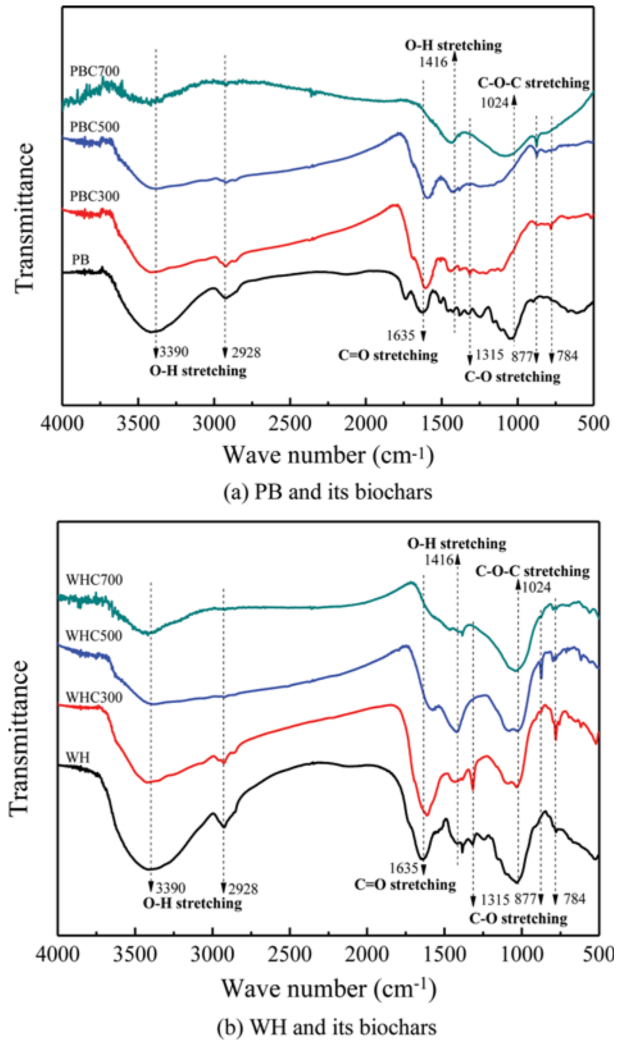

Fig. 5. FTIR curves of biomass and their biochars.

indicate high pyrolysis temperature is helpful to form the developed pore structures. Particularly, S_{BET} and V_T of WHC and PBC increase with increasing pyrolysis temperature, and PBC pyrolyzed at 700 °C gets the highest S_{BET}. However, S_{BET} and V_T of CSC increase first and decrease later with increasing pyrolysis temperature, and the best pore structure of CSC is obtained at 500 °C. In a word, S_{BET} of WHC is pretty lower than that of other two biochars, but WHC shows the best adsorption ability. This indicates the specific surface area of biochar is not the only factor for good adsorption on HMs in soil. Fig. 5 shows the FTIR spectra of two raw biomass (PB and WH) and their biochars pyrolyzed at 300, 500 and 700 °C. It can be seen that raw biomass and biochars contain many surface oxygen-containing groups, such as hydroxyl group, carboxyl group, phenolic hydroxyl, lactone and carbonyl group, etc., and the intensity and number of oxygencontaining functional groups of PBCs become weaker with increasing pyrolysis temperature, which is consistent with the results by Li et al. [26]. However, pyrolysis temperature has little influence on surface oxygen-containing groups of WHCs. This could promote adsorption of heavy metals through cationic metal attraction according to Ahmad's mechanism

[24]. Therefore, combined with the results of Fig. 3, Fig. 5 and Table 2, the good adsorption of WHC500 on HMs of Zn and Pb in soil may be attribute to the co-effects of the specific surface area, pore structure, surface oxygen-containing functional groups and exchangeable metal ions of biochar.

3. Effect of Biochar Addition on the Adsorption of HMs in Soils

Fig. 6 shows the effect of biochar addition on the adsorption of HMs in soil, in which, the biochar is WHC, the pyrolysis temperature is 700 °C, and the biochar additions are 1%, 5% and 10%, respectively. It can be seen from Fig. 6(a) that the concentrations of Zn and Pb in soil leachate reduce when adding 1%, 5%, 10% of WHC into the soil sample, the corresponding minimum values of Zn and Pb are 55.87 μg/g and 1.55 μg/g, respectively. This is consistent with the results by Li et al. [12], who found that the concentrations of availability of Cd, Cu, Pb and Zn in soil decrease significantly while addition of rice straw biochar increases from 0 to 5%. Fig. 6(b)-(c) shows that the adsorption efficiencies of WHC on Zn and Pb in soil increase with the increasing of biochar addition, the adsorption efficiency of Pb is significantly larger than that of Zn at WHC addition of 5% and 10%, and the maximum adsorption efficiencies of Pb and Zn are 93.93% and 32.92%, respectively. However, with the increasing of the WHC addition, the adsorption capacity of WHC on Zn in soil decreases from 1,408.70 μg/g to 274.26 μg/g, and the adsorption capacity of WHC on Pb in soil keep the same level of 240 μg/g. These indicate that WHC has a better fixation ability on Pb in soil with increasing the biochar addition, but too much WHC addition is not helpful for increasing adsorption capacity. Ni and Weng [27] carried out heavy metals adsorption with magnetic black carbon at the condition of 30 reaction days, soil moisture content of 20% and the soil pH of 6.0, and also demonstrated the adsorption efficiency of Pb and Cd in soil increased with addition of magnetic black carbon, the maximum adsorption efficiencies of Pb and Cd (67.45% and 73.27%) were obtained at the addition amount of 8%, and too much addition of magnetic black carbon decreased the adsorption capacity of Pb and Cd. Therefore, the optimum biochar addition amount should be depended on the biochar cost and its adsorption ability on heavy metals in contaminated soil.

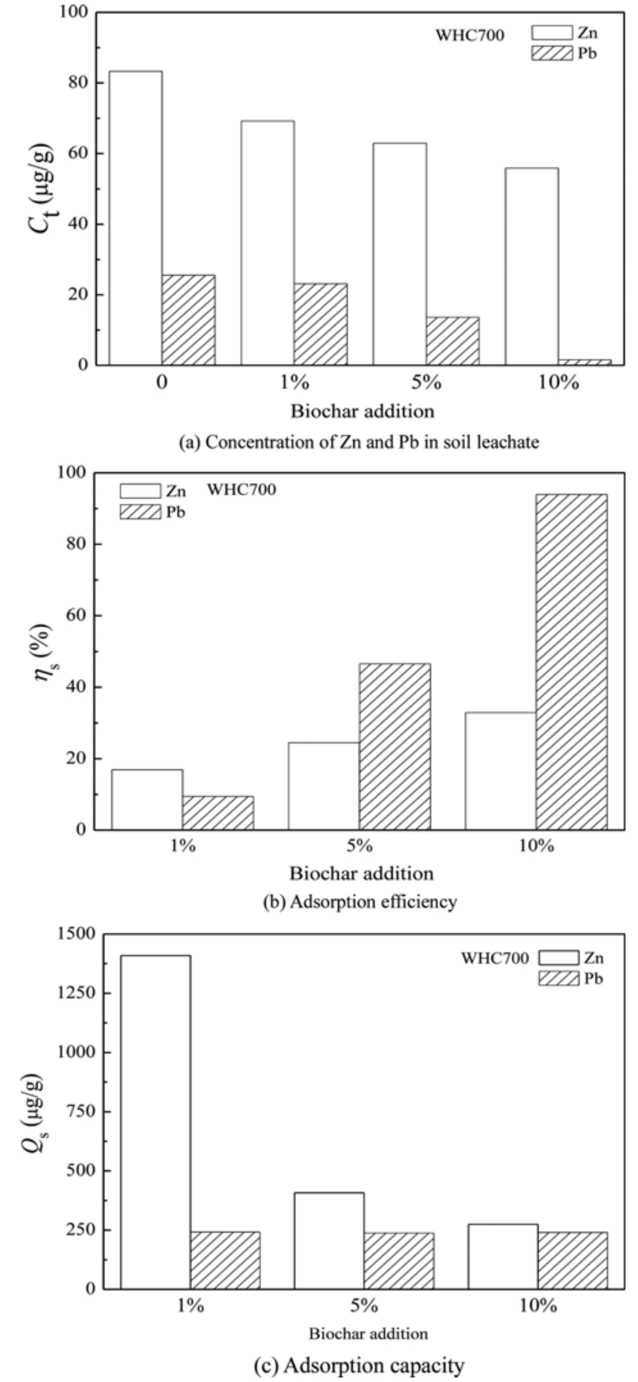

Fig. 6. Effect of biochar addition on the adsorption of HMs in soils.

CONCLUSIONS

1) Biochar species have significantly effect on adsorption of heavy metals of Zn and Pb in soil, and the best HMs fixation effect is obtained through WHC addition at pyrolysis temperature of 500 °C and biochar addition of 5%, whose adsorption efficiency on Zn and Pb is 21.83% and 44.57%, and the relative adsorption capacity of Zn and Pb is 227.65 μg/g and 363.76 μg/g, respectively.

2) Pyrolysis temperature is an important factor affecting the adsorption performance of biochar. The adsorption efficiency of biochar on of Pb and Zn in soil increases gradually with increasing pyrolysis temperature, and the adsorption efficiency of WHC pyrolyzed at 500-700 °C almost gets the same results, which indicates the moderate pyrolysis may be a good choice for biochar preparation.

3) The adsorption efficiency of soil HMs increases and adsorption capacity of soil HMs decreases with increasing WHC addition. WHC has a better fixation ability on Pb in soil with increasing the biochar addition, and the adsorption efficiency of Pb is 93.9% while adding 10% of WHC into the soil.

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