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Adsorption of Pb^{2+} in waste-water by Biochars Derived from Different Crop Residues

YAFEN HAN AND QI LI

Anhui Engineering & Technological Research Center for Coal Exploration-China, School of Environment and Survey Engineering, Suzhou University, Suzhou, 234000

Email: *hanyafen2008@163.com*

Abstract: Nine types of biochar (WH500/600/700、CO500/600/700、SO500/600/700) were prepared by pyrolyzing crop straw(like wheat(WH), corn(CO) and soybean(SO)) at different temperatures (500，600 and 700 °C)under the limited oxygen condition. The absorbability of various biochars to Pb^{2+} in waster-water and its influential factors were investigated. The results indicated that the biochars pyrolyzed at 600 °C showed the highest adsorption rate to Pb^{2+} in waster water, and the adsorption percentage decreased in the order of CO600 (81.9%) > SO600 (64.9%) > WH600 (52.9%). The sorption capacity of Pb^{2+} was enhanced with the input amount of biochar increased. It should be noted that the adsorbability increased slowly when the adsorbent was fewer, the removal rates of Pb^{2+} from waster-water increased significantly when the input amount was over $3.3g{\cdot}l^{-1}$, and equilibrium could be achieved at $10g{\cdot}l^{-1}$. Biosorption of Pb^{2+} was a rapid reaction, and the biosorption of Pb^{2+} on the studied biochars can be segmented into two phases: the first speedy process and the next slow process; Only the pseudo-first-order model presented the best appropriate for all of the tested data and equilibrium could be achieved within 30 to 60 min. pH had great influence on biosorption of the metals, the adsorption of Pb^{2+} was enhanced with the raise of pH, and adsorption percentage of Pb^{2+} increased rapidly when the initial pH increased from 4 to 6. Under the same conditions, the maximum sorption capacity of Pb^{2+} by WH, CO and SO decreased in the order of corn>soybean>wheat. These observations provided useful information in precisely predicting the adsorption behavior of biochar to Pb^{2+} in waster water.

Keywords: *Biochar; Crop Residues; Pb^{2+}; Adsorption; Influential factor*

1. Introduction

In recent years, environmental pollution is now getting worse as Increasing industrialization, especially in China. Of all pollution forms, heavy metals contamination(such as Pb, Cu, Cd and so on) has become a serious crisis because these metals are toxic and non-biodegradable[1], so that it can be absorbed and concentrated into in a variety of organisms, water and plants for a long time. When metal's concentration surpass the natural baseline level, they have detrimental effects on plants, animals and humans[2]. Furthermore, heavy metals are one of important and indispensable raw materials in many industries such as mining stone processing, tannery, fertilizer production, which cause direct or indirect damages to the groundwater and soil[3]. Many conventional and modern methods for metals removal have been used, e.g., ion exchange, precipitation, adsorption, membrane filtration, however, many of these techniques are complex and expensive. So adsorption of heavy metals onto low-cost materials have been testified to be a simple, powerful and economical method[4,5]. Currently, A large variety of adsorbents have been studied and used for heavy metals removal (Pb, Cu, Cd, Cr, etc). Academicians have lately focused onto crop biochars as a new material for the removal of metals. Application of crop biochars also have the actively effects on environmental protection. Biochars have been applied in reducing CO_2 emissions [6], improving crop yields quality [7], purifying waste water and controlling environmental pollution [8]. The mechanisms for Pb^{2+} adsorption by biochars have three main types, including surface adsorption, appearance of Pb precipitates with adsorbent and harmony of Pb^{2+} with electrons of biochar [9]. These different mechanismsare probably attributed to the various characteristic of crop biochars, which mainly rely on the types of raw material and pyrolysis temperatures. Owing to the reason known to all biochar's characteristic are extremely controlled by pyrolytic temperature. However, very few studies apply oneself to discuss the relation between Pb^{2+} adsorption and crop biochar characteristic in view of different pyrolytic temperatures [10-11]. Therefore, establishing the detailed process for Pb^{2+} adsorption on biochars based on various materials at different temperatures is necessary. In this paper, limited oxygen pyrolysis was applied to produce a series of crop biochars at 500–700°C, including wheat, corn, soybean straw. Adsorption and its influential factors of Pb^{2+} to the biochar in mine waster-water were investigated in different reaction conditions. These researches provided useful information in exactly predicting the adsorption behavior of Pb^{2+} in waster water with biochar for preparation of adsorbents.

2. Materials and Methods

2.1. Materials and Reagents

The materials study need included waster-water and crop residues, waster-water was collected from subsurface mine in Luling mines of Suzhou city, China. Waster-water was filled fresh plastic bottles and was transported to of State Engineering & Technological Research Center for Coal Mine Water Disaster Prevention immediately, subsequently, was filtered through a 0.45-µm membrane and collected into the bottle for further investigations. Wheat straw, corn straw and soybean straw were collected from cropland in a suburb of Suzhou City, China. These crop residues were used as raw materials to produce biochar.

Lead standard sample (1000mg/L), hydrochloric acid, nitric acid, and sodium hydroxide were purchased from Shanghai Chemical Reagent Company (Shanghai, China). All the solutions were prepared with water purified in a Milli-Q system. All the chemicals were of analytical grade and were used without further purification. A stock solution containing 1000 mg/L of ions (Merck) was used. By dilution with water, solutions containing different concentrations of Lead (II) ions were prepared.

2.2. Modification of Biomass Material

Wheat straw, corn straw and soybean straw were separately washed with tap water five times to remove the attached dust, then dried at 70~80°C for more than 48 h to obtain crop-straw-derived black carbon. Afterwards, the biochars were produced at 500, 600, 700°C by pyrolyzing crop straw as described by Zheng et al [12]. The wheat-derived biochars (WH), corn-derived biochars (CO) and soybean-derived biochars(SO) were grinded to powder and passed through 0.15 mm sieve (100 mesh) prior to the following sorption experiments. Biochar samples from WH, CO and SO are hereafter named as WHX, COX and SOX, respectively, where X represents pyrolytic temperature.

2.3. Adsorption Experiment

All adsorption tests were conducted in light-resistant place in 25 degrees Celsius, and were oscillated at 110 rpm for 1 min. Pb^{2+} stock solution was prepared using mine waster-water, concentration of which was measured by flame atomic absorption spectrometry(FAAS). Adsorption experiments of Pb^{2+} to the biochar in waster-water were simulated in four different conditions, such as different fires, different adsorption time, change of adsorbent weight, pH effect. Each fraction was defined as follows:

Influence of pyrolyzing temperature: 0.1g of CO500/600/700, WH500/600/700, SO500/600/700 were added into polythene plastic centrifuge tubes and 10ml of prepared waster-water was added into polythene plastic centrifuge tubes with a pipette, and the vials were shaken at 110rpm at 25°C for 2h, After shaking, all mixtures were separated by centrifugation at 1000 rpm for 20 min, and filtered through a 0.45µm Millipore filter. The Pb^{2+} concentration in the filtrate was analyzed using FAAS. All samples were run along with blanks without biochar samples or Pb^{2+}. Because the mass loss of Pb^{2+} was negligible, sorption of solutes by biochars was determined by mass balance.

Influence of adsorbent mass: the adsorption capacities of Pb (II) ions on WH600, CO600 and SO600 were obtained according to the initial runs of adsorbent capacity of each ions. The adsorption were carried out at initial pH of 6 for each solution. During experiments, the biochar weights from 0.01 to 0.3g range (0.01g, 0.05g, 0.1g 0.15g, 0.2g, 0.3g) was used. The single adsorption system was consist of 10 mL of waster-water and different weight of biochar. Solutions were agitated on a shaker at 110rpm with temperature of 25°C and the equilibrium time of 2h. After shaking, all mixtures were separated by centrifugation at 1000 rpm for 20 min, and filtered through a 0.45µm Millipore filter.

Kinetic study (different adsorption time): In the adsorption study of Pb (II) ions onto WH600, CO600 and SO600 materials a batch equilibration technique was used. Firstly, the effect of time on metal ions adsorption onto biochars was investigated vs. the adsorption time from 10 to 300 min range (10min, 30min, 60min, 120min, 180min, 300min) was used. The initial the solution volume of each ion in the single element solutions were prepared to be at the level of 10mL, and the studied adsorbents masses were around 0.1 g ± 0.03 g. The optimal pH in case of each metal ion were adjusted to 6.0. Kinetics solutions were agitated on a shaker at room temperature 25°C at 110 rpm constant speed. After shaking, all mixtures were separated by centrifugation at 1000 rpm for 20 min, and mixtures were filtered with 0.45 µm PTFE hydrophobic syringe filters (Alfa Chem, Poland), then the filtrates were used for analyzing the heavy metals concentration. The last step was the same for each adsorption steps.

pH effect: the impact of pH of solution on Pb (II) ions adsorption onto examined biochars was carried out. In this step, the initial pH value of the solutions was adjusted to 2-9 range in case of Pb (II) keeping the heavy metal ions volume (15mL for WH600, CO600 and SO600) and adsorbent mass at constant value (0.1 g ± 0.03 g). Solutions were agitated on a shaker with temperature of 25°C at 110 rpm and the equilibrium time of 2 h. After shaking, all mixtures were separated by centrifugation at 1000 rpm for 20 min, and mixtures were filtered with 0.45 µm PTFE hydrophobic syringe filters (Alfa Chem, Poland). After reaching the equilibrium state by studied systems the pH was measured to detect the changes in the solutions. On the basis of obtained results the

relation between initial and equilibrium pH was evaluated.

To improve the accuracy of results, all chemicals used in the experiment were of analytical grade, and all the glassware and centrifuge tubes were soaked in dilute nitric acid for 24 h followed by rinsed with MilliQ water three times before use. During each adsorption procedure, reagent blanks were prepared for correcting blank and eliminating analytical bias. Each sample was determined with three replicates to test the reproducibility, and relative standard deviation was less than 10%.

3. Results and Discussion

3.1. The effect of pyrolytic temperature to Pb^{2+} adsorption

The differences of Pb^{2+} adsorption of WHX, COX and SOX are listed in table 1 at different pyrolytic temperature. There are obvious distinctions between these pyrolytic temperatures for each test biochar. Taking into consideration of the previous researches looking for the adsorption of metal ions at different pyrolytic temperature, the findings suggested that adsorption ability of heavy metals differed greatly in different pyrolytic temperature.

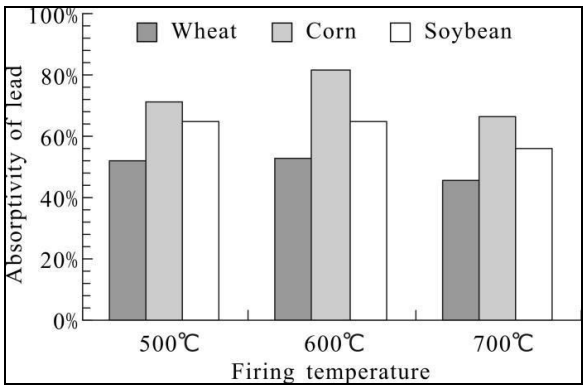

Fig.1 *Pb^{2+} adsorption rate on biochars at different pyrolytic temperature*

For figure 1, adsorption percentages of Pb^{2+} on WHX, COX, and SOX decreased in the order of WH600 (52.9%) > WH500 (51.6%) > WH700 (45.4%) , CO600 (81.9%) > CO500 (70.6%) > CO700 (66.4%) , SO600 (64.9%)>SO500 (64.8%) > SO700 (55.8%), respectively. These results indicated that 600-temperature biochars had the highest adsorptivity, 500-temperature biochars came second, for 700-temperature biochars the lowest. The data also reflected that carbonization degree could be related to the pyrolytic temperature, so we choose the 600-temperature biochars as adsorbing material for further analysis. According to adsorption of different crop biochars, adsorption percentages of Pb^{2+} on WH600, COX600, and SOX600 decreased in the order of CO600>SO600>WH600 under same condition, respectively. The differences between the adsorption results are possibly caused by the different physics and chemical character of the studied materials.

3.2 The Effect of Different Initial adsorbent dosage

The adsorptions of Pb^{2+} of WH600, CO600 and SO600 in different initial dosage are listed in Fig. 2. From Fig. 2, the adsorption capacity of Pb^{2+} of WH600, CO600 and SO600 increased with increasing adsorbent, and the change trend of the adsorption capacity that happen as the adsorbent increases is very similar for three different biochars (Fig. 2). Specifically, it can be indicate that the adsorption capacity of Pb^{2+} onto WH600, CO600 and SO600 was fewer than $3.3g{\cdot}l^{-1}$ in waster-water, the adsorption rates of Pb^{2+} on WH600, CO600 and SO600 were 20.22%, 26.90% and 23.65%, respectively. The removal rates of Pb^{2+} from waster-water on three biochars increased dramatically when adsorbent mass changed from $3.3g{\cdot}l^{-1}$ to $10g{\cdot}l^{-1}$. When adsorbent mass reached $10g{\cdot}l^{-1}$, adsorption percentages of Pb^{2+} on WH600, COX600, and SOX600 were 68.9%、91.4%、81.1%, respectively. The investigated results indicated that crop biochars added in waster-water was over $10g{\cdot}l^{-1}$ for the best adsorption effect. when adsorbent was more than $10g{\cdot}l^{-1}$, the removal rate remains largely unchanged and the balanced state is achieved. After he balance state is achieved, the adsorbent mass and removal rate were estimated again to entirely amplify the above change regulation , and the relationship between the original adsorbent and the removal rate is charted in Fig.2. According to adsorption of different crop biochars, the maximum adsorption percentages of Pb^{2+} on WH600, COX600, and SOX600 decreased in the order of CO600>SO600>WH600 under same condition.

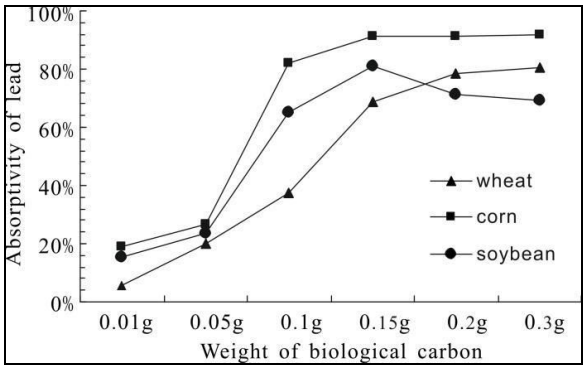

Fig.2 *The adsorption rate of Pb^{2+} in different initial biochar*

3.3. Adsorption Kinetics

The adsorption effect of Pb^{2+} was researched as a function of time, and the results are showed in Fig. 3. Other things being equal, Pb^{2+} adsorption rate increased along with the lapse of time. The biosorption of Pb^{2+} on the studied biochars was two-phase process, which is in keeping with aforementioned research [13-14]. The first speedy

process is put down to the surface sorption of metal ions on biochar surface. The next slow process is linked with the change of adsorbent metal ions from outside to inside of the biochar [15]. The absorption time necessitated to approach balanced state was different for the studied biochar(WH600, CO600, SO600). The adsorption kinetics of Pb^{2+} on WH600 and CO600 was faster than on SO600. The difference could be interpreted by the outside character of biochars. Adsorption kinetics deeply relies on depends chemical and physical properties of crop biochars, which affect the sorption mechanism of Pb^{2+}. The balanced state is arrived more quickly with WH600 and CO600 than with SO600. One reason may be that the content of hydroxyl groups on the WH600 and CO600 surface is higher than on the SO600 surface, and the most arresting feature of hydroxyl groups is lower forcible energy than carboxyl groups [6], and this consent Pb^{2+} to restrain more promptly to hydroxyl groups. Another reason should be adsorptivity of biochar surface. For Pb^{2+}, 30min was enough to arrive the balanced state using WH600 and CO600, however, 60min was adequate to reach the balanced state, which was showed in fig.1. So the sorption kinetics of Pb^{2+} on SO600 is slowest of the crop biochars tested. The differences between the studied biochars may be illustrated by different physicochemical characteristic.

***Table** 1: Dynamic parameters for adsorption kinetics simulated by different equations*

Metal	biochar	kinetic model					
		Pseudo-first order			Pseudo-second order		
		k_1	R^2	a_{eq}	k_2	R^2	a_{eq}
Pb	WH600	0.267±0. 095	0.925	1.37±0.08	0.012±0. 0021	0.764	1.45±0.09
	CO600	0.207±0. 051	0.952	3.12±0.07	0.0035±0. 0011	0.896	3.32±0.11
	SO600	0.367±0. 035	0.947	2.48±0.20	0.0143±0. 0007	0.843	2.67±0.07

***Annotation:** $k_1(min^{-1})$--the rate constants of the pseudo-first order equation, $k_2(ug.g^{-1}.min^{-1})$--the rate constants of pseudo-second-order equation, R--correlation coefficient, a_{eq}--the amount adsorbed mass at equilibrium time $(ug.g^{-1})$.*

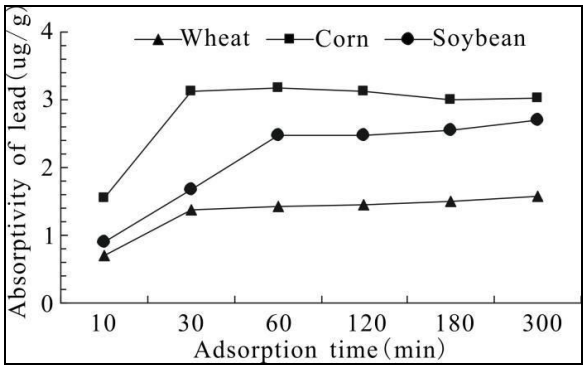

***Fig.3.** Pb^{2+} Adsorption on biomass materials as reaction time*

Dynamic parameters for adsorption kinetics were obtained by studying adsorption systems in table 1. Only the pseudo-first-order model presented the best appropriate for all of the tested data. The information displays that correlation coefficients are higher than 0.925 for all biochars (Table 1). Thinking over the previous research looking for the sorption of metal ions, the findings suggested that the first-order adsorption kinetics are in according with results investigated by other academician[16-17]. It was proposed that the kinetic model of heavy metals ensuing pseudo-first-order is influenced by the time of attain equilibrium. The differences between k_1 values mostly connected to the rate of the adsorbent process, This higher k_1 value showed in Table 1 is related with a faster adsorbent process arriving the balanced state. k_1 values of WH600, CO600 and SO600 were approximately 0.267, 0.207 and 0.367 respectively. Adsorption process of Pb^{2+} on CO600 was the fastest to reach equilibrium status, WH600 came second, for SO600 the slowest. This dates could also be showed what the determined equilibrium adsorbed amount (a_{eq}) was what the maximum adsorption capacity was. According to the maximum adsorption capacity, the maximum adsorption capacity of Pb^{2+} on WH600, CO600 and SO600 were 1.57, 3.19, 2.69ug.g^{-1}, respectively.

3.4. The effect of pH

The effect of the original sample pH on the adsorption rate of Pb^{2+} is taken on in Fig.4. The removal rates of all the investigated samples(WH600, CO600, SO600) to Pb^{2+} were recorded from pH 2 to pH 9. In acidulous conditions, the groups on the surface of the biochar are stable, while ‘‘free” Pb^{2+} ions exist in the waster-water. As the pH increases, the surface group structure character changes. Biochars become instability, and the Pb^{2+} then bind to the WH600, CO600 and SO600 structure. Because the heavy metals precipitate as basic conditions changes, the relationship between the original pH and the adsorption rate of Pb^{2+} was analyzed within a finite range of pH values. The results show that the adsorption rate of the crop biochars increase as the pH increases (Fig.4). Based on Fig. 4, it may be presented that the optimum pH values to reach the most efficient adsorption of Pb^{2+} by WH600, CO600 and SO600 are 6 entirely. The change trend of the adsorption capacity that happen as the pH value increases is very similar for three different biochars (Fig. 4). At lower pH (pH < 4), the removal rate of three studied

biochars are very low and does not surpass 20%. When pH exceeds 4.0, the removal rate increases rapidly to the maximum value for three tested system. But when pH value exceeds 6.0, the removal rate remains largely unchanged and the balanced state is achieved. After the balance status is achieved, the pH and removal rate were estimated again to entirely amplify the above change regulation, and the relationship between the original pH and the balanced pH are charted in Fig.4. According to above mentioned data during the adsorption process, all the tested waster-water grew increasingly basic until the balanced pH value of 6 for Pb^{2+} ions was approached. These differing equilibrium pH values are probably related to pyrolyzing temperature, crop types and different metals [18-19]. For example, the maximum adsorption proportion of WH600, CO600 and SO600 to Pb^{2+} were 48.12%, 81.95%, 74.88% under the same conditions.

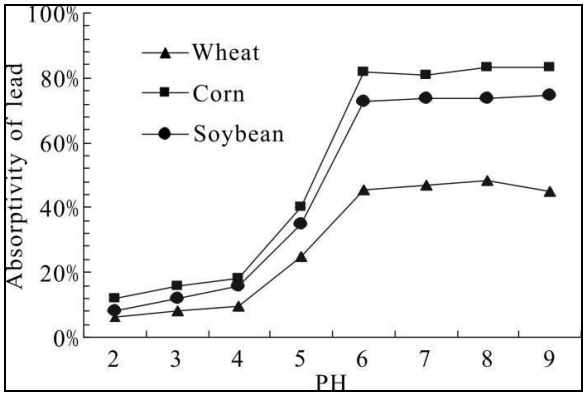

Fig.4. *Effects of solution pH on the removal of Pb^{2+} by biochars*

4. Conclusion

The goal of the article was the analysis and comparison of three biochars (WHX, COX, SOX) to adsorb Pb^{2+} from mine waster-water. Nine types of biochar (WH500/600/700, CO500/600/700, SO500/600/700) were prepared by pyrolyzing crop straw (WH, CO and SO) at different temperatures (500, 600 and 700 °C) under a limited oxygen condition. Adsorption and its influential factors of Pb^{2+} to the biochar in waster-water were investigated. The results showed that: (1) There are obvious distinctions between these pyrolytic temperatures for each test biochar. Adsorption percentages of Pb^{2+} on WHX, COX, and SOX decreased in the order of WH600 (52.9%) > WH500 (51.6%) >WH700(45.4%), CO600 (81.9%) > CO500 (70.6%) >CO700(66.4%),SO600(64.9%)>SO500(64.8%)>SO 700(55.8%). The biochars pyrolyzed at 600 °C showed the highest adsorption rate to Pb^{2+} in waster water, and the adsorption percentage decreased in the order of CO600 (81.9%) > SO600 (64.9%) > WH600 (52.9%). (2) The Adsorption capacity of Pb^{2+} increased with increasing adsorbent. Specifically, its adsorbability increased slowly when the adsorbent was fewer, the removal rates of Pb^{2+} from waster-water increased dramatically when adsorbent was over $3.3g{\cdot}l^{-1}$, and equilibrium could be achieved when adsorbent was more than $10g{\cdot}l^{-1}$; (3) Pb^{2+} adsorption rate increased along with the lapse of time. The biosorption of Pb^{2+} on the studied biochars can be segmented into two phases: the first speedy process is put down to the sorption of metal ions on biochar surface; the next slow process is linked with the change of adsorbent metal ions from outside to inside of the biochar. The adsorption kinetics of Pb^{2+} on WH600 and CO600 was faster than on SO600. Only the pseudo-first-order model presented the best appropriate for all of the tested data and the information displays that correlation coefficients are higher than 0.925 for all biochars. The differences between k_1 values mostly connected to the rate of the adsorbent process, and this higher k_1 value is related with a faster adsorbent process arriving the balanced state. (4) pH had great influence on biosorption of the metals, the adsorption of Pb^{2+} increased with increasing pH. When pH<4.0, the removal rate of three studied biochars are very low and does not surpass 20%; Adsorption percentage of Pb^{2+} increased rapidly when the initial pH increased from 4 to 6; when pH value exceeds 6.0, the removal rate remains largely unchanged and the balanced state is achieved. Under the same conditions, the maximum sorption capacity of Pb^{2+} by WH, CO and SO decreased in the order corn>soybean>wheat. These observations provided useful information in precisely predicting the adsorption behavior of biochar to Pb^{2+} in waster water. The maximal adsorbed mass for Pb^{2+} was observed in mine waster-water. High adsorption capacity makes the crop biochars as useful and economical materials for removal of Pb^{2+} from wastewater.

Acknowledgments

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