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Passivation Effect of Corn Vinasse Biochar on Heavy Metal Lead in Paddy Soil of Pb-Zn Mining Area

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ABSTRACT

The in-lab incubation experiments were conducted to identify the passivation effect of corn vinasse biochar, which was prepared at different temperatures, on heavy metal Pb in paddy soil of the Pb-Zn mining area. The results showed that after 30 days of biochar amended to the soil, the soil pH and organic carbon content increased by 2.72%-8.47% and 27.79%-65.26%, respectively. The CO_3^{2-} and OH^- contained in corn vinasse biochar could react with Pb and generate carbonate and hydroxide of Pb. In comparison with the treatment control, the bioavailable fractions of Pb were reduced by 26.6%, 23.30%, 26.95%, and 35.33%, respectively, in biochar-amended treatments. Exchangeable fractions of Pb decreased by 21.50%, 21.33%, 22.58%, and 22.58% for the treatment 3% (300°C), 6% (300°C), 3% (600°C), and 6% (300°C) corn vinasse biochar, respectively, compared with the treatment control. As a whole, corn vinasse biochar could effectively promote the transformation of Pb in soil from the exchangeable fractions into the Fe-Mn oxide-bound fractions and residue fractions, with a significant passivation effect for Pb in soil and more effective passivation by high-temperature preparation and increased dosage of biochar.

INTRODUCTION

With the rapid development of China's economy, the problem of heavy metal contamination has become increasingly acute, and the pollution of soil heavy metal has become an environmental issue of worldwide concern. The research has shown that excessive accumulation of heavy metals in soil poses a serious threat to human health, food safety, and soil ecosystems (Wang et al. 2020). According to the "National Soil Pollution Survey Bulletin" issued by the Chinese Government in 2014 (Ministry of Environmental Protection 2014), the exceedance rate of total soil sampling sites was 16.1%. The exceedance rate of soil sampling sites in mining areas accounted for 33.4%, in which heavy metals were the dominant pollutants, with the contamination rate of Pb as high as 1.5% (Chinese Ministry of Environmental Protection and Ministry of Land and Resources 2014). Sun et al. (2021) sampled and analyzed the Pb content in Pb-Zn tailings in Danzhai County, Guizhou Province, and the result showed that the Pb content in the 0-20 cm surface soil was15.2 times higher than the background soil value in Guizhou. Lead (Pb^{2+}) is a typical heavy metal that can be absorbed by plants and animals and then enter the human body through the food chain, causing harm to humans due to its poor biodegradability and easy accumulation (Chen et al. 2020, Edenborn et al. 2017). Due to the undesirable discharge of industrial wastewater containing Pb from mining and ore processing, lead battery manufacturing, printing and dyeing, industrial building materials, and the random piling of waste tailings containing Pb, a large amount of Pb has entered the soil, which has caused soil Pb pollution to become a serious environmental problem (Qin et al. 2010). Therefore, environmentally appropriate management of large quantities of smelting waste, especially safe disposal in combination with sustainable remediation technologies, is a challenge of global importance (Mensah et al. 2021, Antoniadis et al. 2022).

Biochar is an efficient and environmentally friendly material for soil remediation (Kumar et al. 2018, Vamvuka et al. 2019), which mainly immobilizes heavy metals in the soil through physical and chemical adsorption, changing the specific chemical form of heavy metals in soil and inhibiting the biological effectiveness of heavy metals (Komkiene & Baltrenaute 2016, Lu et al. 2014). Biochar has become a frontier and hot spot for in-situ remediation of heavy metal pollution in agricultural fields because of its large specific surface area, the abundance of active functional groups on the surface, and excellent complexation and adsorption properties (Ibrahim et al. 2016, Zhen et al. 2013). Currently, biochar is mainly derived from plant straw, animal manure, sludge, and household waste, which has strong adsorption and passivation effects on heavy metal ions, and it has been widely used in soil remediation (Liu & Bai 2022). Ahmad et al. (2016) added soybean straw biochar to the soil, which effectively reduced the acid extraction fraction of Pb and Cu; the higher the pyrolysis temperature, the better the effect of passivation. Park et al. (2011) added chicken manure biochar to soil, which has a strong passivation effect on Pb, Cu, and Cd. Khanmohammadi et al. (2017) showed that the addition of sludge biochar not only improved soil fertility but also had a better immobilization effect on Pb, Mn, and Zn. These research results vary depending on the raw materials of biochar. Vinasse is residues produced after brewing that have pH values in the range of 3.5-5, contain large amounts of Na^+ and K^+ ions, and oxygen-containing functional groups (-COOH, -CO-, -OH), which can provide a large number of adsorption sites for the attachment of heavy metals (Petrovic et al. 2016). At present, few studies are focusing on the remediation of Pb-contaminated soil by biochar derived from corn vinasse. Guizhou is a famous liquor industry destination, and with the flourishing of liquor culture, the amount of liquor vinasse produced after liquor production has increased, and new ways of maximizing its resource utilization need to be explored.

Therefore, in this study, the soil of rice fields in the lead-zinc mining area of Danzhai, Guizhou, was used as the research object. The biochar was prepared as raw material derived from corn vinasse at 300°C and 600°C under anaerobic conditions, respectively. We investigated the effects of the application of corn vinasse biochar prepared at different pyrolysis temperatures on the physicochemical properties of soil and the passivation effect of heavy metals Pb in soil by adopting the improved Tessier five-step sequential extraction method and combined with XRD, FTIR, and SEM-EDX characterization methods, which provided a theoretical basis and technical support for the in-situ remediation of Pb-contaminated soil by corn vinasse biochar and also explored new ways for the development and utilization of corn vinasse.

MATERIALS AND METHODS

The corn vinasse was sourced from Renhuai distillery in Guizhou Province, which was washed with deionized water, dried to constant weight at 60°C, crushed to 60 mesh, and put in a dry place. The Pb-contaminated soil was collected from the paddy field soil (107°32'4" N, 26° 10'56" E) in the Pb-Zn mining area of Danzhai County, Qiandongnan Prefecture, Guizhou Province, with a fine gray-brown loam. The soil was collected by the multi-point method, transported to the laboratory, and prepared for use after natural air-drying and removal of plant roots and stone particles. The physical and chemical properties of the soil are shown in Table 1. The soil is weakly acidic, and the content of heavy metal Pb is 4.2 times the risk screening value (100 $mg.kg^{-1}$, 5.5< PH ≤ 6.5) of the soil pollution risk control standard for agricultural land (GB15618-2018).

Table 1: Soil physical and chemical characteristics.

Total soil Pb content/ [$mg.kg^{-1}$]	pH	SOM/[$g.kg^{-1}$]	CEC/[$cmol.kg^{-1}$]
423.67	5.73	4.54	32.917

Preparation of Biochar

The biochar was produced by an oxygen-limited cracking method with the raw material of corn vinasse (Chinese Baijiu Vinasse (CBV)) under the anaerobic environment at a rate of 5°C-min-1 up to 300 and 600°C and constant temperature for 1 h. After natural cool-down to room temperature, the biochar was removed and reserved, marked with CBV300 and CBV600, respectively, and its physicochemical properties are shown in Table 2.

Soil Cultivation Experiment

A total of 5 treatment groups with 3 replicates were set up by adding 3% and 6% of biochar (CBV300 and CBV600) to 3 kg of the trial soils and setting up a blank control group. The group of soil without biochar addition was labeled as KB. The group of soil biochar additions was labeled as T1, T2, T3, and T4, respectively. Its specific design is shown in Table 3. All pots should be placed in a cool, dry place for ventilation and cultivation and weighed and watered every three days to maintain a soil moisture content of about 50%. After 30 days of incubation, the soil was taken out and placed in a cool and ventilated place for air-drying, grinding, passing 200 mesh, and then the physical and chemical properties, Pb content, and morphology were determined.

Measurement Method

The pH electrode determined the PH of soil and biochar,

Table 2: Physicochemical properties of biochar.

Biochar	PH	BET/m^2. g^{-1}	Total hole volume/$mL.g^{-1}$	C/N element ratio %	Content of alkaline groups/$mmol.g^{-1}$
CBV300	8.61±0.0062	2.3263	0.0234	10.14	0.97±0.16
CBV600	10.46±0.0036	9.0693	0.1267	12.03	1.50±0.02

Table 3: Experimental design treatment groups.

KB	Soil
T 1	Soil +3%CBV300
T 2	Soil +6%CBV300
T 3	Soil +3%CBV600
T 4	Soil +6%CBV600

and the organic matter content (SOM) was determined by the potassium dichromate volumetric method. The cation exchange capacity (CEC) was determined by the hexamine cobalt chloride solution-spectrophotometric method (Liu et al. 2020). Analysis of heavy metal fractions according to the modified Tessier five-step graded extraction method (Essier et al. 1979). The heavy metals in the soil were classified into ion exchange fraction (F1), carbonate bound fraction (F2), iron-manganese oxide bound fraction (F3), an organic bound fraction (F4), and residue fraction (F5). The elemental concentrations in the extracts were determined by flame atomic absorption spectrophotometry. The concentrations of the elements in the extracts were determined by flame atomic absorption spectrophotometry.

The surface pore structure was analyzed by vacuum scanning electron microscopy (SEM-EDX), the elemental analyzer determined the C, H, O, and N content of biochar, the acid-base functional groups on the surface of biochar was determined by Bohm titration (Karamanova et al. 2019), the surface functional groups were determined by Fourier infrared spectroscopy (FTIR), and the crystalline substances contained in the biochar before and after action were determined by X-ray diffraction (XRD).

Data Processing and Analysis

Analytical processing was performed using Microsoft Excel 2019 and SPSS 25, with one-way ANOVA analysis of significant differences using LSD, Waller-Duncan and Tukey' s-b tests, and Origin 2021 for plotting.

RESULTS AND DISCUSSION

Effect of Biochar on Soil Physicochemical Properties

Biochar reduces the risk of soil heavy metal contamination by changing the physicochemical properties of soil and then changing the distribution form of heavy metals in the soil (Tang et al. 2022). SOM, pH, and CEC are key factors in the morphological transformation and migration of metals (Li et al. 2022). The value of pH is closely related to the morphology and bioavailability of heavy metals in soil and the process of sorption and desorption of contaminants in soil (Shi et al. 2016). After 30 days of incubation, there was a significant difference in soil pH and SOM but no significant difference in CEC in the biochar remediation treatment as compared to the KB treatment. Compared with KB treatment, all T1-T4 treatments significantly increased the pH value of the soil (Table 4). The application of CBV300 biochar increased the pH value of the soil by 2.66% and 5.54%, respectively. The application of CBV600 was significantly more effective. The value of pH increased by 6.87% and 8.52%, respectively, which was related to the degree of alkalinity of biochar, and biochar CBV600 contained more alkaline functional group content than CBV300 (Table 2). At the same time, CBV600 contains a large number of alkaline substances such as carbonates, strong oxides, and metal oxides that can undergo rapid hydrolysis or adsorption in contact with soil to reduce the exchange level of exchangeable ions such as H^+ and Al^{3+} in the soil, which makes it more effective in raising the PH value of the soil (Houben et al. 2013, Yuan et al. 2011, Novak et al. 2009).

The organic matter can immobilize heavy metals in the soil by adsorption or chelation and, at the same time, has a reduction effect, which reduces the availability of heavy metals in the soil (Li et al. 2017). The organic matter content of biochar-treated soil was significantly enhanced by 27.85-65.18 % compared to KB (Table 4). Due to the rich content of C elements in biochar, the addition of biochar to soil can enhance the soil C/N ratio and increase the organic matter content of the soil (Chan et al. 2007). The application of biochar can effectively reduce the availability of Pb in the soil.

Passivation of Pb on Biochar

The XRD spectra showed that the mineral composition changed before and after the biochar action (Fig. 1), with new $3PbCO_3.2Pb(OH)_2.H_2O$ peaks found in the range of 2θ=20.85° to 30.27° for CBV300 and $Pb_3(CO_3)_2(OH)_2$ in the range of 2θ=24.64° to 34.15° for CBV600, indicating that the reactions of CO_3^{2-} and OH^- in biochar with Pb, where more Pb complexes were observed in CBV600 than in CBV300 biochar. Cao Harris (2010) explored and found

Table 4: Soil pH and SOM content at different treatments group.

Treatments group	pH	SOM/[g.kg $^{-1}$]	CEC/[cmol.kg^{-1}]
KB	6.26 ±0.0le	6.63±0.04c	28.24±0.41a
T1	6.43 ±0.02d	8.47±0.38a	29.36+0.23a
T2	6.61 ±0.00 c	10.94±0.82b	28.14+2.75a
T3	6.69 ±0.02b	9.20±0.17b	30.38+0.65a
T4	6.79 ±0.03a	9.12±0.42b	30.62+1.40a

Note: For the same column, the same lowercase letter indicates no significant difference under the same cycle and different treatments, and different lowercase letters indicate a significant difference, $p < 0.05$, as below.

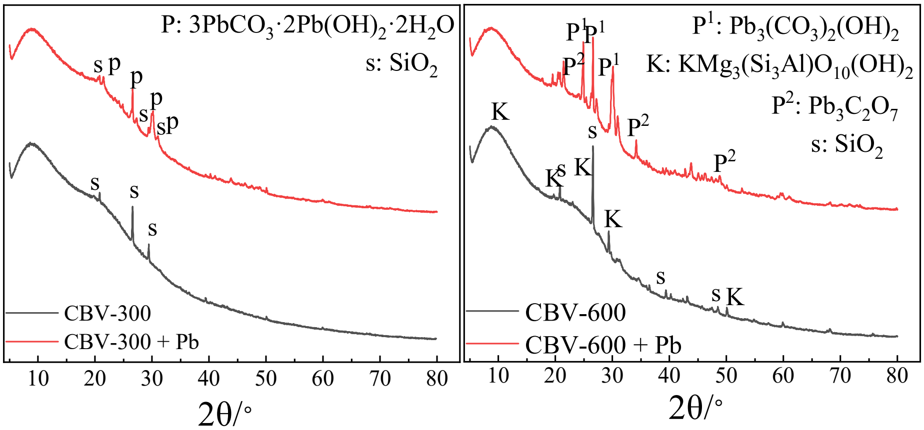

Fig. 1: XRD spectra before and after the action of biochar.

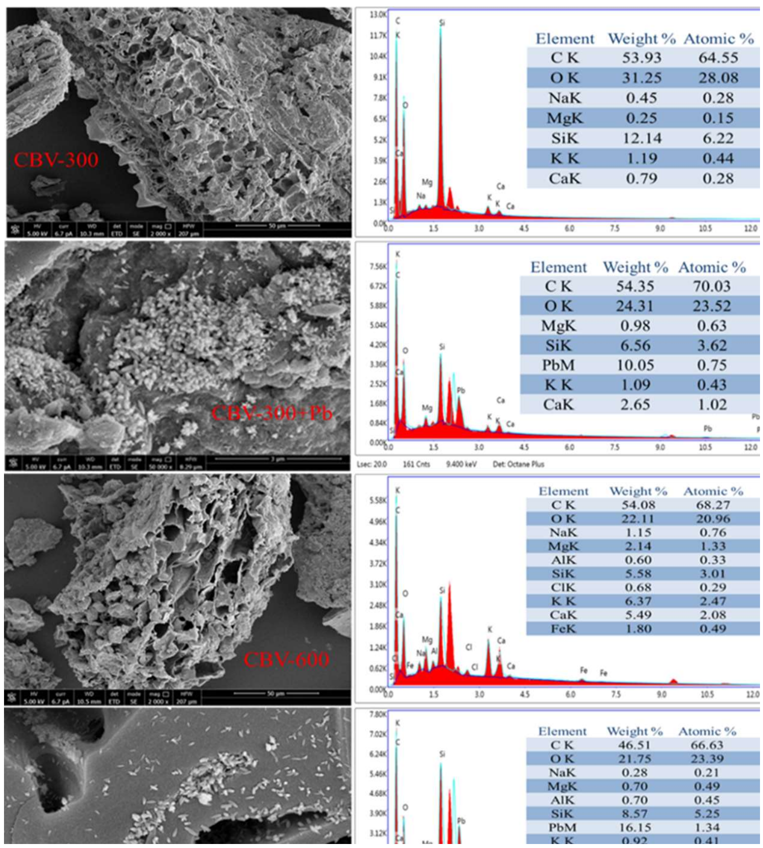

Fig. 2: Scanning electron microscopy spectra (SEM-EDX) of biochar before and after interaction with heavy metals.

that the action of dairy-manure biochar on Pb reacts with Pb to result in precipitates of carbonate and hydroxide. As shown in the SEM electron micrographs (Fig. 2), the size and shape of the particles were different before and after the reaction of biochar, and the surface structure was irregular and contained pore structure, the surface after the action all contained crystalline structure, the results are consistent with the XRD analysis. Combined with the elemental analysis of EDX spectra, it was found that the content and proportion of elements such as Ca, Mg, Na, and K changed, which indicated that ion exchange occurred between Pb and biochar. In addition, the application of biochar increased the soil pH. Pb could combine with OH^- and CO_3^{2-} to form insoluble carbonates and hydroxides, which resulted in reducing the concentration of bioavailable Pb in the soil. In summary, it can be concluded that the vinasse biochar can effectively consolidate the Pb, and its high-temperature pyrolysis biochar (CBV600) is more effective.

Effect of Biochar on the Morphological Transformation of Pb in Soil and Remediation Effect

Researchers generally evaluate the passivation effect of heavy metals in soils by assessing the mobility and bioavailability of heavy metals in terms of the magnitude of their bioavailable fractions (Song et al. 2015). The application of biochar significantly reduced the bioavailable fraction of Pb in the soil compared to KB (Fig. 3). On the 30th day of incubation, the bioavailable fraction of Pb in the soil was significantly reduced in all treatments. T1-T4 treatment groups decreased by 26.61%, 23.30%, 26.80%, and 35.29%, respectively. With the increase in temperature, the content of the bioavailable state of Pb showed a decreased trend; there was no significant difference in the effect of biochar addition at 300°C. However, at the high temperature of 600°C, the treatment group with 6% biochar content applied had a more significant effect than the 3% treatment group, indicating that 6% CBV600 had a better passivation effect. As high-temperature pyrolysis can increase the pH and Eh values of the biochar itself, which changes the surface activity of the crystalline material in the soil and induces a change in the fugitive form of heavy metal ions in the soil to achieve a reduction in the available fraction of Pb (Zhang et al. 2013). It was evident that the biochar prepared by applying different pyrolysis temperatures reduced the content of Pb in the soil to a certain level, and the high-temperature effect was better.

Compared to KB, the existence form of Pb in the soil was changed after the addition of biochar, with the T1-T4 treatment group mainly existing in the Fe-Mn oxide bound fraction (63%, 64%, 66%, 75%) (Fig. 4). The degree of passivation effect of biochar prepared at different pyrolysis temperatures on Pb varied. After 30 days of incubation, the percentages of F1 in the T1-T4 treatment groups decreased by 21.50%, 21.33%, 22.58%, and 22.58%, respectively. The percentages of the sum of F3 and F4 increased by 21.85%, 23.13%, 24.81%, and 34.80%, respectively, indicating that biochar could promote an exchangeable state of Pb in the soil to the Fe-Mn oxide-binding state and the residue state, the result indicated that biochar played a passivating effect on Pb. The effect of high-temperature pyrolysis biochar was more effective. As high-temperature pyrolysis of biochar contains more inorganic mineral elements and alkaline groups, which can exchange or precipitate with Pb^{2+}, the surface functional groups (-OH, -COOH) and rich pore structure of biochar has a strong affinity for Pb (Wagner & Kaupenjohann 2014). The reactions of heavy metals in soil with functional groups on the surface of biochar are as follows.

$$\text{BC-OH+Pb}^{2+}\rightarrow\text{BC-OPb}^{2+}\text{+H}^{+} \quad \ldots (1)$$

$$\text{(BC-OH)}_2\text{+ Pb}^{2+}\rightarrow\text{(BC-O)}_2\text{Pb}^{+}\text{+H}^{+} \quad \ldots (2)$$

$$\text{BC-COOH+Pb}^{2+}\rightarrow\text{BC-COOPb}^{2+}\text{+H}^{+} \quad \ldots (3)$$

$$\text{(BC-COOH)}_2\text{+Pb}^{2+}\rightarrow\text{(BC-COO)}_2\text{Pb}^{+}\text{+H}^{+} \quad \ldots (4)$$

The reaction can form a complex of heavy metals on the surface of biochar through intermolecular hydrogen bonds, which have a solidification effect on heavy metals (Gong et al. 2022). According to the infrared spectrum (Fig. 5), -OH vibrated at 3333 cm^{-1}, C=C and C=O in the aromatic ring vibrated at 1627 cm^{-1} and C=O in the carboxyl group vibrated at 1704 cm^{-1}, and aromatic-CH-peak occurred at 860 cm-1 in CBV600, which proved that functional groups such as -OH, -COOH and electron-containing groups in the vinasse biochar could exchange ions, complexation reactions and electron conjugation reactions with Pb, promote the adsorption and

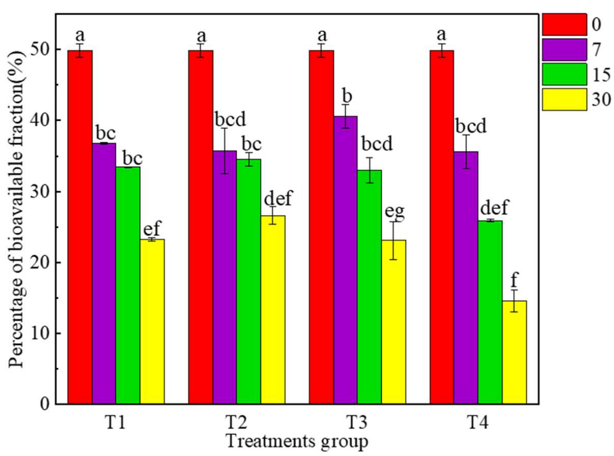

Fig. 3: Effect of biochar on the bioavailable fraction of Pb.

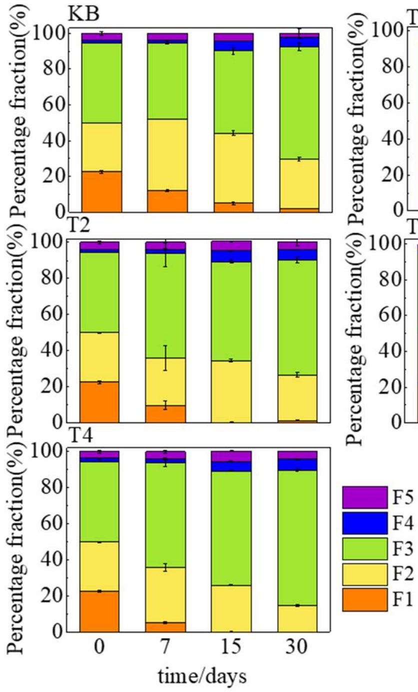

Fig. 4: Effect of biochar on the distribution form of Pb in soil.

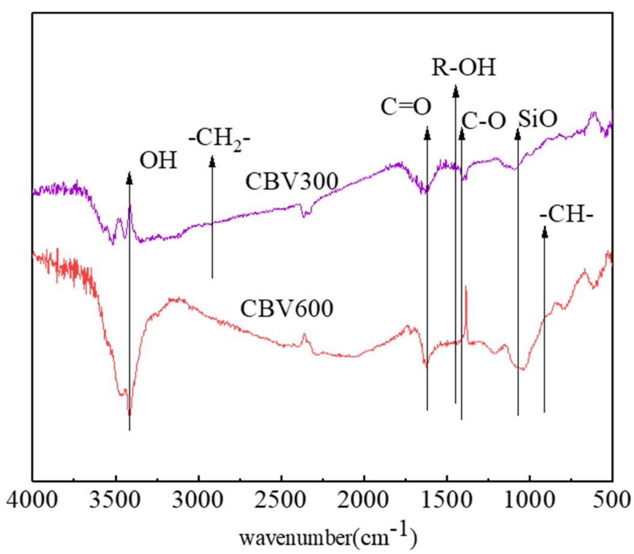

Fig. 5: Infrared spectrum of biochar (FTIR).

precipitation of Pb in soil. In conclusion, corn vinasse biochar has a significant effect on the chemical form of Pb in mine soils. It can promote the transformation of the exchangeable fraction of Pb to the Fe-Mn-oxide bound fraction and residue fraction, significantly reducing the bioavailability of Pb. This process mainly remediates and stabilizes Pb in soils through the occurrence of ion exchange and complexation reactions, and its high-temperature pyrolysis biochar has a better remediation effect.

CONCLUSION

In this study, the application of biochar derived from corn vinasse to Pb-contaminated rice paddy field soil in Pb-Zn mining area significantly improved the value of pH (2.66%-8.52%) and SOM content (27.85%-65.18%) in Pb-Zn mining area rice paddy field soil. In this study, Pb morphology in soil was analyzed by a modified Tessier five-step extraction method. The results showed that after 30 days of biochar application incubation, the bioavailable state of Pb in the 3% CBV300, 6% CBV300, 3% CBV600, and 6% CBV30 treatment groups decreased by 26.61%, 23.30%, 26.95%, and 35.33%. The soil, the exchangeable state of Pb, was transformed to Fe-Mn oxide-binding and residue states, with the better effect of 6% CBV600. This study combined XRD, SEM-EDX, and FTIR characterization methods and found that functional groups such as -OH, -COOH, and -C=O and electron-containing surface functional groups in the biochar were involved in ion exchange, complexation, and π-electron conjugation reactions with the Pb and the increase of basic groups at high temperatures, which promoted the precipitation of lead ions. In conclusion, the application of corn vinasse biochar could change the exchangeable state of Pb in the soil to the residue state and Fe-Mn oxide-bound state and promote the passivation of Pb in soil, which could reduce the migration and transformation rate of heavy metals in soil. At the same time, high-temperature treatment and increasing the amount of biochar had more significant effects on the passivation of Pb in the soil of the paddy field.

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