RESEARCH ARTICLE

Preparation of sludge biochar and its adsorption performance under different modification conditions

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Sludge biochar as an efficient adsorbent material is widely used in the field of pollution removal. However, the adsorption performance of traditional sludge biochar is limited and affected by many factors. To solve the insufficient adsorption performance of traditional sludge biochar, this research modified sludge biochar with $MgCl_2$, $KMnO_2$, KOH, and other materials and explored the adsorption performance of sludge biochar by setting different acid and alkali environments, heavy metal concentrations, and biochar contents. The results showed that the higher the biochar content, the higher the removal rate of Cd, Cu, and Pb. When the biochar content was 7 g/L, the maximum removal rate was 95.4%. Sludge biochar demonstrated the best adsorption effect on three heavy metal ions when the pH was within 6 to 6.5. In addition, the higher the concentration of heavy metals in sewage, the better the adsorption effect of sludge biochar. The modified sludge biochar had more voids and significantly improved adsorption capacity. The modified KOH biochar (KC) sludge biochar had the best overall performance. In the single-sample Cd^{2+} adsorption performance test at five different concentrations, when the Cd^{2+} concentration was 50 mg/L, the adsorption capacity of KC reached 47.3 mg/L, which was higher than the 34.6 mg/L of the original sludge biochar. Modified sludge biochar could effectively adsorb multiple types of heavy metal ions. This research provided new ideas for sludge resource utilization and technical support for heavy metal pollution control and promoted the sustainable development of pollution control.

Keywords: sludge biochar; heavy metal ions; adsorption performance; heavy metal adsorption; cadmium ion removal; biochar functionalization.

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Introduction

With the accelerated development of industry and urbanization, water pollution and other problems have become increasingly serious, among which heavy metal ions, organic pollutants, *etc*. have become important topics of social concern [1]. Sludge is a kind of waste rich in organic matter and minerals. If it can be used rationally, it can achieve resource utilization and environmental management [2]. Sludge biochar can form solid materials containing carbon through high-temperature pyrolysis. Its rich functional groups and specific area make it of great application value in the field of pollution removal [3]. The decontamination of sludge biochar is considered from two aspects including physical adsorption and chemical adsorption. Physical adsorption mainly adsorbs heavy metal ions through the pore structure and specific surface area of biochar. The larger the specific area and richer pore structure, the stronger the

physical adsorption capacity [4, 5]. Chemical adsorption involves the chemical bonding between functional groups and heavy metal ions. Functional groups like carboxyl groups and phenolic hydroxyl groups can combine with metal ions in heavy metals through ion exchange or coordination to achieve chemical adsorption [6, 7].

The preparation of sludge biochar is based on pyrolysis. Controlling pyrolysis time, temperature, and rate can significantly optimize the adsorption capacity and improve the decontamination performance [8]. In addition, the adsorption performance of sludge biochar can be improved through modification treatment. Common modified materials include $MgCl_2$, KOH, $FeCl_3$, H_3PO_4, *etc*., which can be introduced into sludge biochar by means of impregnation, pyrolysis, *etc*. to effectively enhance the positive charge on the biochar surface, thereby optimizing the adsorption performance for heavy metal ions [9, 10]. Functional groups such as carboxyl and phenolic hydroxyl groups can effectively combine heavy metal ions such as Cu^{2+}, Zn^{2+}, Pb^{2+}, Cd^{2+} through ion exchange or coordination, significantly improving the adsorption capacity [11, 12]. The adsorption capacity of sludge biochar is affected by the concentration of heavy metal ions, the pH environment, and the dosage of sludge biochar [13]. According to relevant research, there are obvious differences in the adsorption of heavy metal ions by sludge biochar under different pH environments. When the pH is within 6 to 7, it helps to the adsorption effect of heavy metal ions such as Cu^{2+} and Pb^{2+} [14]. Sludge biochar as a new type of adsorption material has good application effects in sewage purification, heavy metal treatment, and other fields. However, traditional unmodified sludge biochar has limited effective adsorption capacity in practical application due to the influence of pollution source type, concentration, and pH environment, which is the key problem to be solved urgently in the field at present.

This study prepared modified sludge biochar to optimize its adsorption capacity and decontamination performance and systematically investigated the adsorption performance of modified sludge biochar for heavy metal ions under different modification conditions. KOH was employed as the typical modifier to prepare modified sludge biochar, and the adsorption performance of biochar samples was tested under different environmental conditions. The characteristics of modified sludge biochar were analyzed to reveal its adsorption mechanism for heavy metal ions. The research improved the adsorption performance of sludge biochar for heavy metal ions by using different modified materials to modify sludge biochar and optimizing the modification conditions, which provided new ideas for sludge resource utilization. Further, the research systematically analyzed the relationship between the characterization characteristics and adsorption performance, revealing the adsorption mechanism of heavy metal ions by modified sludge biochar. The results of this research provided technical support for sludge resource utilization and heavy metal pollution control in environmental engineering and had important theoretical reference and practical application value for developing environmental protection and material science communities.

Materials and methods

Sludge sample

The sludge sample used in this study was collected from the Wuhan Hankou Sewage Treatment Plant (Wuhan, Hubei, China) on October 15, 2024 with a total collected amount of 50 kg. The sample was sterile sludge after anaerobic fermentation and mechanical dehydration.

Sludge biochar preparation

The sludge sample was dried in DHG-9070A electric constant temperature blast drying oven (Thermo Fisher Scientific, Waltham, MA, USA) at 98°C for 2.4 h. After grounding to an 18-mesh

sieve, the sample was placed in SX2-8-10N Muffle furnace (Shanghai Yiheng Scientific Instruments, Shanghai, China) for pyrolysis at 600°C for 3.8 h. The sample was then naturally cooling down and weighed using FA2004 electronic balance (Mettler Toledo, Zurich, Switzerland) and stored in a sealed bag. The prepared sludge biochar had an ash content of 72.8% and a pH value of 5.65.

Preparation of modified sludge biochar

$MgCl_2$ modified biochar (MgC) was prepared by mixing 15 g of sludge biochar sample with 0.5 mol/L $MgCl_2$ solution before transferring to a sealed glass bottle for impregnation for 12 h followed by drying at 106°C for 22 h. The dried sample was pyrolyzed in a tube furnace with N_2 gas and temperature rise rate of 20°C/min at 600°C for 1.8 h. The pyrolyzed samples were then rinsed with deionized water, dried at 106°C for 22 h, grounded into powder, and stored for later use. $KMnO_4$ modified biochar (MnC) was done by mixing 15 g of sludge biochar sample with 0.1 M $KMnO_4$ solution at a volume ratio of $KMnO_4$ solution to sludge sample of 5:1. The mixture was then placed in a sealed glass bottle and mixed thoroughly for 12 h before drying at 106°C for 22 h followed by pyrolyzing in a tube furnace with N_2 gas at temperature rise rate of 10°C/min at 600°C for 2 h. After natural cooling down, the sample was washed repeatedly with $NaHCO_3$ solution and distilled water before finally dried at 106°C for 22 h. KOH modified biochar (KC) was performed by mixing KOH solid and sludge biochar and grounding at a mass ratio of 18 g:180 g. The mixture was put into a tube furnace with N_2 flow rate of 40 mL/min and temperature rise rate of 20°C/min at 600°C for pyrolysis for 2 h. After cooling down to room temperature, the sample was weighed and subjected to multiple suction filtrations with deionized water under a Buchner funnel until the pH reached 7. The product was dried at 106°C for 22 h.

Preparation of heavy metal pollution solution

Pb standard solution was prepared by dissolving 1.5980 g of solid $Pb(NO_3)_2$ into an appropriate amount of deionized water. After adding 1 mL of concentrated nitric acid to inhibit hydrolysis, the solution was diluted to a fixed volume to obtain 1 g/L Pb^{2+} standard stock solution. Cu standard solution was obtained by dissolving 1.2675 g of solid $CuCl_2{\cdot}2H_2O$ with deionized water. After adding 1 mL of concentrated nitric acid for stabilization, the solution was diluted to a fixed volume to prepare a 1 g/L Cu^{2+} standard stock solution. Cd standard solution was prepared by dissolving 2.7718 g of solid $CdCl_2{\cdot}H_2O$ in deionized water followed by adding 1 mL of concentrated nitric acid and diluting to a fixed volume to obtain a 1 g/L Cd^{2+} standard stock solution. Zn standard solution was obtained by dissolving 2.0162 g of solid $ZnCl_2{\cdot}6H_2O$ with deionized water containing 1 mL of concentrated nitric acid and diluting to a fixed volume to prepare a 1 g/L Zn^{2+} standard stock solution.

Heavy metal mixture configuration

Heavy metal mixed solutions were prepared by mixing the single metal standard stock solutions with deionized water, in which the concentration of the target heavy metal was set as a gradient of 10, 20, 30, 40, and 50 mg/L, while the concentrations of the other three heavy metals were fixed at 5 mg/L. Briefly, 0.5 mL of each Cu^{2+}, Cd^{2+}, Zn^{2+} stock solutions were mixed with 1.0, 2.0, 3.0, 4.0, and 5.0 mL of Pb^{2+} stock solution and diluted with deionized water in a 100 mL volumetric flask to obtain mixed samples 1, 2, 3, 4, and 5, respectively. In addition, gradient mixed solutions with Cu^{2+}, Cd^{2+}, and Zn^{2+} as the target heavy metals were prepared, respectively, following the same strategy.

Sample characterization analysis

Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), and X-ray diffraction (XRD) were applied to characterize the raw sludge biochar and modified sludge biochars (MgC, MnC, KC) with all characterizations conducted in triplicates to identify surface functional groups, observe microscopic morphology and pore structure, and analyze crystal phase composition of biochar samples, respectively. FTIR analysis was conducted by mixing 1 mg dried biochar samples with KBr powder at a mass ratio of 1:100 followed by

grounding uniformly and pressing into transparent pellets. KBr powder served as the infrared transparent carrier and tableting medium to stably transmit infrared light. The pellets were tested by using Thermo S Nicolet iS50 FTIR (Thermo Fisher Scientific; Waltham, MA, USA) with a scanning range of 400 - 4,000/cm, a resolution of 4/cm, and 32 scans per sample. The functional groups of the samples were identified according to the absorption peak positions. SEM analysis was performed by drying the biochar samples to constant weight at 60°C before being fixed on a metal sample stage with conductive adhesive and sputter-coated with gold for 60 s. The surface morphology and pore structure of the samples were observed by using MIRA LMS SEM (TESCAN, Brno, Czech Republic) at an accelerating voltage of 15 kV and a fixed magnification of 2,000×. The pore distribution of raw and modified biochar was compared and analyzed. XRD analysis was done by grounding the dried biochar samples and passing through a 200-mesh sieve and spreading evenly on a glass sample holder. Rigaku Miniflex 600 XRD (Nippon Science Corporation, Tokyo, Japan) was employed for detection with a Cu Kα radiation source (λ = 1.5406 Å), a scanning angle (2θ) range of 10 - 80°, a scanning speed of 5°/min, a tube voltage of 40 kV, and a tube current of 15 mA. The crystal phase composition and structure of the samples were identified by matching the diffraction peaks with the standard XRD card database.

Adsorption performance testing

Exact amounts of modified biochar (MgC, MnC, KC) and unmodified sludge biochar with gradient dosages of 1, 2, 3, 5 and 7 g/L were accurately weighed and mixed with 10 mL of heavy metal single standard solution and mixed solution in conical flasks, respectively. The mixtures were placed in a ET-Q Gas bath constant temperature oscillator (Eppendorf, Hamburg, Germany) at 25°C, 180 rpm, for 24 h with the biochar content, solution pH value, and heavy metal concentration as independent variables. After the reaction, the mixtures were centrifuged at 4,000 rpm for 10 min. The residual heavy metal concentration in the supernatant was determined by using NeXION 1000 Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (PerkinElmer, Waltham, MA, USA). The influence of each variable on the adsorption performance was statistically analyzed. The adsorption capacity (q_e) (mg/g) and heavy metal removal rate (R) (%) were calculated as follows.

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad (1)$$

where C_0 was the initial concentration of heavy metal ions in the solution (mg/L). C_e was the equilibrium concentration of heavy metal ions in the supernatant (mg/L). V was the volume of the heavy metal solution (L). m was the mass of the added sludge biochar (g). The heavy metal removal rate was calculated as below.

$$R = \frac{C_0 - C_e}{C_0} \times 100\% \quad (2)$$

Heavy metal speciation analysis

European three-stage four-step sequential extraction method was employed to analyze the speciation of weak acid extractable, reducible, oxidizable, and residual of Cd, Cu, Pb, and Zn in raw sludge biochar for evaluating heavy metal morphological stability. All experiments were performed in triplicates. After each extraction step, the supernatant was separated by centrifugation at 4,000 rpm for 10 min. The heavy metal concentrations in extracts were determined by using ICP-MS. The morphological stability coefficient was calculated as the ratio of (oxidizable + residual) fractions to total heavy metal content. Weak acid extractable was conducted by mixing 1 g biochar with 40 mL of 0.11 mol/L CH_3COOH at 25°C, 180 rpm, for 16 h. Reducible was performed by mixing the residue with 40 mL of 0.5 mol/L $NH_2OH{\cdot}HCl$ (pH 2) at 25°C, 180 rpm, for 16 h. Oxidizable was done by adding 10 mL 30% H_2O_2 (pH 2-3) to the residue followed by heating at 85°C for 1 h with intermittent shaking. Then, another 10 mL H_2O_2 was added for 1 h heating. After cooling down, 50

mL of 1 mol/L CH_3COONH_4 (pH 2) was added and shaken as above. The residue was digested with HNO_3-HF mixed acid on HT-300 heating plate (Bio Rad, Hercules, CA, USA), and the digested solution was diluted for detection.

Statistical analysis

SPSS 28 (IBM, Armonk, NY, USA) was employed for statistical analysis of this study. All experimental data were expressed as mean ± standard deviation (SD) of three replicates. Independent samples t-test was used to compare the statistical significance of experimental data between two groups, while one-way analysis of variance (ANOVA) was used for multi-group comparisons. *P* value less than 0.05 was defined as statistically significant, while *P* value less than 0.01 indicated extremely significant differences. Crystal structure analysis of the samples was conducted by using HighScore Plus (https://www.malvernpanalytical.com/tw/products/category/software/x-ray-diffraction-software/highscore-with-plus-option). Image data plotting was completed by using SigmaPlot (https://systat-sigmaplot.com).

Results

Analysis of characteristics of sludge biochar and modified sludge biochar

The sludge contained heavy metals with the average concentrations of each metal as 482 mg/kg Cu, 12.2 mg/kg Cd, 617.2 mg/kg Zn, 150.1 mg/kg Pb, 140.1 mg/kg Ni, and 120.8 mg/kg Cr. Furthermore, the pH of the sludge was 4.974. According to the national standard GB18918-2002, when the pH was less than 6.5, the limits of Cd, Cr, Cu, Pb, Ni, and Zn were 5, 5, 800, 300, 100, and 2,000 mg/kg, respectively. When the pH was ≥ 6.5, the limits were 20, 20, 1,500, 1,000, 200, and 3,000 mg/kg, respectively. The results of FTIR analysis on the prepared sludge biochar showed that there was obvious vibration in the low-frequency band of 1,500/cm, which made it difficult for the sample to reflect characteristics, but it could effectively identify the material structure such as the SiO_2. In the wavelength greater than 1,500/cm, characteristic peaks were used to assist in characterizing functional groups. A characteristic peak appeared at 1,572/cm due to vibrational lifting in aromatic hydrocarbons, and 3,512/cm had higher oxygen-containing functional groups like -OH (Figure 1a). The characteristics indicated that the sludge biochar had basic adsorption characteristics. The XRD analysis results of sludge biochar showed four relatively strong diffraction peaks appeared in the sample mainly due to SiO_2. The results suggested that the prepared sludge biochar had incomplete pyrolysis and SiO_2 appeared. In addition, diffraction peaks containing $PbAs_2O_6$ were also found at 31.50° and 62.48°, which indicated that the sample also contained a small amount of Pb material (Figure 1b). The FTIR analysis results of modified sludge biochar demonstrated that the four sludge biochars all had characteristic peaks in the wavelength range from 810/cm to 1,034/cm. Further analysis found that the modified sludge biochar had stronger fluctuations than the original sludge biochar, which was mainly related to the stretching vibration of Si-O-Si. The wave peak in the 1,652/cm section was mainly caused by the expansion and contraction of the C=O structure. In addition, there were also obvious characteristic peaks at 3,415/cm, which was related to the stretching change of -OH, and the characteristic peaks of modified sludge biochar were stronger than those of the original sludge biochar (Figure 1c). The XRD analysis results of modified sludge biochar showed that the four sludge biochars had obvious diffraction peaks at 26.1°, which could be identified as SiO_2 by reference to FTIR. In addition, diffraction peaks also appeared at 21.1° and 50.3°, all of which were SiO_2 (Figure 1d). In addition, index cards were used to analyze carbon elements. The stabilities of the three modified sludge biochars were improved, and no modifying reagent was found in the SEM analysis. SEM analysis of four types of sludge biochar demonstrated that the three modified sludge biochars had significantly more voids than the original sludge biochar. Many voids were attached to the surface of modified KC, while the structural voids of MgC

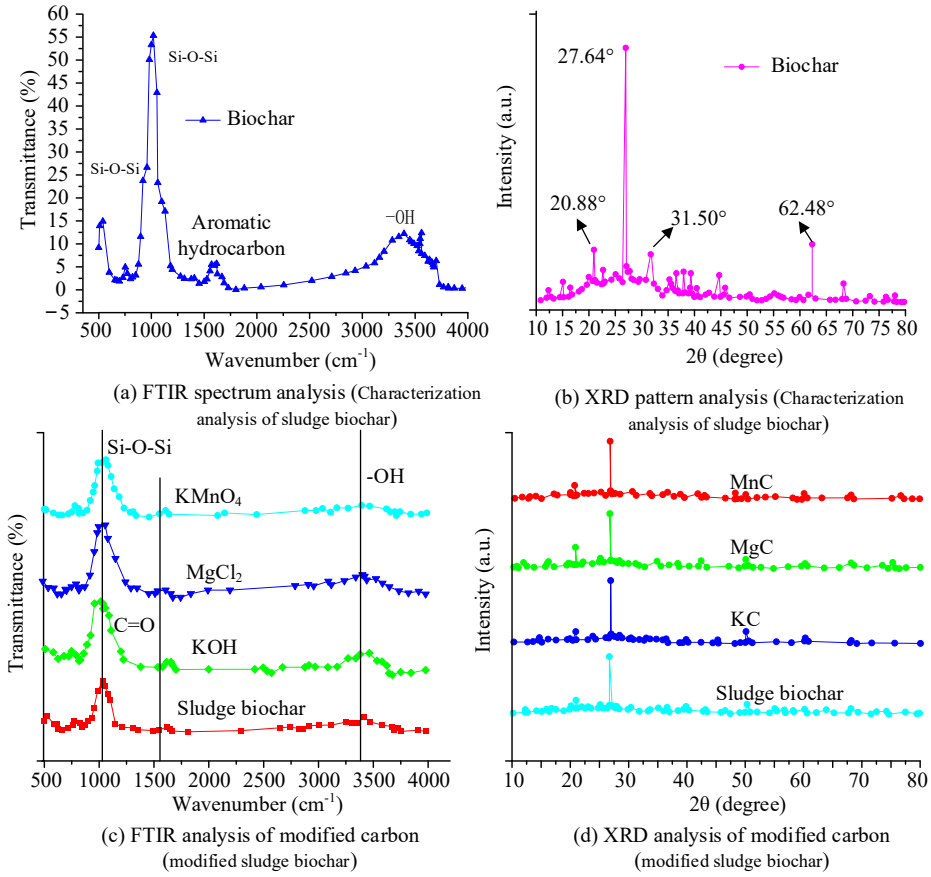

Figure 1. Characterization analysis of sludge biochar.

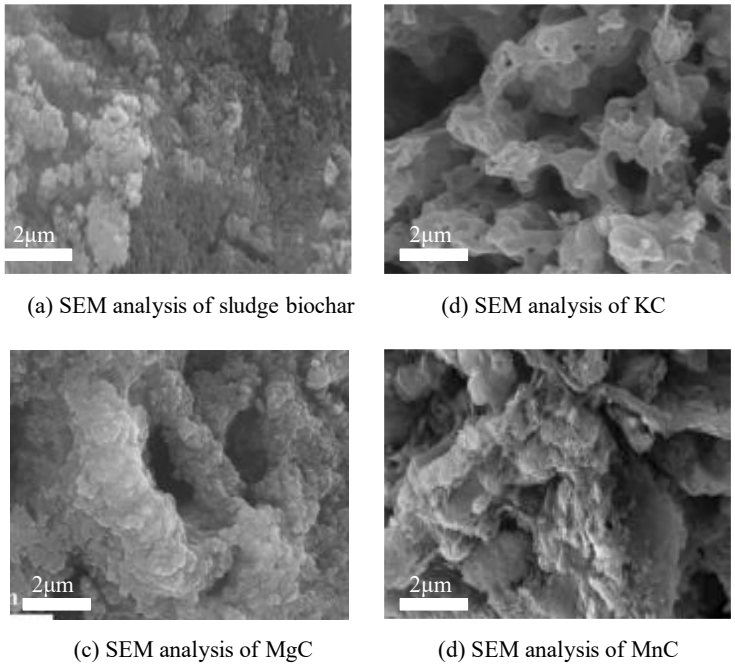

Figure 2. SEM analysis of four types of sludge biochar.

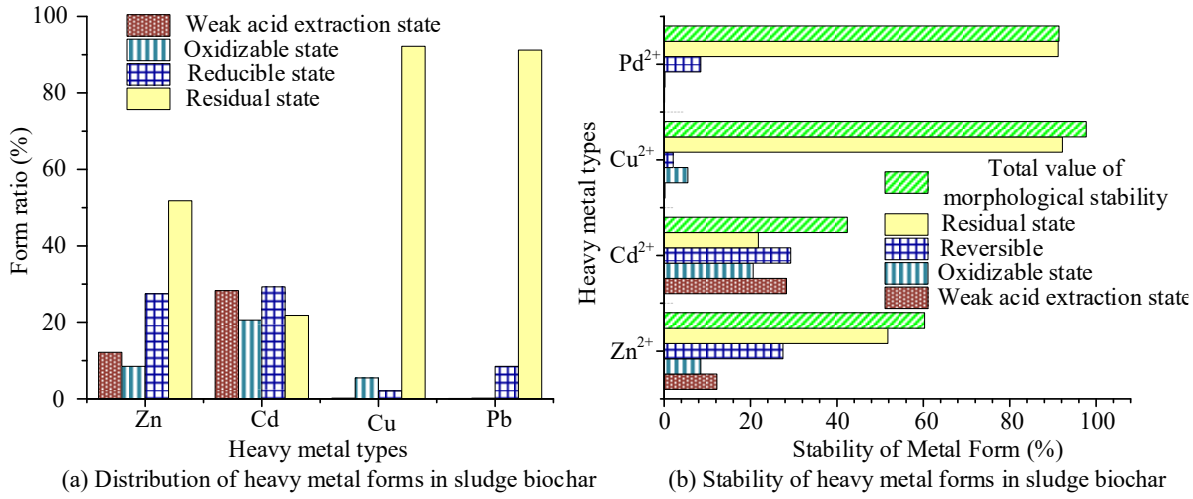

Figure 3. Distribution of heavy metals and stability analysis of metal forms in sludge biochar.

and MnC were significantly increased (Figure 2). The increase in the voids of the modified biochar could be more conducive to improving its own adsorption capacity.

Stability analysis of sludge biochar and modified sludge biochar

The results showed that sludge biochar metals were divided into four forms through technical extraction, and there were obvious differences in the proportions of different heavy metal forms. Zn, Cu, and Pd were mainly in the residual state with Zn accounting for 52.1%, while Cu and Pd both exceed 90%. Cd was mainly in the reducible state and the weak acid extractable state, accounting for 31.1% and 30.1% respectively, while the reducible state in Zn also accounted for 28.8% (Figure 3a). The stable form was expressed as the ratio of oxidizable state + residual state to the total form of heavy metals. The morphological stability of Pb^{+} and Cu^{2+} exceeded 90%, indicating that they had low ion mobility and high stability. The morphological stability of Cd^{2+} and Zn^{2+} were 41.8% and 60.1%, respectively, and Zn^{2+} had a certain stability (Figure 3b). However, the stability of Cd^{2+} was average. Since the Cd^{2+} content in sludge biochar was low, relevant studies reported that the Cd^{2+} leaching rate in Cd heavy metal adsorption experiments was extremely low, and it could also be considered to have stability and safety. Sludges contained heavy metals, and their speciation in biochar determined secondary pollution risk. Modification biochar with $MgCl_2$, $KMnO_4$, and KOH could optimize heavy metal occurrence forms and improve stabilization. The stability differences between raw and modified sludge biochars for environmental safety evaluation were compared and demonstrated that, in raw sludge biochar, Cu and Pb were mainly in the residual fraction with low acid-extractable proportions, while Cd and Zn had high unstable fractions and low stability coefficients of 41.8% and 60.1%, respectively. After modification, the acid-extractable and reducible fractions of heavy metals decreased significantly, while the oxidizable and residual fractions increased, and the stability coefficients were greatly improved. The stability coefficients of Cu and Pb in all modified biochars exceeded 96%, and the stabilities coefficients of Cd and Zn were also increased to more than 67%. The stabilization effects were ranked as KC > MnC > MgC > raw sludge biochar. KC modification showed the best performance with the stability coefficients of Cd and Zn reaching 76.1% and 81.4%, respectively (Table 1). Modified biochars had higher heavy metal stability and lower leaching risk, which met the safety requirements of sludge resource utilization.

Table 1. Comparison of heavy metal speciation distribution and stability coefficients between sludge biochar and modified sludge biochars.

Sample type	Heavy metal	Acid-extractable (%)	Reducible (%)	Oxidizable (%)	Residual (%)	Speciation stability coefficient (%)
Raw sludge biochar	Cd	30.1	31.1	12.3	26.5	41.8
Raw sludge biochar	Cu	2.1	5.2	12.5	80.2	92.7
Raw sludge biochar	Pb	1.5	4.8	11.3	82.4	93.7
Raw sludge biochar	Zn	18.5	28.8	12.7	40	60.1
MgC-modified biochar	Cd	12.4	20.3	25.6	41.7	67.3
MgC-modified biochar	Cu	0.8	3.1	18.2	77.9	96.1
MgC-modified biochar	Pb	0.6	2.5	16.8	80.1	96.9
MgC-modified biochar	Zn	8.2	15.7	22.4	53.7	76.1
MnC-modified biochar	Cd	10.5	18.2	27.1	44.2	71.3
MnC-modified biochar	Cu	0.7	2.8	19.5	77	96.5
MnC-modified biochar	Pb	0.5	2.1	17.2	80.2	97.4
MnC-modified biochar	Zn	7.5	14.3	23.8	54.4	78.2
KC-modified biochar	Cd	8.3	15.6	30.2	45.9	76.1
KC-modified biochar	Cu	0.5	2.2	21.3	76	97.3
KC-modified biochar	Pb	0.3	1.8	18.5	79.4	97.9
KC-modified biochar	Zn	6.1	12.5	25.3	56.1	81.4

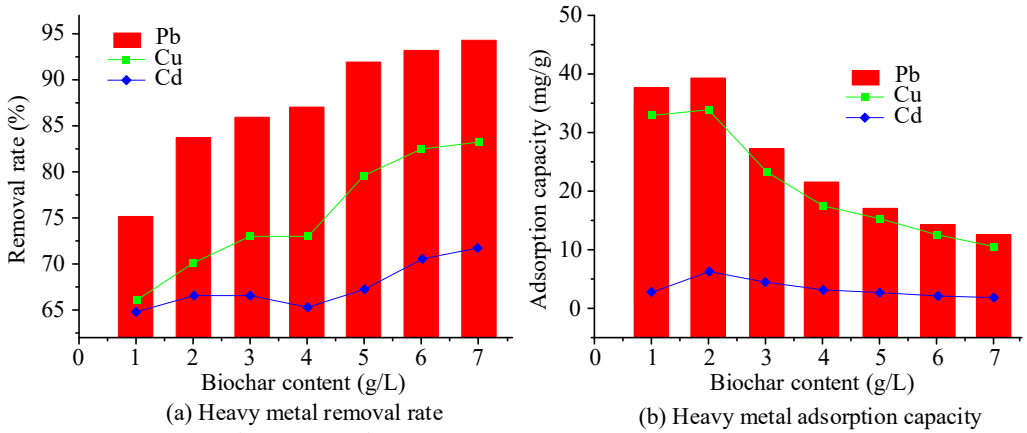

Figure 4. Removal rate and adsorption capacity of heavy metals.

Analysis of sludge biochar adsorption performance

The results of heavy metal removal rates under different biochar contents showed that, as the biochar content increased, the removal rate of Cd, Cu and Pb by sludge biochar gradually increased. However, sludge biochar had the best removal rate of Pb. When the biochar content was 7 g/L, the maximum removal rate was 95.4%, while the maximum removal rates of Cu and Cd were 83.7% and 72.4%, respectively (Figure 4a). The heavy metal adsorption capacity under different biochar contents showed that, as the biochar content increased, heavy metal contaminating ions like Cd^{2+}, Cu^{2+}, and Pb^{2+} adhered and expanded on the biochar surface. When the biochar content continued to expand and reach a certain concentration, the metal adsorption amount gradually decreased. The heavy metal adsorption capacity reached the maximum value when the biochar content was 2 g/L, where the adsorption rate of Pb^{2+} reached the maximum of 35.7%, which was the highest among the three heavy metal ions (Figure 4b).

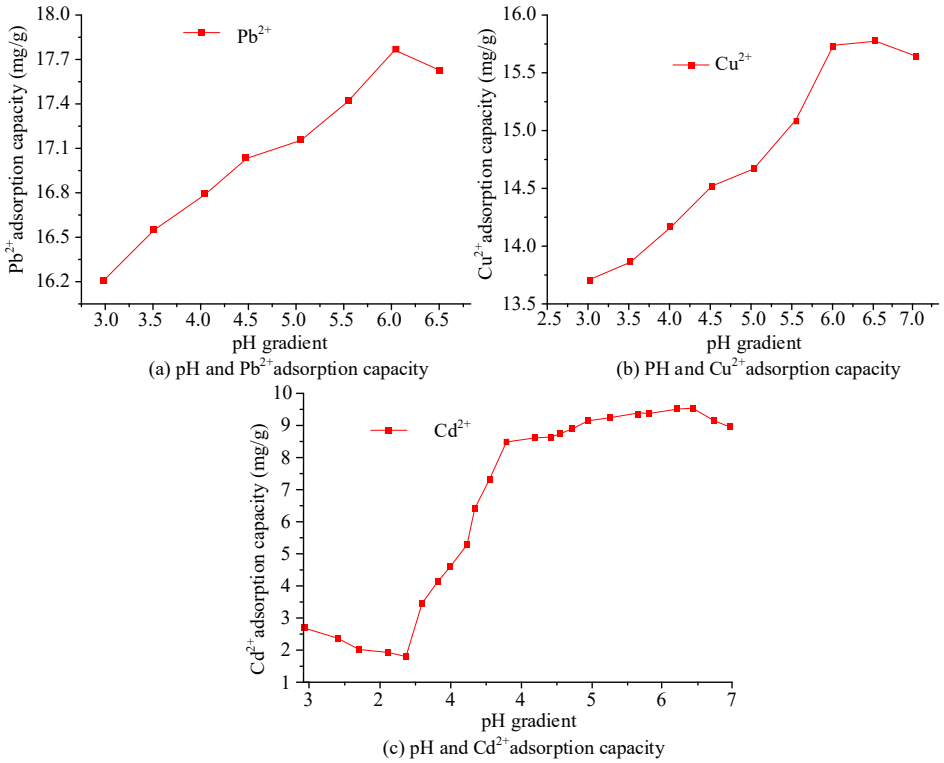

Figure 5. The effect of pH value on the adsorption capacity of sludge biochar.

The pH of the sewage also affected the adsorption effect of the three types of heavy metals. The adsorption amounts of Pb^{2+} at different pH values showed that, when the environmental pH was 6, the Pb^{2+} adsorption effect was maximized with the adsorption amount of 17.8 mg/g (Figure 5a). When the pH was 6.5, the sludge biochar had a maximum adsorption capacity for Cu^{2+} as 15.7 mg/g (Figure 5b). The best Cd^{2+} adsorption capacity was obtained at pH 6.5 as 9.6 mg/g (Figure 5c). The sludge biochar had the best adsorption effect on three heavy metal ions when the pH value was within 6 to 6.5. The concentration of heavy metals in sewage also affected the adsorption effect. The higher the concentration, the better the adsorption effect. When the concentrations of Pb^{2+} and Cu^{2+} reached 250 mg/L, the maximum adsorption capacities were obtained as 36.1 mg/g and 44.0 mg/g, respectively (Figure 6a). When the Cd^{2+} concentration was 50 mg/L, the adsorption capacity of Cd^{2+} by sludge biochar was the largest as 34.6 mg/g (Figure 6b).

Adsorption performance of modified sludge biochar

In the single sample Cd^{2+} adsorption performance test at five different concentrations, the higher the Cd^{2+} concentration, the stronger the adsorption performance of the modified sludge biochar. The KC adsorption performance was the best. When the concentration of Cd^{2+} was 50 mg/L, MgC, MnC, and KC all achieved their maximum adsorption capacities as 43.2, 45.1, and 47.3 mg/L, respectively, which were all higher than the original sludge biochar's 34.6 mg/L, indicating that the modified sludge biochar had significantly improved the Cd^{2+} adsorption capacity. Further, the adsorption performance of the five kinds of mixed samples was lower than that of single Cd^{2+}. The higher the Cd^{2+} concentration in the mixed sample, the stronger the adsorption performance on Cd^{2+}. KC had the best adsorption capacity in mixed sample 3 with an adsorption capacity of 29.5 mg/L, while the adsorption capacities of MnC and MgC were 25.3 mg/L and 24.4 mg/L, respectively (Figure 7a). The

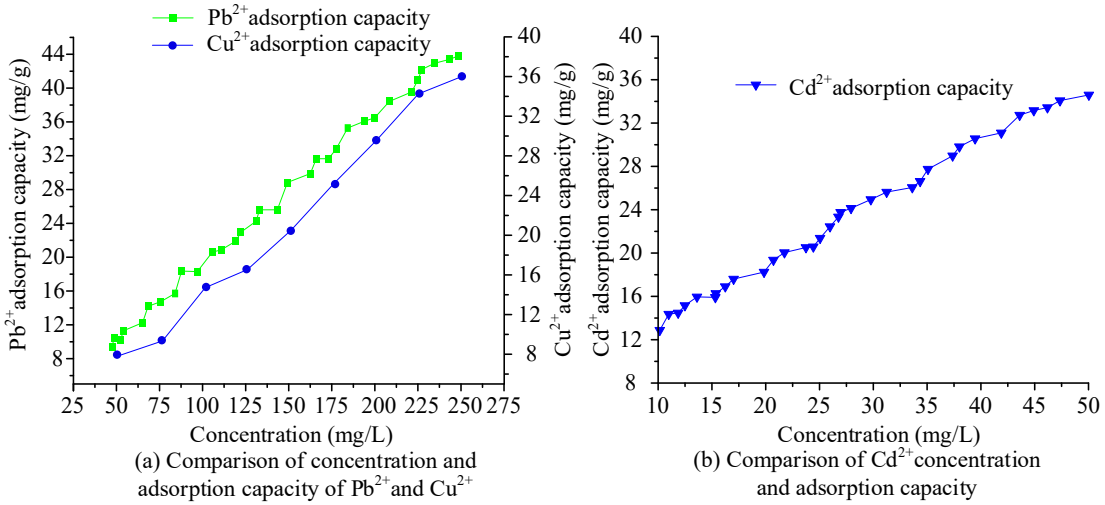

Figure 6. Relationship between sludge biochar and concentration.

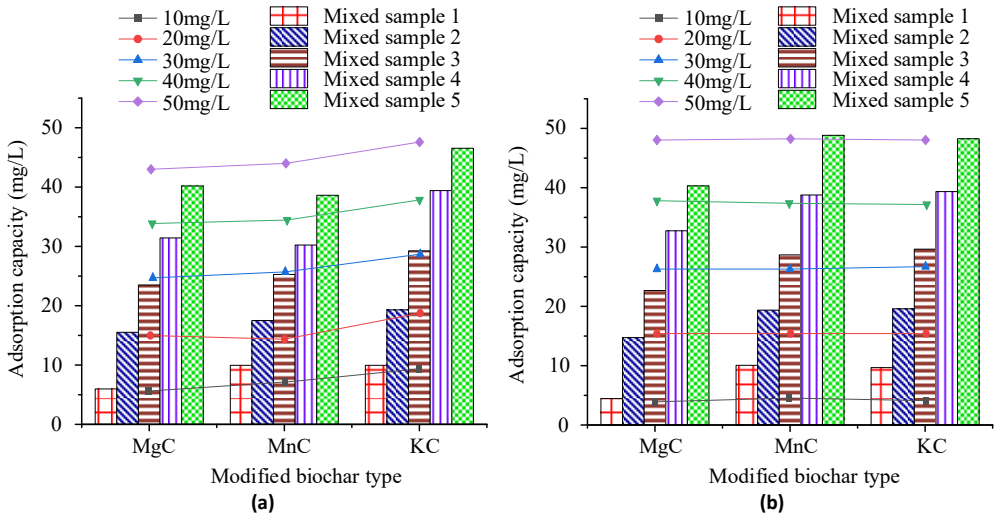

Figure 7. Analysis of adsorption performance of modified sludge biochar on Cd^{2+} (**a**) and Zn^{2+} (**b**).

adsorption performance results of three modified sludge biochars for Zn^{2+} and Zn^{2+} mixed samples showed that, when adsorption for single Zn^{2+}, the higher the concentration of modified sludge biochar, the better the adsorption effect. When the Zn^{2+} was 40 mg/L, the adsorption capacities of MgC, MnC, and KC were 38.2, 37.2, and 97.0 mg/L, respectively. Overall, the adsorption performance of the three modified sludge biochars in a single Zn^{2+} adsorption experiment was close and better than the original sludge biochar. In the mixed sample test, the adsorption capacity of the three modified sludge biochars decreased to a certain extent. However, in the high-concentration mixed sample 5, the adsorption performances of MnC and KC were improved as 49.8 mg/L and 49.2 mg/L, respectively, while MgC performed generally only 42.1 mg/L (Figure 7b). The adsorption effect on Pb^{+} showed that, in a single Pb^{2+} adsorption test, the adsorption effect of KC was better than that of MnC and MgC. When the Pb^{2+} concentration was 50 mg/L, MgC, MnC, and KC all reached the maximum adsorption effects

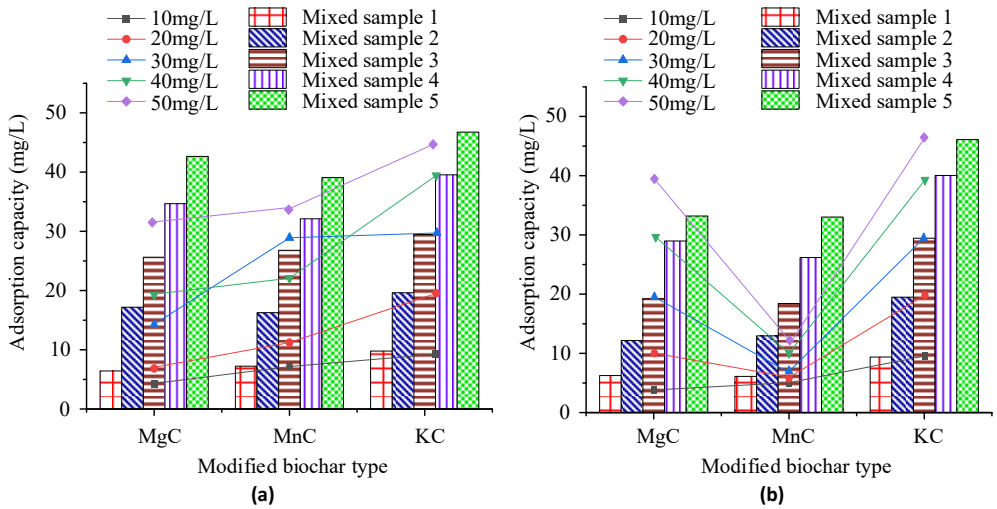

Figure 8. Analysis of adsorption performance of modified sludge biochar on Pb^{+} (**a**) and Cu^{2+} (**b**).

of 46.3, 34.1, and 32.1 mg/L, respectively, which were better than the original sludge biochar. The adsorption rate of KC reached 92.6%. In the mixed sample test, both MgC and KC had better adsorption properties, and the adsorption effect was better than that of single Pb^{+}. In high-concentration mixed sample 5, the adsorption capacity of KC was 47.2 mg/L, while the adsorption capacities of MnC and MgC were 39.7 and 43.5 mg/L, respectively (Figure 8a). In a single Cu^{2+} adsorption experiment, the adsorption capacity of MnC for Cu^{2+} was weaker than that of MgC and KC, which might be related to the influence of its functional groups. When the maximum concentration of Cu^{2+} was 50 mg/L, the maximum adsorption capacity of MnC was 12.3 mg/L, which was lower than that of KC and MgC (46.1 mg/L and 40.3 mg/L). However, in the low concentration Cu^{2+} adsorption test of 10 mg/L, MgC and MnC performed close to each other, which was weaker than KC adsorption performance. In the mixed sample adsorption performance test, after mixing multiple heavy metal substances, the adsorption performance of MnC was improved, and the adsorption performance at high concentration was close to that of MgC, which suggested that, under high-concentration mixed samples, the adsorption performance of MnC was no longer affected by a single Cu^{2+}, and its adsorption capacity was improved in mixed samples of various concentrations. The adsorption performance of MgC in the mixed sample was somewhat lower than that of single Cu^{2+}, which indicated that MgC had a greater affinity for the adsorption of Cu^{2+}. Overall, KC had excellent adsorption capacity under high concentration mixing of five samples. The adsorption capacities of MgC, MnC, and KC modified sludge biochar were 45.8, 34.1, and 34.2 mg/L, respectively (Figure 8b). The results indicated that modified sludge biochar could effectively improve the adsorption performance, and the heavy metal adsorption performance of different modified sludge biochar was affected by concentration and metal ions, and the adsorption effects were different. The modified KC sludge biochar had the best overall performance.

Discussion

As a coupling carrier for sludge resource utilization and heavy metal pollution control, sludge biochar mainly realizes the functional transformation of sludge through the pyrolysis process [15, 16]. Sludge biochar decontamination mainly relies on its physical and chemical adsorption characteristics. The porosity of sludge biochar provides adsorption sites for physical

adsorption, while functional groups such as surface carboxyl and phenolic hydroxyl groups achieve chemical adsorption through ion coordination and exchange [17]. Some active materials can improve the adsorption performance of sludge biochar such as $MgCl_2$, $KMnO_4$, KOH, *etc*. [18]. $MgCl_2$ is introduced into the sludge biochar through the impregnation method, which can increase the positive charge density on the surface of the biochar, play a pore-forming role during the pyrolysis process, and effectively improve the adsorption capacity. $KMnO_4$ has strong oxidizing properties and can oxidize organic matter on the surface of biochar. Its combination with sludge biochar can generate more functional groups such as carboxyl groups and quinone groups to enhance the chemical adsorption performance of the material. KOH as a strong base etches the surface of biochar through activation, significantly increases the pore structure and specific surface area, and greatly enhances the adsorption capacity [19]. This research found that the heavy metal removal rate gradually increased as the dosage increased because more dosages provided sufficient adsorption sites. However, excessive addition might cause biochar agglomeration and the effective specific surface area decrease. The results verified that adsorption efficiency should be balanced between the total number of sites and the interface contact efficiency [20]. The adsorption effect was optimal when the pH was within 6 - 6.5, which was due to the interfacial chemical effect. The deprotonation of the functional groups in this pH range was appropriate, which enhanced the electrostatic attraction with heavy metal cations. If the pH was too low, the protonation of the functional groups of the material would cause repulsion and affect the sludge biochar adsorption effect. In the heavy metal adsorption experiments with different concentrations, the influence of heavy metal concentration followed the law of mass transfer kinetics as the greater the heavy metal concentration, the better the adsorption performance of sludge biochar [21]. In the modified sludge biochar, SEM showed that the modified carbon had more pores than the original carbon, and the characteristic peaks of hydroxyl and carboxyl groups in FTIR were stronger, confirming the optimized structure and the increased functional groups. In addition, the three modified sludge biochars differed in their adsorption properties. In the single heavy metal system, KOH had good adsorption properties for Cd^{2+}, Pb^{+} and Cu^{2+}, while MnC had better performance in Zn^{2+}. In the mixed sample system, the adsorption capacities of the three modified carbons were slightly lower than that of the single sample, which indicated that there was a competitive adsorption relationship for heavy metal ions. KC had the highest overall adsorption capacity and the largest decrease in competition, indicating that the adsorption system dominated by pore structure was more sensitive to ion competition. The prepared modified sludge biochar performed well in the heavy metal adsorption test, which verified that the modified sludge biochar could optimize the adsorption performance by increasing the pore capacity and the functional groups on the surface of the material. However, there were shortcomings. The research only considered factors such as pH and heavy metal concentration and ignored external influencing factors. Further, only common modified materials were explored, which could not determine the best modified materials. Future research should consider various factors such as temperature and experimental environment and add more modified material analysis to improve the application effect of sludge organisms.

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