



Remediation of Heavy Metals Contaminated Soil Using Biochar-Modified Kaolin by Stabilization Technique

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ABSTRACT: Soil contamination with heavy metals threatens ecosystems and human health. The objective of this paper is to evaluate the remediation of heavy metals (Pb, Cr, Cd, and Zn) contaminated soil using biochar-modified kaolin for the stabilization of Pb, Cr, Cd, and Zn in soils. Scanning electron microscope (SEM) X-ray diffraction (XRD) were used to analyze surface morphologies and phase compositions of remediation materials. Stock solutions of different metals were spiked onto the pre-weighed soil with 1mg/kg Pb, 2mg/kg Cr, 1.5mg/kg Zn, and 1mg/kg Cd concentrations above standards, respectively. This was then allowed for 1month aging period for acclimatization. After the aging period, the spiked soil was then treated with kaolin, biochar and biochar-modified kaolin in a 45 mL polypropylene centrifuge tube and left for a week period for the remediation process. The toxicity characteristics leachability procedure (TCLP) test was used to assess the leachability tendency of residual metals in the treated soil. Amongst the biochar-modified kaolin, the 7:3 (kaolin:biochar ratio in grams) was the most effective compared to raw kaolin and biochar alone in stabilizing the target metals in the treated soil. To optimize the whole process, pretesting of the composites was conducted under constant temperature, dosage, contact time and pH. The residual concentrations of the target metals after TCLP were 2.321%, 10.846%, 0.091%, 0.295% and 3.796%, 22.133%, 0.136%, 0.561% after 30 minutes and 1-hour extraction for Pb, Cr, Cd, and Zn, respectively. The research findings demonstrate that biochar modification enhances kaolin's ability to firmly adsorb the target metals. Thus, the research presents a promising approach for the cost-effective and sustainable remediation of heavy metals in contaminated soils.

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The pollution of soils with heavy metals due to industrial and urban activities has become severe, potentially detrimental to the ecosystem and human health (Hu *et al.*, 2017). Natural processes such as precipitation and surface runoff, as well as various human activities like metal ore mining and smelting,

industrial discharge, automobile emissions, and agricultural practices (such as using synthetic pesticides, industrial waste, and industrial or domestic sludge for wastewater irrigation) have made soils a major reservoir for heavy metals, which has led to heavy metal contamination of urban and agricultural

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soils (Ou JieYong *et al.*, 2018). Recently in Nigeria, the primary sources of metals in agricultural soils include atmospheric deposition, livestock manure, fertilizers, agrochemicals, mining, auto-mechanic workshops, sewage irrigation, and sewage sludge (Shibdawa *et al.*, 2019; Olofade *et al.*, 2015; Umar and Adamu, 2014; Adamu *et al.*, 2013; Wuana and Okieimen, 2011; Adamu *et al.*, 2011).

Heavy metals are inorganic chemical hazards commonly found in contaminated soils. Unlike organic contaminants, which can be oxidized to CO₂ by microbial action, heavy metals are non-degradable and cannot be completely eliminated through natural attenuation or chemical degradation. As a result, they persist for a long time after being introduced (Babakura *et al.*, 2023; Adamu, 2021; Adamu *et al.*, 2019; Adamu *et al.*, 2018; Adamu *et al.*, 2017; Adamu *et al.*, 2016; Adamu and Anderson, 2015; Kumpiene *et al.*, 2008). Therefore, considering their ecological and human toxicity through direct ingestion or contact with contaminated soil, as well as their impact on the food chain (soil-plant-human or soil-plant-animal-human), and the drinking of contaminated ground water, the remediation of soils contaminated by heavy metals deserves significant priority (Wuave *et al.*, 2024a; Wuave *et al.*, 2024b). In response to the negative effects of heavy metal contamination, there has been ongoing development of various technologies to remediate affected soil. The traditional method of dealing with heavy metal contamination involves excavating the soil and dumping it into landfills. However, this method is often not considered cost-effective or environmentally sustainable (Wuave *et al.*, 2024a; Wuave *et al.*, 2024b; Boisson *et al.*, 1998). Several alternative options have been explored as less intrusive and more cost-effective solutions. One technology that has garnered significant attention is *in situ* immobilization of heavy metals in soils through the addition of different amendments. This method involves adding an amendment to soil to increase the proportion of total soil metal content within the solid phase, either through precipitation or increased metal sorption, thereby reducing metal solubility (Oste *et al.*, 2002).

The presence of these materials reduces the movement of heavy metals into water, plants, and other parts of the environment (Yao *et al.*, 2012). As a result, the metals are not able to pose a risk to human health through the food chain. Therefore, the goal of immobilization is not to eliminate the metal contaminant from the soil, but to decrease its bioavailability and activity. This in turn alleviates the impacts of metals on plants, animals, and groundwater. Many different amendments have been

proposed and tested for *in situ* immobilization of heavy metals in soils. In recent years, clay minerals have been used as amendments for remediating heavy metal-polluted soil due to their low cost, abundant reserves, and high performance. Clay minerals are one of the most abundant materials on earth. Clay minerals have played a crucial role in the development of human civilization. They have been used for environmental protection, including the disposal and storage of hazardous chemicals and heavy metals (Ismadji *et al.*, 2015). This has led to extensive research on various clay minerals, such as sepiolite, illite, calcite, goethite, montmorillonite, bentonite, and kaolinite, for remediating heavy metal-contaminated soils. (Hossain *et al.*, 2010) More recently, there has been increasing interest in the potential of biochar for sorption of contaminants and their subsequent immobilization, particularly in the remediation of contaminated soil and groundwater (Qiu *et al.*, 2022; Ahmad *et al.*, 2014). Biochar has a high surface area and favorable pore architecture, enabling efficient retention of contaminants, including metals/metalloids and organic pollutants in soils (Qiu *et al.*, 2022; Mohan *et al.*, 2014).

In order to harness remediation abilities of these materials, modification techniques such as synergistic combinations of clays and biosorbents (both being low cost adsorbents) has received our attention. Hence, the objective of this paper is to evaluate the remediation of heavy metals (Pb, Cr, Cd, and Zn) contaminated soil using biochar-modified kaolin.

MATERIALS AND METHODS

Synthesis of biochar-modified kaolin sorbent: The following mass ratios in grams (9:1, 8:2, 7:3, 6:4, 5:5, 4:6, 3:7, 2:8, 1:9) of purified kaolin and biochar were weighed and labeled as A, B, C, D, E, F, G, H, I. These were then added to a 1L beaker containing 300 mL of 0.1 M NaOH solution. The mixture was stirred and heated in an oven at 105°C until it was completely dry. Afterwards, the samples were placed in separate crucibles and calcined at 300°C for 6 hours. The resulting dark powdery material was washed multiple times to remove any remaining NaOH, and then dried at 105°C. The pure kaolin, the biochar, and the biochar modified kaolin sorbents are referred to as KLN, BC, and BMK, respectively.

Characterization of the sorbents: X-ray diffraction (XRD) patterns for KLN, BC, and BMK were obtained using a powder X-ray diffraction (XRD) system from PANalytical model X'Pert PRO. The system used Cu-K α ($\lambda = 1.54 \text{ \AA}$) radiation at 45 kV and 40 mA. All the samples were scanned from 5.0 to 90° at a scan speed of 2° per minute. The FTIR spectra

were recorded on a Varian spectrometer model 7000 using KBr pellet samples within the range of 500 to 4000 cm⁻¹. The surface morphologies of KLN, BC, and BMK were examined using a Zeiss EVO40 scanning electron microscope (accelerating voltage 20,000 V, and working distance 10,000-10,500 mm). Samples were coated with a thin layer of gold, mounted on a copper stub using double stick carbon tape, and surfaces were made conductive with gold coatings. Elemental compositions of the samples were determined from SEM analyses.

Soil incubation experiments: The experiment involved adding stock solutions of different metals onto pre-weighed soils in separate glass jars. These solutions were spiked to obtain concentrations of 1mg/kg Pb, 2mg/kg Cr, 1.5mg/kg Zn, and 1mg/kg Cd above the NSDWQ, EPA, and WHO standards. The jars were vigorously shaken overnight to ensure the metals were evenly distributed in the soils. The mixture was then allowed to age for 1 month for acclimatization. After the aging period, the spiked soils were ready for the controlled experiment. For individual tests, a specific amount of spiked soil and 1, 2, 4, or 6% of kaolin, biochar, and biochar-modified kaolin were mixed in a 45 mL polypropylene centrifuge tube. A certain moisture content was achieved by adding 2.5mL of deionized water to the mixture. The tubes were vortexed for approximately 2 minutes and then placed on a rotator for a one-week incubation period. Control experiments were conducted without any sorbents, and untreated soils will be mixed with raw kaolin and raw biochar separately.

Toxicity characteristics leachability tests: The research performed Toxicity Characteristic Leaching Procedure (TCLP) tests to assess the potential for soil metal leaching and to evaluate the effectiveness of various biochar-modified kaolin samples in immobilizing metals. In the initial experiment, I used a 4% adsorbent-to-soil ratio, and then I conducted additional experiments with different adsorbent dosages and extraction times (Mohamed *et al.*, 2023). The test procedure followed the US EPA standard protocol TCLP Method 1311 (Yu *et al.*, 2009). In short, pre-weighed soil samples were mixed with a leaching solution (pH = 2.88±0.05) at a liquid-to-solid ratio of 1:20. The leaching solution is prepared by adding 5.7 ml of glacial acetic acid (CH₃CH₂OOH) to 1 liter of deionized water. The containers were rotationally shaken for 18 hours at 30 rpm. After centrifugation, the concentration of metal in the supernatants was measured using atomic absorption spectrophotometry (FAAS, Buck Scientific VGP210). Calibration curves were prepared for each metal element. The experiment varied time and dosage as

shown above, while pH and temperature were kept constant. To optimize the whole process, pretesting of the composites was conducted under constant temperature, dosage, time and pH. The composite that exhibited optimal removal was determined for further analyses under varying time and dosage. The stabilization of the tested metals in the soils through soil amendment with biochar modified kaolin was determined based on the leachability test using the following equation:

$$\text{Leachability} = C_L V_L / C_S M_S \times 100 \text{ (Alghanmi et al., 2015).}$$

Where C_L is the concentration of metal in the leachate (mg/L); V_L is the volume of added extraction solution (L); C_S is the concentration of metal in the untreated soil (mg/Kg); M_S is the mass of soil (Kg).

RESULTS AND DISCUSSION

Characterization of kaolin and biochar: The XRD pattern of the raw kaolinite, biochar and BMK materials are shown in Fig. 1b. The various visible peaks on the spectra of all the synthesized materials demonstrate the presence of crystalline minerals and inorganic materials. The XRD patterns of raw kaolinite clay and biochar were identified. In the XRD pattern of kaolinite, the strongest intense peaks were identified at $2\theta=12.46^\circ$; 25.06° and 26.68° (Fig. 1a) and lower diffraction kaolinite peaks than previous peaks were revealed at $2\theta=36.7^\circ$; 39.50° ; 42.56° ; 50.3° ; 55.16° and 62.4° . Four peaks of cellulose in biochar are displayed at $2\theta=21.8^\circ$; 22.4° ; 24.3° and 30° , peak of carbon-containing minerals mellite is at $2\theta=14.8^\circ$, and calcite at $2\theta=39.78^\circ$ (Fig. 1a) (Smith *et al.*, 2017; Sarkar *et al.*, 2010). The kaolinite peaks were found in BMK materials, which endorsed successfully implanted biochar onto the kaolinite matrix. Peaking is a sign of interactions between kaolinite and biochar during synthesis. Peaks of kaolinite at $2\theta=25.06^\circ$ in BMK 5:5 was shifted to 25.2° , and $2\theta=36.86^\circ$ in BMK 7:3 was shifted to 36.48° (Fig. 1b). The other peaks in biochar based materials were impurities corresponding to calcite and quartz. The XRD analysis of biochar (Fig. 1b) displayed peaks at $2\theta=23.04^\circ$, which indicated the presence of graphite. Other peaks were identified at $2\theta=29.3^\circ$; 39.44° ; 43.1° ; 47.34° and 48.28° , and indicated the presence of calcite. Likewise, the peak of cellulose in HC is at $2\theta=22.4^\circ$ and the peak of mellite is at $2\theta=14.8^\circ$. In biochar, mellite was lost during the pyrolysis process of BMK. Therefore, the changes in the results of the BMK composite showed that the synthesis method used successfully implanted kaolinite onto the kaolinite matrix.

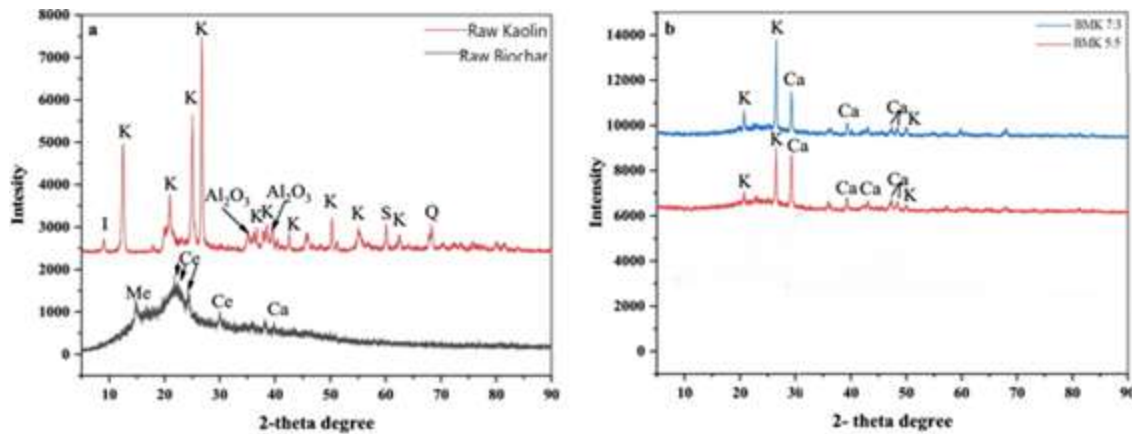


Fig 1: XRD of Kaolin

Fig. 2 (A), (B) and (C) show the SEM morphological micrograph of the kaolin sample at 500x, 1000x and 1500x magnifications. The sample retained a dispersed lump-like morphology, a tetrahedral or hexagonal imperfect crystal shapes. The average particle size is estimated at 500 μm constant. SEM images in the figure above show the surface morphology of the synthesized materials.

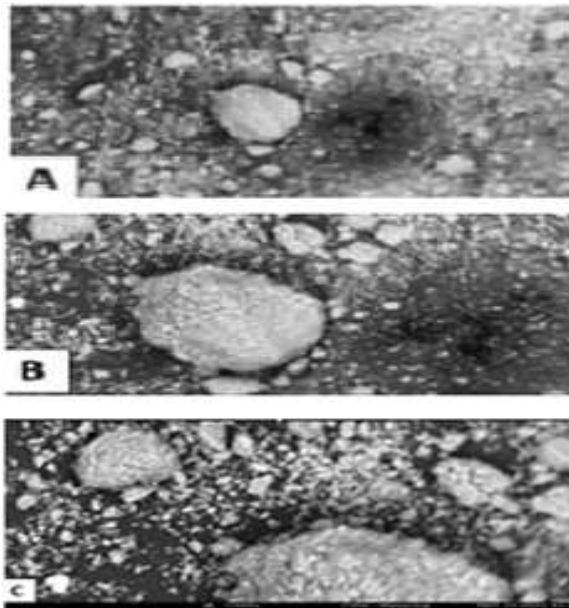


Fig. 2 (A), (B) and (C) show the SEM morphological micrograph of the kaolin sample at 500x, 1000x and 1500x magnifications.

Comparing raw biochar and Kaolin with their modifications will help us to study the morphological changes during pyrolysis. Pyrolysis converted the crystalline surface of biochar into porous and amorphous materials, as presented in the above figures. The surface area of the raw materials was generally coated by thin film structures and were more irregular on the BMK showing that at

3000 \times magnification (Fig. 3), the biochar was well onto the surfaces and within the pores kaolinite. The decomposition and volatilization of biomass caused a small number of pores with different sizes to appear on raw kaolinite and biochar. In addition, the SEM images showed that kaolinite was not entirely covering the surfaces of the BMK material.

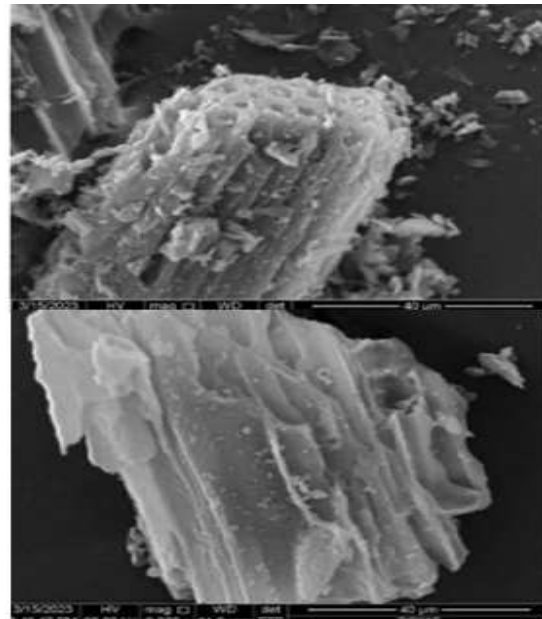


Fig. 3 shows the SEM morphological micrograph of the BMK sample at 3000x magnifications.

Leachability tests: To obtain the best composite for replications in the experiment, an optimization was conducted in two batches A and B where dosage is 4% and time were varied while temperature is 27-30 $^{\circ}\text{C}$ and pH 2.88 were kept constant. The summary of findings for the removal of the targeted heavy metals by each modified composite is in the table below. The residual concentrations of metals in leachates of the batch A after 30 minutes of extraction is presented in Table 1.

Table 1: Residual concentrations of metals in leachates in batch A experiment

Modifications	Pb (%)	Cr (%)	Cd (%)	Zn (%)
A	0.06918	0.26211	0.00668	0.00412
B	0.05361	0.17944	0.00097	0.00440
C	0.02321	0.10846	0.00091	0.00295
D	0.08030	0.24826	0.00237	0.00389
E	0.08242	0.13597	0.21810	0.00329
F	0.06799	0.12182	0.70454	0.00314
G	0.05120	0.38294	0.49680	0.00345
H	0.06759	0.41076	0.79323	0.00321
I	0.01047	0.48585	0.80812	0.00343
CK	0.53601	0.55925	0.66199	0.85512
CB	0.34793	0.42988	0.47664	0.51087

Based on the results in Table 1, the modification with the least amount of metals in the leachate was Modification C (with the 7:3 kaolin:biochar ratio in grams). The modification allowed only 2.321%, 10.846%, 0.091%, and 0.295% of Pb, Cr, Cd, and Zn, respectively to leach out of the spiked concentrations after the soil was treated with the adsorbent. These results are significant because they show the potential of the modification (treatment with the adsorbent) for reducing the leaching of heavy metals (Pb, Cr, Cd, and Zn) from contaminated soil. The modification significantly reduced the amount of lead (Pb), chromium (Cr), cadmium (Cd), and zinc (Zn) that leached out of the soil. The reported concentrations (2.321%, 10.846%, 0.091%, and 0.295%) are much lower compared to the initial spiked concentrations (1, 2, 1, 1.5 mg/Kg). This implies that only 2% of 1 mg/Kg of Pb, 10% of 2 mg/Kg of Cr, 0.091% of 1 mg/Kg of Cd, and 0.295% of 1.5 mg/Kg of Zn were leached out after the soil's remediation management. This suggests that the adsorbent effectively captured a large portion of the heavy metals within the soil matrix. This can potentially minimize the risk of metal contamination in the surrounding environment, such as groundwater or agricultural land. The fact that these reductions were achieved using only 4% dosage of adsorbent per Kg of soil indicates good efficiency. This could be advantageous for practical applications due to potentially lower remediation costs. However, the experiment seems to be conducted under controlled conditions. Real-world application might require adjustments based on specific soil properties and environmental factors. Overall, these results are promising for developing a method to reduce heavy metal mobility in contaminated soils using the tested adsorbent. In the same vein, Table 2 presents the residual concentrations (after treatment) of various metals (Pb, Cr, Cd, Zn) for different modifications (labeled A-CK, CB) in Batch B of the experiment. This represents the residual concentrations of metals in leachates of the batch B after 1 hour of extraction. Compared to results that might be expected from Table 1 (with similar initial concentrations), the residual metal concentrations in Batch B seem

generally higher across all modifications. This suggests that the treatment in Batch B might be less effective in immobilizing metals compared to Batch A (with both batches used the same initial metal concentrations). Despite the increased residual concentrations, based on the results in Table 2, the modification with the least amount of metals in the leachate was still Modification C (with the 7:3 kaolin:biochar ratio in grams).

Table 2: Residual concentrations of metals in batch B experiment

Modifications	Pb (%)	Cr (%)	Cd (%)	Zn (%)
A	0.07598	0.31825	0.00018	0.00291
B	0.06592	0.38388	0.01923	0.00285
C	0.03796	0.22133	0.00136	0.00561
D	0.07592	0.34904	0.48453	0.00218
E	0.02645	0.49669	0.01028	0.00289
F	0.05824	0.56333	0.00775	0.01693
G	0.06571	0.63957	0.00559	0.00443
H	0.07001	0.69436	0.00131	0.00429
I	0.06488	0.97226	0.07486	0.00553
CK	0.73601	0.75925	0.86199	0.65512
CB	0.70793	0.66988	0.87664	0.71087

The modification allowed only 3.796%, 22.133%, 0.136%, and 0.561% of Pb, Cr, Cd, and Zn, respectively to leach out of the spiked concentrations after the soil was treated with the adsorbent. Therefore, similar to Table 1, there is significant variation in the effectiveness of different modifications. Yet, Modification C still shows the lowest residual concentration for all the metals. However, this suggests that extending the extraction time from 30 minutes to 1 hour can lead to increased leaching, even with the modification. This emphasizes the importance of optimizing treatment time to achieve a balance between maximizing adsorption and minimizing residual metals. Overall efficiency, even in Batch B with a longer extraction time, the leached metal percentages are relatively low (highest being 22.133% for Cr). This suggests the modification offers promising overall efficiency in reducing metal mobility. In short, these results are encouraging for the potential of the modification to treat contaminated soil. However, optimizing the treatment duration seems crucial for maximizing its effectiveness.

Conclusion: This study investigated the potential of biochar-modified kaolin for the cost-effective and sustainable remediation of heavy metal-contaminated soils. Pb, Cr, Cd, and Zn were targeted due to their detrimental effects on ecosystems and human health. Biochar modification significantly enhanced kaolin's ability to immobilize Pb, Cr, Cd, and Zn in soil compared to raw kaolin and biochar alone. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) analyses provided insights into the surface morphology and phase composition changes upon

biochar modification, potentially contributing to improved adsorption capacity. The Toxicity Characteristic Leachability Procedure (TCLP) test demonstrated significant reductions in leachable metal concentrations after treatment with biochar-modified kaolin. Therefore, this research demonstrates the promising potential of biochar-modified kaolin for sustainable and cost-effective heavy metal-contaminated soil remediation. This approach offers a valuable strategy for mitigating the environmental and health threats posed by heavy metal-contaminated soils. Future research could explore large-scale application and investigate the long-term stability of biochar-modified kaolin under various environmental conditions. Additionally, optimizing the regeneration process for reusability could further enhance the economic and environmental benefits of this remediation technique.

Declaration of Conflict of Interest: The authors declare no conflict of interest

Data Availability Statement: Data are available upon request from the first author or corresponding author.

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