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Research article

Effect of *Citrus sinensis* peel-derived biochar on the concentrations of heavy metals in soil irrigated with municipal wastewater

Sanusi Yakubu Lawal[1,*], *Samaila Muazu Batagarawa*[2], *Aminu Musa*[3]

[1]Department of Industrial chemistry, Federal University Dutsinma, Katsina State, Nigeria
[2,3]Department of Pure and Industrial Chemistry Umaru Musa Yaradua University, Katsina, Katsina State, Nigeria

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ABSTRACT

Wastewater is widely used for irrigation and can accumulate heavy metals in the soil, potentially causing harm to human health and the environment. In this study, a pot experiment was used to assess the impact of biochar in soil properties enhancement as well as mitigation of the concentration of Cd, Cu, Zn and Pb to lettuce (Lactuca Sativa) in a soil irrigated with wastewater. Citrus peel biochar pyrolised at 400°C was applied on soil in three levels (0%, 5%, and 10%). The lettuce plant was harvested after 28 days of planting, the concentrations of heavy metals were determined in roots and leaves using atomic absorption spectroscopy (AAS). The result obtained shows the increase in soil properties with an increase in biochar concentration, the result also shows the p-value of leaves and roots as 0.744 which is not statistically significant at 95% confidence interval. The result obtained shows a decrease in the uptake of heavy metals by the lettuce plant in both parts of the plants (leaves and roots) as follows: the concentration of cadmium was not detected at all levels in both roots and leaves, in un-amended samples (Leaves) the concentration of Cu, Pb, and Zn were 0.141, 0.252, and 1.116 respectively while in roots the concentration of Cu, Pb, and Zn were 0.205, 0.266, and 1.248 respectively. At 5% amendment (leaves) Cu decreased by 48.2% (0.073), Pb by 42.86% (0.144), and Zn by 66.13% (0.378). At 10% amendment (leaves) Cu decreased by 71.63% (0.040), Pb by 53.57% (0.117), and Zn by 48.03% (0.580). While in roots almost similar reductions were observed. These findings show the potential of biochar as an effective strategy for soil properties improvement as well as reducing heavy metals uptake by the plants, with implications for sustainable agriculture in an environment contaminated with heavy metals.

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1. Introduction

Heavy metals enter the environment from natural and anthropogenic sources. The most important natural sources are mineral weathering, erosion and volcanism. Anthropogenic sources, on the other hand, include mining, smelting, electroplating, pesticide and (phosphate), fertilizer use, agricultural biosolids, sludge dumping, industrial waste, and atmospheric deposition (Ali et al., 2013). Various heavy metals pose potential risks to human health due to their toxic properties. Among these, cadmium, chromium, copper, mercury, lead, zinc, iron, arsenic, cobalt, and nickel are particularly noteworthy. Exposure to these metals can occur through various sources such as contaminated water, air, soil, and food. Cadmium, for instance, is associated with kidney damage, while mercury can lead to neurological disorders. Lead exposure is linked to developmental issues in children (Sharma et al. 2021; Rahmat et al. 2023; Halyal et al. 2023). The accumulation of these metals in the body over time can result in chronic health problems, making it crucial to monitor and control their presence in the environment to safeguard human well-being (Rahmat et al. 2023; Halyal et al. 2023). They occur naturally in small amounts in soil and industrial wastewater through various chemical processes such as precipitation and filtration. This poses little problem as they are generally limited in quantity or exceed acceptable limits (Halnor, 2015). Several traditional methods have been used to mitigate soils contaminated with heavy metals, but each method has several drawbacks such as low feasibility, unsustainability, cost, and environmental destruction (He et al., 2019). One inexpensive and environmentally friendly remediation method is the use of biochar, a solid carbonaceous material. Biochar is obtained by thermo chemical conversion or pyrolysis of plant or animal biomass or

* **Corresponding Author:**
Email: lyakubu@fudutsinma.edu.ng (S. Y. Lawal)
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manure (commonly called feedstock) at temperatures around 200-700°C in a less oxygen environment. Biochar has been reported to improve soil properties and affect redox processes within the soil. Such as increasing the pH of acidic and neutral soils, increases water retention, and accelerate the decomposition of pentachlorophenol.

Furthermore, a number of studies have shown that biochar can effectively reduce the mobility/bioavailability of heavy metals in contaminated soils and thus effectively reduce the uptake of heavy metals by crops (He et al., 2019). Many studies were carried out on biochar application, but most of them were batch experiment and didn't include waste water contamination mitigation. This study will assess the impact of citrus peel biochar on the toxicity of heavy metals in waste water irrigated soil. The objectives of this study are: (1) To prepare biochar from orange peel using pyrolysis process (2) To characterize the prepared biochar using FTIR analysis (3) To carryout pot experiment with pant grown in the study area (lettuce plant) (4) To compare the effect of biochar in amended and un-amended plant sample.

2. Methodology

2.1 Sample collection

Topsoil (0–20 cm depth) was collected by simple random sampling using spade at the Lambun Sarki irrigation site. Collected soils were weighed, properly mixed, placed in nylon bags and appropriately labeled (Anukwa & Nkang, 2020). The orange peels were collected from an orange selling place "Yan lemu of Kofar durbi quarters, Katsina town. The collected peels were washed several times with tap water to remove stones and dust. The peels were then air dried for 3 days then washed with distilled water to remove any adhering particulate matter. The sample was then dried in an oven at 70°C for 24hrs and ground to pass a 2mm sieve (Sial et al., 2019).

2.2 Biochar preparation

The dried peels were cut into small pieces and stored prior to pyrolysis. The sample of dry orange peels were placed in ceramic crucibles (Tran et al., 2016), weighed and wrapped in aluminum foil to reduce oxygen contact. The pyrolysis of the orange peels was carried out at temperatures of 400°C for 1 hour. After pyrolysis, the crucibles were removed from the muffle furnace and placed in a fume hood until room temperature was reached. The biochar was then crushed by mortar and pestle, sieved with 2mm mesh and stored in a well labeled polyethene bag (Lima et al., 2020).

2.3 Biochar characterization

Functional groups were identified by Fourier transform infrared spectroscopy (FT/IR-620). FT-IR spectra were recorded on a spectrometer using potassium bromide (KBr) pellets and a wave number range of 4000–600cm-1 (Oh et al., 2012)

2.4 Pot experiment

The pot experiment was carried out in a prepared green house at Lambun Sarki irrigation site, Katsina town. Black polyethylene bags were filled with 2kg of the air-dried soil, the soil samples were mixed with biochar at two levels (i.e. 5% and 10% /kg soil) by measuring and adding 100g and 200g of biochar respectively to the soil then followed by thorough mixing for each level. Biochar was not added to the control (0%) (Ghorbani et al., 2019)

2.5 Post-harvest treatment

Plant samples (lettuce) were cut into pieces, divided into roots and leaves, washed with tap water and then rinsed with distilled water. The samples were air dried and ground to a fine powder using a ceramic mortar and pestle and stored in labeled polyethene bags.

0.5 g of plant samples were digested with 10cm3 of aqua regia on a hot plate in a fume hood until clear solutions were obtained. Distilled water was added periodically to prevent the solution from drying out. Solution was filtered through Whatman No. 42 filter paper into a standard 50 cm3 volumetric flask and then topped up to the mark with distilled water (Bhardwaj et al., 2023).

2.6 2.6 Heavy metals analysis

Samples heavy metals were determined using Atomic absorption spectrophotometer PerkinElmer PinAAcle 900H. Absorbance was determined using blank solution as a reference (Sun et al., 2020).

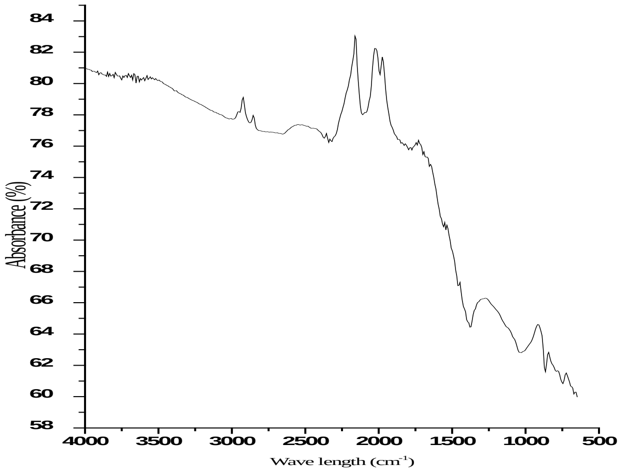


Figure 1. FTIR spectra of biochar.

3. Result and Discussion

3.1 Biochar FTIR analysis

The result illustrated in figure 1 shown the FTIR peaks observed in the biochar spectrum at various wave numbers is indicating the presence of aliphatic hydrocarbons (2900 cm-1), and functional groups like hydroxyls (3757.2cm-1), CO2 (2340.8cm-1,) alkynes (2109.7cm-1), and CO stretching (1100 cm-1) suggest the presence of diverse carbonaceous structures and functional moieties in the biochar, indicating its complex composition and potentially beneficial properties as a high-quality biochar for agricultural or environmental applications. A study by (Oh et al., 2012) on characterization of biochar from three different biomass, the presence of peaks at 2915 cm−1 (C–H), 2364 cm−1 (CO2), and 1080 cm−1 (C–O) was observed in orange peel biochar. (Abdelaal et al., 2021) characterized the orange peel biochar a peak was observe 2900 cm-1, 1400cm-1 and 11000-10000 cm-1.

3.2 Heavy metals analysis of lettuce plant (Leaves)

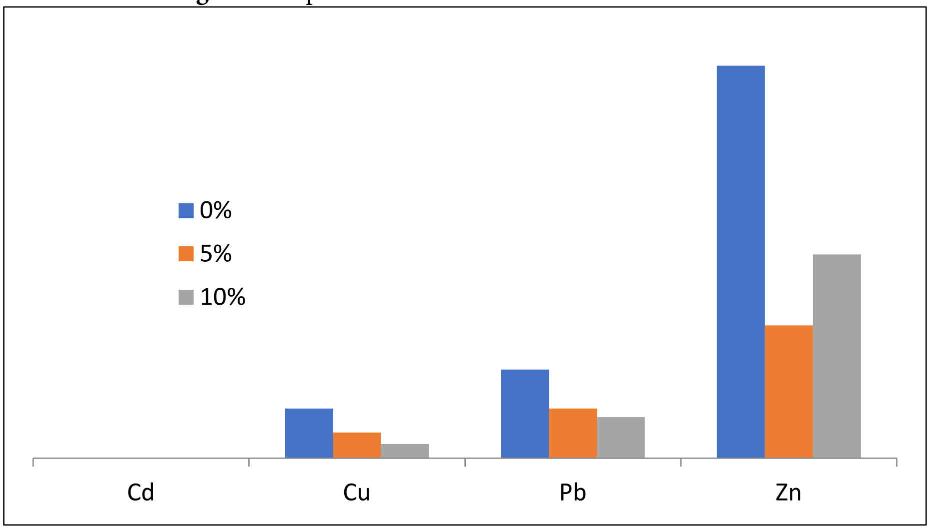
The AAS analysis of leaves from sample as shown in figure 2 demonstrated distisnctive trends in heavy metal concentrations. Cadmium (Cd) was not detected in any of the samples. In the case of copper (Cu), the concentration decreased from un-amended sample to 5% amended sample (0.141 mg/kg to 0.073 mg/kg), reflecting a percentage decrease of approximately 48% and there is also a decrease from un-amended to 10% amended sample (0.141 mg/kg to 0.040 mg/kg), representing a percentage decrease of approximately 72%. Similarly, lead (Pb) levels decreased from 0.252 mg/kg in un-amended sample to 0.141 mg/kg of 5% amended sample, resulting in a decrease of about 44% and the decrease was also found from un-amended sample to 10% amended sample (0.252 mg/kg to 0.117 mg/kg), resulting in a decrease of about 54%. For zinc (Zn), concentrations decreased from 1.116 mg/kg in un-amended sample to 0.378 mg/kg of 5% amended sample, marking a decrease of 66% and concentrations decreased from 1.116 mg/kg in un-amended soil to 0.580 mg/kg 10% amended soil, reflecting a decrease of around 48%. This interpretation provides a comprehensive analysis of the percentage decreases in heavy metal concentrations for each element when transitioning from un-amended soil to 5% amended soil and also from un-amended soil to 10% amended soil.

Table 1: Result of heavy metals analysis in the leaves of lettuce plant.

Sample Concentration (Mg/Kg)				
Metal	**0%**	**5%**	**10%**	**CNS/FAO Permissible Limit**
Cd	ND	ND	ND	0.05
Cu	0.141±0.0015	0.073±0.0013	0.040±0.0007	10.000
Pb	0.252±0.0092	0.144±0.0101	0.117±0.0124	0.20
Zn	1.116±0.0031	0.378±0.0056	0.580±0.0049	20.00

KEY: 0% = Control sample, 5% = Sample amended with 5% Biochar, 10% = Sample amended with 10% Biochar, ND = Not detected, CNS/FAO = China National Standards/ Food and Agriculture Organization

Figure 2: Representation of biochar effect in Lettuce Leaves.



KEY: 0% = Control sample, 5% = Sample amended with 5% Biochar, 10% = Sample amended with 10% Biochar, ND = Not detected

3.3 Heavy metals analysis of lettuce plant (Roots)

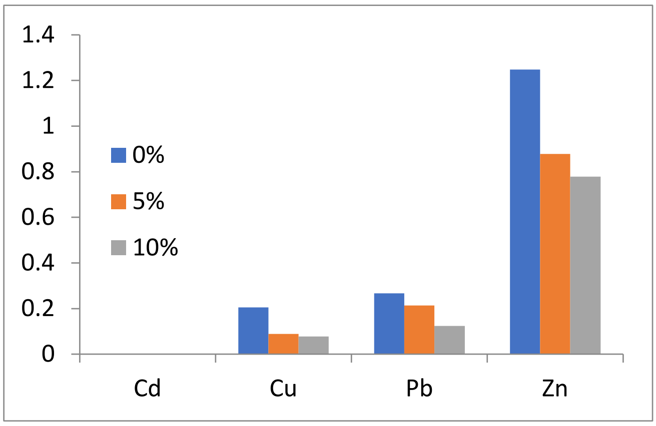
In the assessment of heavy metal concentrations in roots notable variations emerged as shown in figure 3 Cadmium (Cd) was not detected in any of the samples. Analyzing the transition from un-amended to 5% amended sample, copper (Cu) concentrations decreased from 0.205 mg/kg to 0.088 mg/kg, reflecting a significant percentage decrease of approximately 57%. Concurrently, moving from un-amended to 10% amended sample, copper (Cu) concentrations decreased from 0.205 mg/kg to 0.078 mg/kg, representing a notable percentage decrease of approximately 62%. Lead (Pb) levels decreased from 0.266 mg/kg to 0.213 mg/kg from um-amended to 5% amended sample, resulting in a percentage decrease of about 20%. Moving from un-amended to 10% amended sample Lead (Pb) levels decreased from 0.266 mg/kg to 0.123 mg/kg, resulting in a percentage decrease of about 54%. Zinc (Zn) concentrations decreased from 1.248 mg/kg un-amended to 0.878 mg/kg 5% amended sample, marking a percentage decrease of around 30%. Moving from un-amended to 10% amended sample, zinc (Zn) concentrations decreased from 1.248 mg/kg to 0.778 mg/kg, reflecting a percentage decrease of around 37%.

Table 2: Result of heavy metals analysis in the roots of lettuce plant.

Sample Concentration (Mg/Kg)				
Metal	**0%**	**5%**	**10%**	**CNS/FAO Perm. Limit**
Cd	ND	ND	ND	0.05
Cu	0.205±0.0032	0.088±0.0015	0.078±0.0026	10.00
Pb	0.266±0.0016	0.213±0.0061	0.123±0.0013	0.20
Zn	1.248±0.0012	0.878±0.0033	0.778±0.0019	20.000

KEY: 0% = Control sample, 5% = Sample amended with 5% Biochar, 10% = Sample amended with 10% Biochar, ND = Not detected, CNS/FAO = China National Standards/ Food and Agriculture Organization

Figure 3: Representation of biochar effect in Lettuce Root.



KEY: 0% = Control sample, 5% = Sample amended with 5% Biochar, 10% = Sample amended with 10% Biochar, ND = Not detected

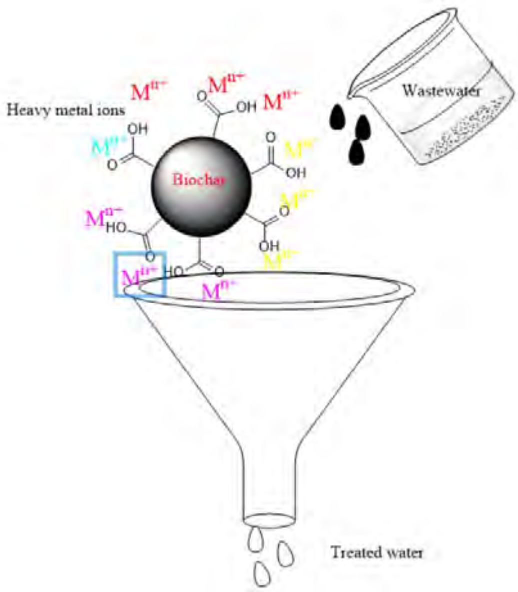


Figure 4: Representation of biochar complexation with heavy metal ions.

This study's findings align with a growing body of research exploring the impacts of biochar on heavy metal accumulation in plants. Numerous studies have reported biochar's ability to immobilize heavy metals in soils and subsequently reduce their uptake by plants. For instance, Campos & De la Rosa, (2020), demonstrated that biochar amendment significantly decreased the uptake of copper, and lead in cabbage, supporting the results observed in this study. Similarly, Namgay et al., (2010) conducted a comprehensive meta-analysis and concluded that biochar application reduced maize shoots As, Cd and Cu concentrations, especially at the highest trace element application rates, but the effects on shoot Pb and Zn concentrations were inconsistent. The reaction of biochar with heavy metals is presented in figure 4 assumed the mechanism of action.

4. Statistical Analysis

The independent T-test describes the comparison among each pair of analyzed elements in leaves and roots of the irrigated plant. The result from the figures 2-3 shows the p-value of leaves and roots as 0.744 which is not statistically significant at 95% confidence interval. This interpretation presents the percentage decreases in heavy metal concentrations for each element in the roots, highlighting the transitions from un-amended to 5% amended sample and un-amended to 10% amended sample under waste water irrigation conditions.

5. Conclusion

In conclusion, this study encapsulates the diverse impacts of citrus peel biochar on heavy metal toxicity. The consistent trends observed across different parts of the plant highlight biochar's potential as an effective tool in mitigating heavy metal accumulation. These outcomes show the biochar effect across various parts of the plant, reaffirming the significance of biochar in sustainable agricultural practices.

Conflict of Interest

There is no conflict of interest.

Funding

No.

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