

RESEARCH ARTICLE

EFFECT OF MORINGA SEED POD POWDER BIOSORBENT ON THE BIOSORPTION OF Pb AND Cu AS INFLUENCED BY pH, CONTACT TIME, DOSAGE, AND CONCENTRATION OF THE EXTRACTION SOLUTION

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ABSTRACT

The aim of this study was to investigate the effect of Moringa Seed Pod Powder (MSPP) as a biosorbent on the biosorption of Pb^{2+} and Cu^{2+} influenced by pH, contact duration, dosage, and extracting solution concentration. Soil samples were collected using a soil auger at depths ranging from 0 to 20 cm using a systematic (grid) sampling procedure. Moringa seed pod samples were air-dried in the shade, crushed with a stainless steel mortar and pestle, regrind with an electric grinder, and sieved (2-mm). The influence of pH on metal adsorption capacity was divided into three phases: slightly acidic, neutral, and alkaline. The adsorption of Cu^{2+} and Pb^{2+} peaked in alkaline conditions, with values of 85.71% and 99.13%, respectively, and was lowest in neutral soil pH for both metals. The adsorption of Pb^{2+} gradually increases as the dose and shaking time change from 2 g for 2 h (97.20%) to 4 g for 4 h (98.81%), where it reaches a maximum, and then slightly drops as the dose and shaking time increase to 6 g and 6 h (98.14%). While Cu^{2+} exhibits a substantial drop in adsorption from 89.09% at (2 g 2 h) to 84.65% at (4 g 4 h), it rapidly increases to 89.99% (6 g 6 h), which represents the maximum adsorption. It is recommended to use moringa seed pod powder for the removal or remediation of Pb^{2+} and Cu^{2+} polluted soils.

KEYWORDS

Bisorption, MSPP, pH, concentration, time

1. INTRODUCTION

Heavy metals are involved in many industrial, urban, and agricultural operations and can be hazardous to individuals, plants, and animals (Alengebawy et al., 2021). Understanding the mechanisms of metal sorption in soil and biomass is critical because these processes govern the strength of metal- soil or metal- biosorbent interactions (Abiodun et al., 2023). Heavy metal intake by plants results in sequential buildup in tissues, as well as subsequent accumulation in human and animal tissues along the food chain (Tefera et al., 2021). Heavy metals are regarded as a significant contributor to soil contamination (Dawaki, 2021). Various metals, including Cu, Ni, Cd, Zn, Cr, and Pb, contribute to soil heavy metal pollution (Yadav et al., 2015). Heavy metal sources in the environment can be both natural (geogenic or lithogenic) and anthropogenic. Commercially manufactured inorganic fertilizers, particularly phosphate fertilizers, contribute to global heavy metal transport (Zafarzadeh et al., 2018). Heavy metals applied to agricultural soils via inorganic fertilizers may leak into and pollute groundwater (Rashid et al., 2023). Considerable heavy metal concentrations have been found in soils and plants along roadsides in urban and metropolitan regions (Tian et al., 2013). For example, hundreds of fatalities from lead poisoning were documented in Zamfara state in 2010 (Adelaja et al., 2011). Although some heavy metals serve essential functions in biological systems, they are often harmful to living organisms, depending on the amount and period of exposure. In toxicology, it is commonly understood that "excess of everything is bad." Even in low quantities, non-essential heavy metals (Cd, Pb, Cu, and Hg) and metalloids (As) can be harmful (Ahmed, 2013). In terms of their presence in plants (for example, moringa) as an issue for the public, arsenic ranks first, followed by Cd and Pb, and human intake of Cd and Pb has been discovered to be highest through the consumption of heavy metal-contaminated food

(Alengebawy et al., 2021). The purpose of this study was to examine the effect of pH, contact duration, dose, and concentration of the extracting solution on the biosorption of Pb and Cu using moringa seed pod powder (MSPP) as the biosorbent.

2. MATERIALS AND METHODS

2.1 Study Area

The study was carried out at Sabon Garin 'Ya'ya in the Taura local government area of Jigawa State, Nigeria, between latitude $10^{\circ} 2' 2''N$ and longitude $9^{\circ} 2' 59''E$ in Nigeria. The region has two different seasons (wet and dry). The wet season mostly lasts for 3 – 4 months, usually from May's end to September and early October. The dry season is also divided into two periods. The first is the Hammatan, which begins at the end of the wet season and is distinguished by haze, dust, wind, and severe cold. The second phase is the hot season, which begins around April and ends when rain begins.

2.2 Soil Sampling Technique

A systematic (grid) sampling technique was applied to collect samples from a randomly selected 4500 m² study field at 22 m intervals. Using a soil auger, twenty soil samples were collected and bulked at depths of 0 to 20 cm. The composite sample was air-dried in the shade and sieved using a 2-mm sieve before being sent to the laboratory for analysis.

2.3 Soil Analyses

The particle size distribution was determined using the Bouyous hydrometer technique (Mustapha et al., 2021). Textural classes were

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defined using the USDA textural triangle. The core approach described was used to determine the soil bulk density by (Nabayi et al., 2019). CaCl₂ was used to determine the soil pH using 1:2 modified soil solutions. A laboratory pH meter (glass electrode) was used to test pH. After shaking the soil in 1 M KCl and titrating it against 0.5 M NaOH, the exchangeable acidity was measured. The organic carbon content was determined using the Walkley Black technique defined using K₂Cr₂O₇ and sulfuric acid (wet oxidation) by (Mustapha et al., 2023). The total nitrogen concentration of the prepared samples was determined using the Macro-Kjeldahl technique after digestion, distillation, and titration. The available phosphorus was extracted using a procedure reported by Mustapha et al. (2021) and measured using a spectrophotometry (22PC MODEL at 860nm). As Mustapha (2021) reported, exchangeable bases (Ca, Mg, K, and Na) in soil were extracted using a 1 N ammonium acetate (1N NH₄OAc) solution buffered at pH 7.0. A flame emission spectrophotometer (FES) was used to determine the levels of Na and K. Ca and Mg were determined using an atomic absorption spectrophotometer (AAS). Micronutrients (Mn, Mo, Cu, and Zn) were extracted with 0.1 M HCl and quantified by AAS using a spectroscopic approach.

2.4 Biosorbent Sampling and Preparation

Moringa seed pod samples were collected in Jigawa State, Nigeria, from the Sabon-garin 'ya'ya Taura local government. The pods were air-dried in the shade before being pulverized using a stainless steel mortar and pestle, regrinding with an electric grinder, and sieving (2-mm). For the treatment of the biosorbent, the approach described was adapted and used by (Adelaja et al., 2011). The pH measurements were performed in the soil science laboratory of Bayero University Kano's Faculty of Agriculture using a pH meter (glass electrode). A mechanical shaker was used to agitate the aqueous solution at the soil science laboratory of the Faculty of Agriculture. The biosorbent was separated from the aqueous solution using filter sheets. They were ground with a steelless mortar and pestle, regrinding with a laboratory grinder, and sieving with a 2-mm laboratory sieve. According, provided a technique that was modified and used to treat the biosorbent (Adelaja et al., 2011).

2.5 Biosorption Experiment

The significance of pH on the biosorption of heavy metal ions such as Pb²⁺ and Cu²⁺ was examined by placing 2 g of adsorbent in a 120 ml plastic bottle containing 50 ml of 50 ppm lead and copper solutions. The pH was adjusted during the investigation using 0.05 M NaOH and 0.1 M HCl. The pH range of interest was 5.5–9.5, and the solution was agitated for 1 h using a mechanical shaker. The solution was filtered using Whatman filter paper after being agitated. Before analysis, the solution was refrigerated. Adsorbent (2 g) was measured in a 120-ml plastic bottle containing 50 ml of a well-known solution (lead or copper) with an initial concentration of 50 ppm at pH 7. The pH was adjusted using 0.05 M NaOH and 0.1 M HCl. The contact time will be between two and six hours. The adsorbent dose ranged from 2 to 6 g, depending on the amount of adsorbent used. The agitation period was 3 h, and the pH of the aqueous solution was maintained at 7 (neutral). Concentrations ranging from 10 to 50 ppm were used in the pH study. The percentage (%) adsorbent was determined using the following equation:

$$\% \text{ adsorbent} = \frac{C_0 - C_a}{C_0} \times 100$$

Where C₀ = initial concentration of solution

Ca = concentration of the solution after adsorption.

The metallic ion concentration residue from the experiment was determined by atomic absorption spectroscopy using an atomic absorption spectrophotometer.

2.6 Statistical Analysis

Data obtained from the study were subjected to descriptive statistics using the statistical functions of Microsoft Excel version 2019.

3. RESULTS AND DISCUSSION

3.1 Particle Size Distribution

The particle size distribution results reveal that sand has the highest percentage (88.96%), clay particles (6.56%), and silts have the lowest percentage fraction (4.48%). According to USDA and UK-ADAS textural triangles, the soil in the research location belonged to the loam sand textural class. This research concurred with who also reported selected physical and chemical properties of soils in Dutse, Jigawa state, Nigeria, under varied tillage approaches (Nabayi et al., 2019).

3.1.1 Soil pH and EC

The pH values in water and calcium chloride were 5.97 and 4.85, respectively, whereas the EC was 0.0322 μs, Table 1. According to the findings, the soil in the research area is moderately acidic and non-saline. This was supported by who also reported certain selected physical and chemical properties of soils at Dutse, Jigawa state, Nigeria, under different tillage approaches (Nabayi et al., 2019). This is also reiterated by the findings of (Onokebhabge et al., 2021).

Parameters	Mean
Sand (%)	88.96
Clay (%)	6.56
Silt (%)	4.48
pH in H ₂ O	5.97
pH in CaCl ₂	4.85
OC (%)	0.13
TN (%)	0.39
A.P (Cmol kg ⁻¹).	17.33
Ca (Cmol kg ⁻¹).	0.968
Na (Cmol kg ⁻¹).	0.277
Mg (Cmol kg ⁻¹).	0.712
K (Cmol kg ⁻¹).	0.556
EA (Cmol kg ⁻¹).	0.5
EC (μs)	0.0322
Fe (mg kg ⁻¹)	9.6201
Cu (mg kg ⁻¹)	9.8838
Zn (mg kg ⁻¹)	0.5552
Mn (mg kg ⁻¹)	12.9797

Key: EA = exchangeable acidity, EC = electrical conductivity, OC = organic carbon, AP = available phosphorus, TN = total nitrogen.

3.1.2 Organic Carbon

The organic carbon content of soil in the study area is (0.13%). This is due to the scarcity of sparse trees and short grasses, and the fertility status of the soil in the area in question is very low. This agrees with the findings of who reported the spatial variability of soil fertility in the Nigerian Sudan savanna (Shehu et al., 2015).

3.1.3 Total Nitrogen

The result shows that the nitrogen content in the study area is (0.39%). The total nitrogen content in the study area was very high because of the growth of legumes in the previous growing season. The results agreed with those reported who investigated selected physical and chemical attributes of a Ustalf under different tillage practices at Dutse, Jigawa state, Nigeria by (Nabayi et al., 2019).

3.1.4 Available Phosphorus

The content of available phosphorus in the sampled soil was 17.33 Cmolkg⁻¹. The sampled soil has very high available phosphorus. This agrees with who reported the spatial variability of soil fertility in the Nigerian Sudan savanna: Evidence from a university research farm at an intensive sampling scale (Nabayi et al., 2019).

3.1.5 Exchangeable Bases

The results show that the potassium content is very high with a calculated mean (0.556 Cmolkg⁻¹). The calcium content is (0.968 Cmolkg⁻¹). Magnesium content is (0.712 Cmolkg⁻¹) and Sodium content is (0.277 Cmolkg⁻¹). The results showed that calcium is low, whereas magnesium and sodium are moderate according to (Dawaki et al., 2018).

3.1.6 Micronutrients

The results of micronutrient analysis delineated that iron (9.62 mg kg⁻¹), copper (9.88 mg kg⁻¹) and manganese read (12.98 mg kg⁻¹). The results show that the sampled soil has high iron, copper, and manganese contents. The high iron values are attributed to the acidity of the soil (Mustapha, 2021). The zinc content in the area studied was moderate. This agrees with the findings of who reported on soil nutrient status under *Acacia Senegal* wild plantation in the Sahel zone of Jigawa state, Nigeria (Ampitan et al., 2022).

3.2 Effect of pH on the Biosorption of Cu²⁺

The effect of pH on the adsorption capacity of Cu²⁺, as depicted in (figure 1) was divided into 3 phases, with the first phase being slightly acidic. The

adsorption capacity at pH (5.5) was 84.62%, and at pH 6.5, the adsorption capacity declined to 83.69%. The second phase is the neutral state, i.e., pH 7.0, where the adsorption capacity sharply declined to 77.65%. The third phase was the alkaline condition where the adsorption capacity gradually increased at pH (8.5) to 82.12% and maximally increased to reach 85.71% at pH (9.5). The result indicated that the slight decrease in the adsorption

capacity of Cu^{2+} was due to a decrease in H^+ and the sharp increase at pH (9.5) was due to the accumulation of hydroxyl ions (OH^-). This agrees with the findings on the biosorption of Pb^{2+} from aqueous solution using Rooibos shoot powder (RSP) of (Sheku et al., 2016). This result is also in agreement with the report of on the effect of organic acids and inorganic ions on lead desorption from soils (Xiao et al., 2022).

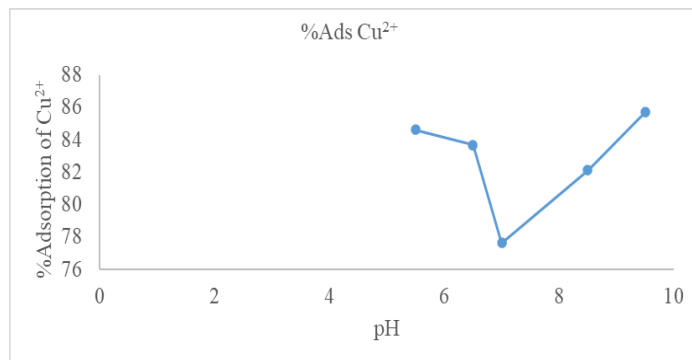


Figure 1: Effect of pH on the Biosorption of Cu^{2+}

This study is also supported by the research of who reported the biosorption of heavy metals from aqueous solutions by *Saccharomyces cerevisiae* (Farhan and Khadom, 2015). As a reported the removal of cadmium from aqueous solutions using chitin (Danial and Dardir, 2023). This result was also supported who investigated the effect of pH on the biosorption of chromium and other heavy metals by *Pseudomonas fluorescens* 4F39 by (Silva et al., 2009).

3.2.1 Effect of pH on the Biosorption of Pb^{2+}

The adsorption capacity of Pb^{2+} under different pH values of the extracting solution is shown in Figure 2. The adsorption capacity of Pb^{2+} at pH (5.5) was observed to be 97.84%, which slightly declined to 97.58% at pH (6.5),

and gradually decreased to 96.55% at pH (7.0). However, the adsorption capacity rises and reaches a maximum at pH 8.7 (99.13%) and finally declines at pH 9.5 (96.15%. This result indicates that the reduction in the adsorption of Pb^{2+} could be attributed to an increase in the concentration of hydroxyl ions (OH^-). This result is consistent with the findings of who investigated the biosorption of $\text{Pb}(\text{ii})$ ions from aqueous solution using *Moringa oleifera* pods (Kanu et al., 2016). This is also in agreement with the findings of who studied the biosorption of Pb^{2+} from aqueous solution using Rooibos shoot powder (RSP) (Kanu et al., 2016). It was also accepted who reported the removal of cadmium from aqueous solution using chitin by (Benguella and Benaissa, 2002). This result is also in line with the findings of on the biosorption of heavy metals (Abdelfattah et al., 2016).

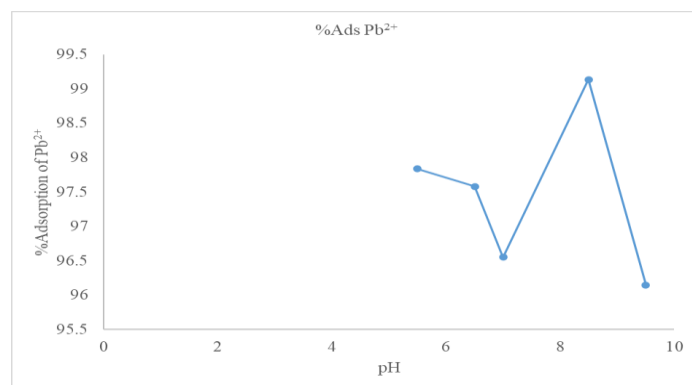


Figure 2: Effect of pH on the biosorption of Pb^{2+}

3.2.2 Effect of The Initial Concentration of The Extracting Solution on The Biosorption of Pb^{2+}

Figure 3 delineates the adsorption capacity of Pb^{2+} at various concentrations (10, 20, 30, 40 & 50 ppm) of the extracting solution. The concentration of 10 ppm with (98.57%) is very high, followed by 40 ppm with (97.92%); however, 30 ppm with (94.19%) has the lowest adsorptive capacity of Pb^{2+} . The results show that 20 ppm and 50 ppm (96.86% &

97.19%) have moderate adsorptive capacitance. The observed result depicted that Pb^{2+} adsorption capacity is very high at 10 ppm. This result agrees with the study of Ahmed et al. (2010), who studied the effect of adsorbate initial concentration on the removal of Pb from aqueous solution using carbon nanofibers. This is also supported by the findings of Ahmed et al. (2010), who investigated the effect of adsorbate initial concentration on the removal of Pb from aqueous solution using carbon nanofibers.

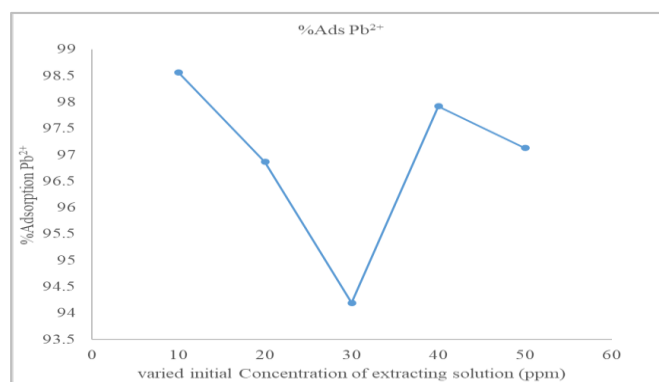


Figure 3: Effect of the Initial Concentration of the Extracting Solution on the Biosorption of Pb^{2+}

3.2.3 Effect of the Initial Extracting Solution on the Biosorption of Cu²⁺

As shown in Figure 4, the effect of the initial concentration on the adsorption capacity of Cu²⁺ was observed. An increase in the concentration of Cu²⁺ in the solution from 10 ppm to 20 ppm led to an increase in the adsorption capacity of Cu²⁺ from (78.58% to 87.54%). Furthermore, a slight decline in adsorption was observed as the concentration increased to 30, 40 and 50 ppm (86.14%, 86.21% & 86.52%). The results agreed with those of who studied the biosorption of Pb²⁺ ions from aqueous solution using *Moringa oleifera* pods (Adelaja et al., 2011). This is also in line with who studied the biosorption of Pb²⁺ from aqueous solution using Rooibos shoot powder (RSP). This is also in agreement with the findings of who investigated the effect of organic acids and inorganic ions on lead desorption from soils (xiao et al., 2022).

3.2.4 Effect of contact time and dosage on the biosorption of Pb²⁺

Figure 5 depicts the effect of biosorbent dosage and shaking time on the adsorption capacity of Pb²⁺. The result shows that there is a gradual increase in the adsorptive behavior of Pb²⁺ as the dose and shaking time change from 2 g for 2 h (97.20%) to 4 g for 4 h (98.81%), where it reaches maximum, and then adsorption slightly declines as the dose and shaking

time increase to 6 g and 6 h (98.14%). This means that there is maximum adsorption at 4 g 4 h (98.81%). This result was in agreement with the findings of who investigated the biosorption of iron by amended *Aspergillus versicolor* from polluted water sources (Hassouna et al., 2018). This was also in line with who used Sour Sop seeds (Sheku et al., 2015). This is also in line with who worked with Rooibos shoot powder (Sheku et al., 2015). These assertions are based on the Freundlich adsorption model or the Freundlich isotherm. This is also in agreement with who reported the biosorption of heavy metals from aqueous solutions by *Saccharomyces cerevisiae*.

3.2.5 Effect of Contact Time and Dosage on The Biosorption of Cu²⁺

As shown in Figure 6, the effect of contact time and dosage on the adsorption capacity of Cu²⁺, and the result depicted that there is a sharp decrease in the adsorption of Cu²⁺ from 89.09% at (2 g 2 h) to 84.65% at (4 g 4 h) and sharply increases to 89.99% (6 g 6 h), which shows the maximum adsorption. This means that biosorbent dosage has a direct influence on the adsorption capacity of Cu²⁺. This result corroborates the findings of who studied the biosorption of Pb (ii) ions from aqueous solution using *Moringa oleiferapods* (Adelaja et al., 2011). This is also in agreement with Sheku et al. (2015), who reported on Rooibos shoot powder.

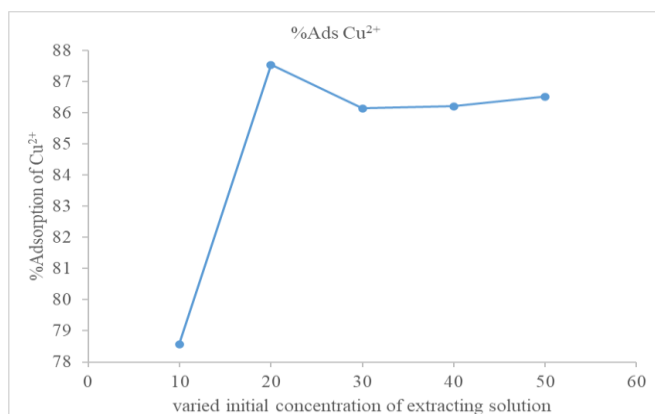


Figure 4: Effect of the Initial Extracting Solution on the Biosorption of Cu²⁺

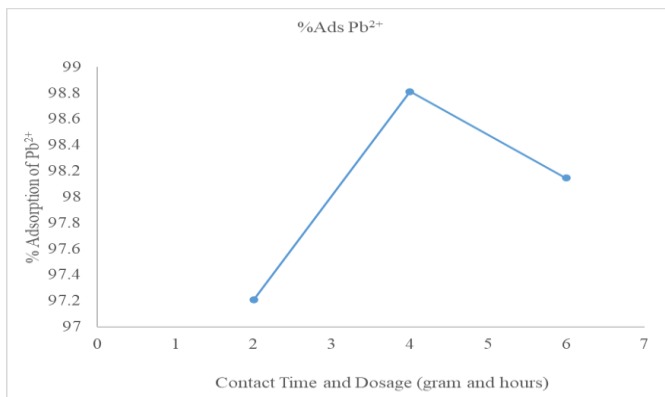


Figure 5: Effect of contact time and dosage on biosorption of Pb²⁺

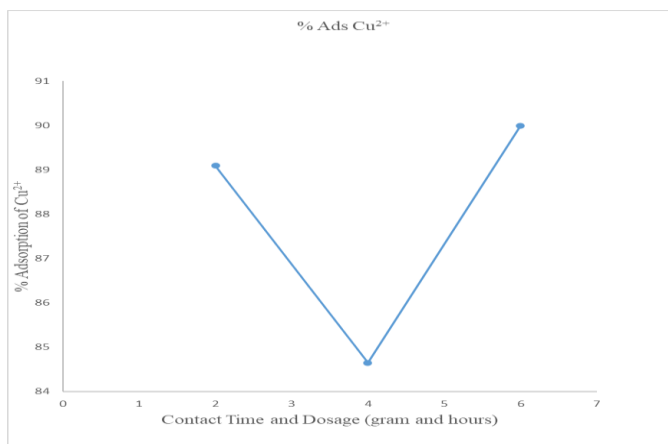


Figure 6: Effect of contact time and dosage on biosorption of Cu²⁺

4. CONCLUSION

In conclusion, for the pH studies, the result delineated that hydroxyl ions (OH⁻) has the significant effect on the adsorption of Pb²⁺ and Cu²⁺ as both metals were adsorbed more (99.13% & 85.71%) in alkaline media when Moringa Seed Pod powder was used as an amendment. However, a low initial concentration of the extracting solution is effective for the adsorption of Pb²⁺ and Cu²⁺ in aqueous solution. Dosage of the biosorbent and shaking time have a significant influence on the adsorptive capacity of Pb²⁺ and Cu²⁺, but the effect is more obvious in Cu²⁺. Finally, it is obvious that Moringa seed pod powder is very effective for the removal of Pb²⁺ and Cu²⁺ in alkaline soils, as the percentage adsorption of both metals was found to be higher than that of other biosorbents such as Rooibos shoot powder.

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