Remediation of Soils Contaminated with Cadmium by In-Situ Immobilization Process Using Animal Bones

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ABSTRACT

To immobilize lead, chromium, and cadmium in clay, sandy, and laterite soils, researchers looked at using animal bones (cow, pig, and horse) as a material. After being heated to 700 °C for four hours, the bone byproducts were carbonized. Three types of animal bone ash were used for analysis: cow bone ash (CBA), pig bone ash (PBA), and horse bone ash (HBA). SEM was the tool used for this process. Additionally, physicochemical analyses were carried out on the soil samples, which included clay, sandy, and lateritic soil types. The physicochemical analysis of the additives revealed that calcium phosphate is their primary component. The scanning electron microscopy (SEM) analysis of the amendments showed their high pore density and the specific atomic weights of the carbon, phosphorus, and calcium ions. The order of increasing calcium and phosphorus concentration is CBA>PBA>HBA. The XRF analysis showed that the percentage of CaO and P2O5 in CBA, PBA, and HBA, respectively, is 49.25%, 46.64%, and 33.857 percent. The presence of several functional groups, such as aromatic phosphates and ethylene, which immobilize and stabilize metals in soil, was indicated by the FTIR analysis. Results from a batch immobilization experiment showed that CBA was the most effective sorbent for removing Pb, Cr, and Cd from lateritic, clay, and sandy soils. Lateritic soil removed 74.55% of Pb from 100 g of soil, followed by clay soil at 71.2 % and sandy soil at 63.3 %.

Keywords: Remediation, immobilization, animal bones, soils, cadmium, in-situ

INTRODUCTION

The atomic number of cadmium is 48, its atomic weight is 112.4 g, its density is 865 g/cm^3, and its melting and boiling points are 320.9 and 765 degrees Celsius, as befits a transition element. As one of the three major heavy metals that aren't absolutely necessary, it is of great concern. Histologically, it is a basic oxide. Group 2B, period IV of the periodic table is where it belongs. Along with an electro negativity of 1.69, its first ionization potential is 8.993 V. Located just below zinc on the periodic chart, it shares many chemical properties with zinc, a micronutrient vital to plant and animal life. Because of their physical and chemical similarities, zinc and cadmium are suitable metals to swap for one another. As zinc is a vital micronutrient, the metabolic pathway may become dysfunctional when cadmium is used in place of it, which could explain cadmium's toxicity [1]. Despite cadmium's utility in making cadmium-nickel rechargeable batteries, plastic stabilizers, and corrosion protection coatings for iron and steel, it is the most hazardous element to which a man may be exposed in the job or the environment [2]. [3 4] state that cadmium can hurt the kidneys if it builds up over time. Cadmium is known to induce hypertension, lung cancer, and a host of other disease in humans [5].

For soils polluted with cadmium metals, several solutions have been elaborated. Some examples of these are: Chemical leaching and flushing of soil either at an off-site location or at the site of excavation, Electro kinetic migration, Solidification/stabilization, In order to mitigate soil contamination, several methods have been proposed, including dilution, encapsulation, dig-and-dump, vitrification, immobilization and phyto-remediation [6 7]. Soil remediation using immobilization technology involves combining organic and inorganic materials to hasten the slowdown of metal mobility and toxicity. Through sorption, precipitation, and

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composition processes, the primary goal of immobilization is to change the initial soil metal concentration of more stable phases.

Benefits of in-situ immobilizations include low environmental invariability; low energy and labor needs; a simple and quick process; less waste production; and high public acceptance. Specific soil properties, such as organic matter concentration, grain size distribution, and pH, greatly influence the mobility of heavy metals in soil. Due to the solubility and mobility of heavy metals in acidic environments, acidic soils promote the spread of contaminants, whereas alkaline soils tend to accumulate them [8].

Heavy metals immobilization using binders like calcium carbonate, Portland cement, and phosphate rock has been documented, however their usefulness is constrained due to their inherent challenges throughout the course of the process. Therefore, alternative binders that are inexpensive, user-friendly, and easily accessible should be sought out for heavy metal cleanup.

Animal bones, which are a common source of trash and pollution, can be used to remove heavy metals from soil.

A lack of research on animal bones as additions to immobilize cadmium from soils is a present limitation, though. For that reason, the purpose of this research is to examine how well and how efficiently animal bones, a byproduct of the meat industry, may be used to immobilize cadmium metal in soils that have been contaminated.

MATERIALS AND METHODS

Cow bone, Horse bone, Pig bone, Lateritic soil. Other materials used include: distilled water, sodium hydroxide (NaOH), Hydrochloric acid, Lead nitrate ($PbNO_3$), Cadmium chloride ($CdCl_2.H_2O$), Chromium Nitrate ($CrNO_3$), Magnesium chloride ($MgCl_2$), sodium ethanoate (CH_2COON_a), DTPA- Diethylene-tri-amine pent acetic acid, Triethanolamine (TEA), Potassium Dichromate (K_2Cr_207), Ferrous Ammonium sulphate ($(Fe(NH_4)_2$ (Sp): $6H_2O$ phosphoric acid (H_3P0_4), Sodium fluoride (NaF), Diphenyl amine ($C_6H_5NH_6H_5$). Atomic Absorption photo spectrophotometer (AAS), Model; AA 430 N, Scanning Electron microscope (SEM), (Perk Elma 2400 elemental analysis.

Collection of Soil Samples

Soil samples were taken from three distinct sites in Enugu State, Nigeria, ranging in depth from zero to twenty centimeters on three separate occasions. Soil samples were taken from Amagunze that included clay. In the Nkanu East Local Government Area of Enugu State, Nigeria, at coordinates 6.382541 and 7.486532. A lateritic soil sample was taken from the Emene site. Located in the Enugu East LGA of Enugu State at coordinates 6.488203 and 7.57282).

The Obollo-Afor location in Enugu State's Udenu Local Government Area was examined for the collection of sandy soil at coordinates 6.920592475 and 752099387.

Collection of the Bone Samples (amendments).

The three immobilizing agents were collected as follows

Cow and pig bone were collected at Oye, Emene Central Abattoir in Enugu East L.G.A while horse bone was collected at the Abattoir of the Obollo -Afor main market in Udenu L.G.A. of Enugu State.

Preparation of the Amendments.

The technique outlined by Abdulrahman et al. (2015) was used to prepare the bone samples. After being thoroughly rinsed with water, the bones were sliced into pieces using a cutlass. They were then washed again and again to eliminate any surface contaminants. After rinsing with de-ionized water, the bones were placed in an oven set to 80 degrees Celsius to dry. After being ground with a mechanized crusher, the dried bones were moved to a furnace and left to burn for five (5) hours at 700 ^oC.

Chemical Analysis of the Bone Samples

The samples were characterized with SEM, and AAS to determine functional groups, morphology, mineral composition and heavy metal content respectively.

Determination of Surface Morphological using SEM

The surface morphology of the immobilizing agents (CBA, HBA and PBA) was studied using model 302 Hitachi high Field emission scanning, Electron Microscope and the images at Umaru Musa Yaradua University Katsina state. Nigeria.

Determination of Metal Content using AAS

The total soil & bone heavy metal contents were determined by the method of aqua regia extraction as described by [9]. The analysis was performed with an Atomic Absorption photo spectrometer (AAS) model AA340N.

Physicochemical Analysis of the Soil Samples

Classification of the Soil Sample

The pH of the water was assessed utilizing the standard testing method specified in ASTMD1293-95 while the electrical conductivity was measured with an EC meter. Orion Star A212 by Thermo Fisher Scientific Soil Organic Carbon was quantified using the wet oxidation method established by [10]. The CEC was ascertained following the methodology outlined by [11]. The soil's phosphate concentration was assessed using [12]. The Micro Kjeldahl method, as outlined by [13] was employed to ascertain total nitrogen content. The potassium concentration in the soil was determined using the Jenway PFP7 Flame Photometer [14]. The concentration of iron oxide in the soil sample was assessed using the methodology outlined by [13] .77 grams of ammonium acetate were dissolved in 760 milliliters of water and the pH was corrected to 7 using acetic acid or ammonium hydroxide, diluted to a total volume of 1 liter. The A.O.A.C. technique [13] was employed to ascertain moisture content.

Spiking of the Soil with the Heavy Metals

In accordance with the procedure detailed by [15]. The soils were spiked. A wood plate and grinding rod were used to grind 1 kilogram of soil and 3 grams of additives. A 2 mm sieve is used to filter the material. Sodium nitrate, cadmium chloride, and lead nitrate were added to the bulk of the metal species. After that, the entire thing was shook vigorously in a plastic bag. This time, we used 5 grams of the adjustments in our experiment..

Incubation Experiment

[16, 17, 18] detailed the procedure for incubating the soil samples and amendments. In a 500 ml container, 100g of air-dried polluted soils were combined with different additives at a specified rate of application on a dry basis. Each of the three soils was amended with three different types of bone ash: cow, horse, and pig. 25% NaNO3 was added to the mixture as an electrolyte background. Using a pipette, deionized water was added to the soils until their water holding capacity reached 65%. Deionized water was continuously introduced to the experiment to make up for the water that was lost. For the sake of realism, the containers were set out in open air. At2,7,14,30,60,150, and 300 days into the incubation process, we collected samples. During the incubation process, the soil pots were mixed well. The following is a rundown of the percentages of immobilized cells derived from the DTPA-Extractable fraction at each time:

$$\%\, Immobilized = \left(\frac{CO - Ct}{Co}\right) 100 \qquad (1)$$

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Where Co- Initial metal conc. in mg/kg

Ct = concentration of metal at time t (mg/kg).

DTPA (Diethylene-triamine-penta-acetic acid) was used to get the heavy metals out of the soil, following the steps outlined by Lindsay and Norwell in 1978 and updated by ISO 14870 in 2001.

To make the DTPA-TEA solution, 0.01% CaCl2, 0.005% DTPA, and 0.1 molL-1 Tri-ethanolamine (TEA) triethanolamine were mixed together until the pH was 7.3.1mol.The pH was changed with an HCl solution (L-1). Ten grams of the 0.15-millimeter-thick dirt samples were put into a 500ml container. 20 ml of DTPA-TEA extracting solution was put into an Erlenmeyer flask, and it was mixed for two hours. The mixture of dirt was spun at 2000 rpm for 15 minutes and then left to sit for an hour. It took 50 ml of pure water to thin out the clear supernatant (Noha et al., 2013). An Atomic Absorption Spectrometer (AAS) type AA340N was used to find out how much DTPA could be extracted.

RESULTS AND DISCUSSIONS

Chemical Properties of the Bone Samples.

Selected properties of the animal bones used for the immobilization study are shown in table 1.

Table 1: Selected Chemical Properties of the bone samples

Parameter			**Bone samples**	
	Units	**CBA**	**HBA**	**PBA**
Total carbon (TC)	%	17.80	16.76	20.92
Phosphate (P_2O5)	%	40.49	33.87	33.85
Cadmium (Cd)	mg/kg	0.01	0.02	0.03
Chromium (Cr)	mg/kg	0.001	0.001	0.02
Lead (Pb)	mg/kg	0.05	0.07	0.06
Calcium (Ca)	%	32.05	30.32	32.25

The bone samples' total carbon (TC) levels vary from 16.76% to 20.92%, with pig bone having the highest TC level. As per the research conducted by [19]. on the subject of activated cow bone powder and its effectiveness in removing cadmium from palm oil, the carbon surface has an effect on sorption, while the hydro apatite arrangement provides sites for the attraction of cadmium ions. According to Table 4.2, all three bone samples had high Total carbon levels, suggesting they work well as immobilization agents. The bone samples show elevated calcium and phosphate percentages; the pig bone has the greatest concentration, the horse bone the lowest, and the cow bone the highest.

According to the results of the bone sample analysis, it belongs to the same family as hydroxyapatite (Ca5 (PO4)3 OH (HA), the most prominent member of a vast class of substituted compounds with a comparable structure [20]. Solubility, ion exchange, and precipitation make up the fundamental process. The presence of soluble metals and phosphorus is necessary for the creation of pyromorphite, an insoluble compound [21]. The immobilization sequence for all metals is CBA>PBA>HBA because phosphate and calcium levels in cow bone are higher than in pig and horse bone.

The results for the concentrations of Pb, Cr, and Cd, as shown in table 1, were all within the acceptable range according to the guidelines set by the WHO/FAO and the European Union. The lead concentrations in the bone samples are between 0.05 and 0.07 mg/kg. Relative to the 0.3 mg/kg standards set by the WHO/FAO and the EU, this is significantly lower.

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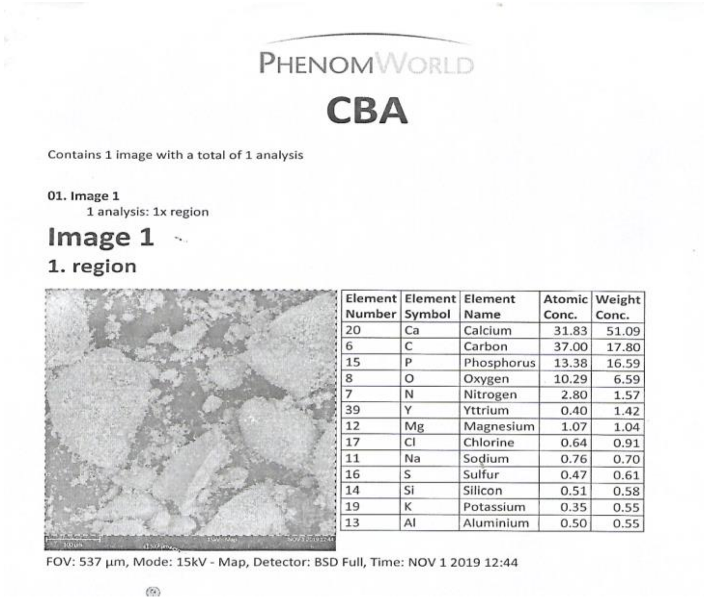
Similarly, the bone samples contain chromium (Cr) in the range of 0.001 to 0.002 mg/kg. With 1.3 mg/kg as the World Health Organization/Food and Agriculture Organization's norm and 0.3 mg/kg as the European Union's, this is absolutely negligible. From 0.01% to 0.3 mg/kg of Cd is present. This is lower than the 0.2 mg/kg threshold set by the World Health Organization and the European Union [22].



PHENOMWORLD

CBA

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01. Image 1
1 analysis: 1x region

Image 1

1. region

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
20	Ca	Calcium	31.83	51.09
6	C	Carbon	37.00	17.80
15	P	Phosphorus	13.38	16.59
8	O	Oxygen	10.29	6.59
7	N	Nitrogen	2.80	1.57
39	Y	Yttrium	0.40	1.42
12	Mg	Magnesium	1.07	1.04
17	Cl	Chlorine	0.64	0.91
11	Na	Sodium	0.76	0.70
16	S	Sulfur	0.47	0.61
14	Si	Silicon	0.51	0.58
19	K	Potassium	0.35	0.55
13	Al	Aluminium	0.50	0.55

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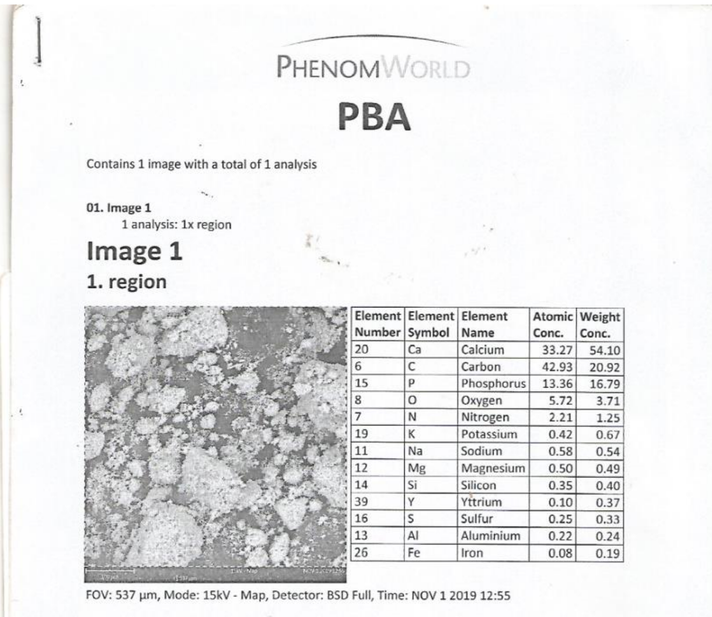
Fig 1: SEM Image of CBA



PHENOMWORLD

PBA

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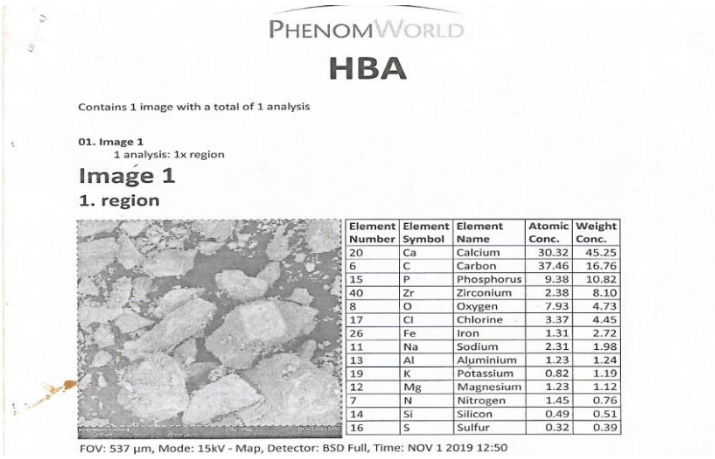
1. region

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
20	Ca	Calcium	33.27	54.10
6	C	Carbon	42.93	20.92
15	P	Phosphorus	13.36	16.79
8	O	Oxygen	5.72	3.71
7	N	Nitrogen	2.21	1.25
19	K	Potassium	0.42	0.67
11	Na	Sodium	0.58	0.54
12	Mg	Magnesium	0.50	0.49
14	Si	Silicon	0.35	0.40
39	Y	Yttrium	0.10	0.37
16	S	Sulfur	0.25	0.33
13	Al	Aluminium	0.22	0.24
26	Fe	Iron	0.08	0.19

FOV: 537 µm, Mode: 15kV - Map, Detector: BSD Full, Time: NOV 1 2019 12:55

Fig 2: SEM Image of PBA



PHENOMWORLD

HBA

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01. Image 1
1 analysis: 1x region

Image 1

1. region

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
20	Ca	Calcium	30.32	45.25
6	C	Carbon	37.46	16.76
15	P	Phosphorus	9.38	10.82
40	Zr	Zirconium	2.38	8.10
8	O	Oxygen	7.93	4.73
17	Cl	Chlorine	3.37	4.45
26	Fe	Iron	1.31	2.72
11	Na	Sodium	2.31	1.98
13	Al	Aluminium	1.23	1.24
19	K	Potassium	0.82	1.19
12	Mg	Magnesium	1.23	1.12
7	N	Nitrogen	1.45	0.76
14	Si	Silicon	0.49	0.51
16	S	Sulfur	0.32	0.39

FOV: 537 µm, Mode: 15kV - Map, Detector: BSD Full, Time: NOV 1 2019 12:50

Fig 3: SEM Image of HBA

SEM Analysis Result

The analysis of the morphological constituents of the modifications was conducted using SEM Analysis. Every one of the changes had a huge amount of pore spaces. Since adsorption is a surface phenomenon, it is facilitated when there are available pore spaces. On top of Figures 1-3 display the morphologies of the precepts, which include cow bone ash (CBA), pig bone ash (PBA), and horse bone ash (HBA).CBA contains 31.83% calcium and 13.38% phosphorus (Fig. 1). Figure 2 displays a picture of pig bone ash. It shows black patches with a low atomic number (42.93% carbon), a brilliant feature with a carbon concentration of 33.27%, and phosphorus content of 13.36%. However, the atomic concentration of phosphorus is 9.38%, calcium is 30.32%, and carbon is 37% in horse bone ash. Figure 3

This proves beyond a shadow of a doubt that calcium phosphate is the key ingredient in every single amendment. Research conducted by [22].996) indicates that phosphate continuing materials can effectively immobilize cadmium in enclosed soils. Since the amount of phosphorous determines the immobilization, the

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micrograph shows that cow bone ash has a higher quantity than pig bone ash, which means that CBA will have higher mobilization than pig bone ash and lowest mobilization in horse b one ash [23, 24].

Physicochemical Properties of the Soil

Some selected physicochemical properties of the soil are presented in Table 2.

Table 2: Selected physiochemical properties of the soils.

Parameter			Soils	
	Units	Clay	Sandy	Laterite
Sand	%	32.20	77.20	20.28
Silt	%	5.60	7.42	55.36
Clay	%	60.20	15.38	24.36
Textual	Class	Clay	Loamy sand	Silt loam
Ph		6.45	6.82	6.54
Organic Matter	%	31.736	4.20	2.46
EC	Ds/m	1228.40	18.66	25.36
CEC	Cmol/kg	6.22	5.02	5.61
$P_{2\ O5}$	Cmol/kg	1.12	1.45	0.98
TN	Cmol/kg	1.63	1.82	1.70
Tis	Cmol/kg	9.68	10.53	8.56
Moisture	%	42.30	21.67	12.42
Cd	mg/kg	0.02	0.01	0.03
Cr	mg/kg	0.01	0.02	0.03
Pb	mg/kg	0.30	0.53	0.11
Fe_20_3	Cmol/kg	0.65	0.34	26.23
Al_20_3	Cmol/kg	0.21	0.26	4.33

Soils that were present were clay, loamy sand, and silt loam. In terms of metal mobility in soils, soil texture is a key factor. The soil's texture reflects the clay and mineral oxide particle size distribution. [25] States that these compounds have a significant role as adsorption media.

In comparison to sandy soil, clay soil maintained a greater amount of metals. Soil adsorption capacity is directly proportional to its clay material composition. Soil pH values are between 6.45 and 6.82. As the pH rose, metal sorption rose as well. In other words, the mobility of metals increases when the pH value decreases because more metals are present in the solution. All of the soil samples had acidic pH values, which made desorption and movement easier. According to [26] heavy metal mobility in soil was highest at low pH.

In comparison to sandy and lateritic soils, the organic content of the investigated clay soil was much higher (Table 4.1). When studying metal sorption and desorption, organic matter is an important parameter (Sherene, 2010). Metals are retained by soil solids due to organic matter, which reduces their mobility and bioavailability.
Despite the fact that organic soil components aid heavy metal retention, an overabundance of these materials

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might cause complexation events that make metals more soluble [25] . Due to its significantly larger organic matter content (31.72% vs. 4.20% for sandy soil and 2.46% for lateritic soil), clay soil would be better able to immobilize than sandy soil. Metals cling to organic materials because it enhances their adsorptive surface area [27].

Each of the three soil samples had a phosphate concentration between 0.98 and 1.45 Cmol/kg. Soils with low phosphate levels are thus defined as such. In most cases, insoluble complexes can be formed by coordinating metal ions with anions like sulphate. Research conducted by [28] demonstrated that lead may be efficiently immobilized to varying degrees by using phosphate rock, namely flu apatite (Ca10¬ (PO4)6 Fe). The soils have extremely low concentrations of cadmium (Cd), chromium (Cr), and lead (Pb). These concentrations are below the regulatory levels, which are 0.48 for Cd, 11.0 for Cr, and 200 for Pb.

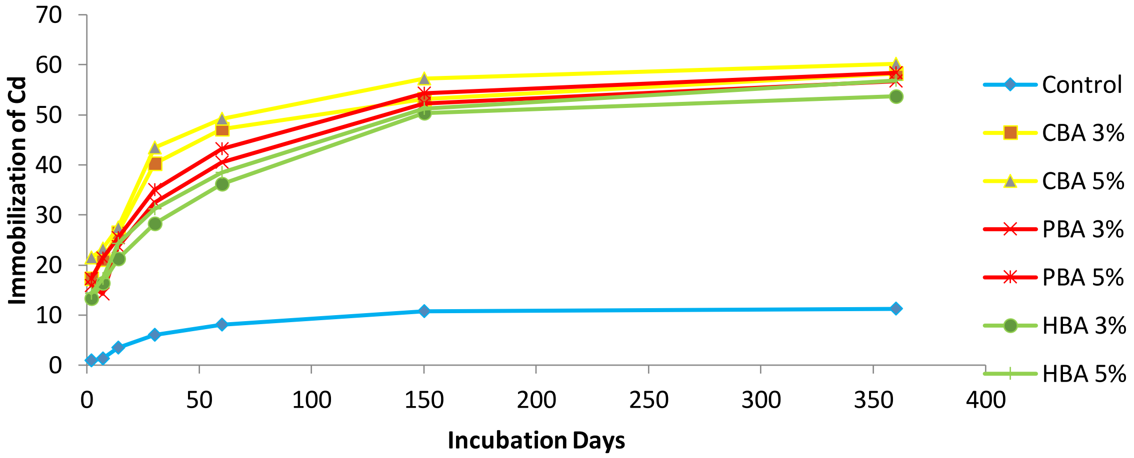

Fig 4: Immobilization of Cd in Clay Soil

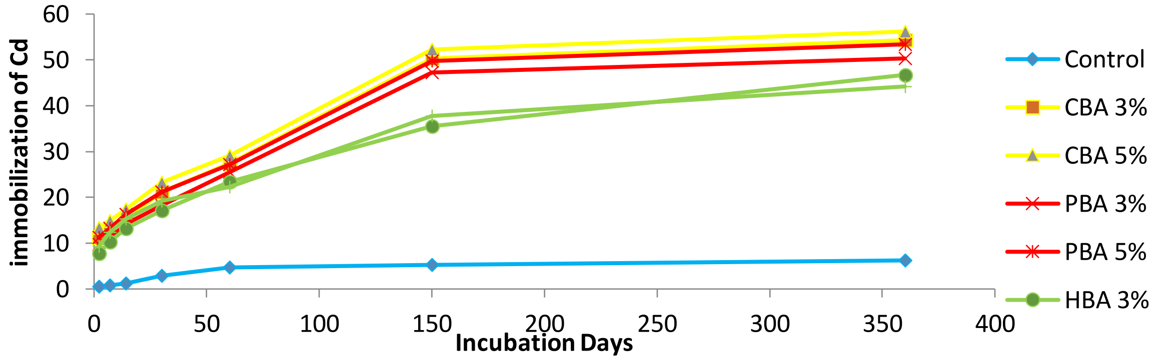

Fig 5: Immobilization of Cd in Sandy Soil

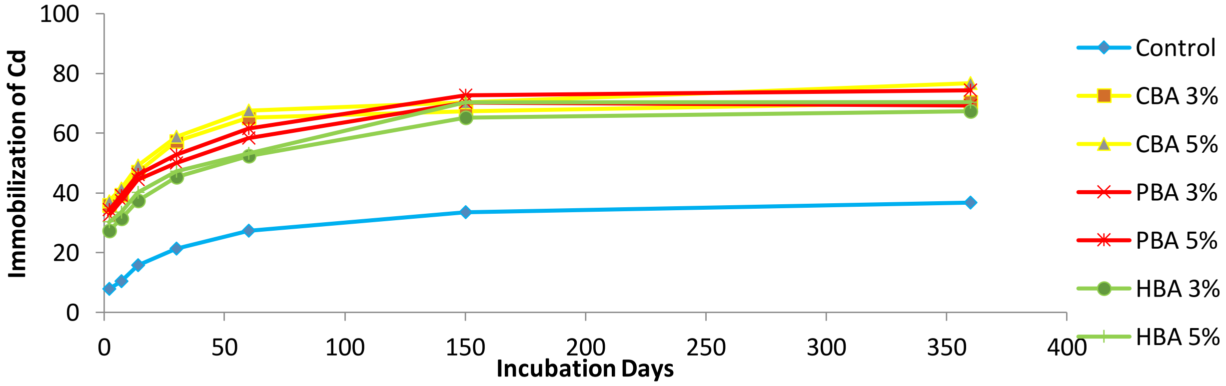

Fig 6: Immobilization of Cd in Laterite Soil

Cd immobilization in clay, sandy, and lateritic soils is illustrated in Figures 4–6. As shown in Figure 4, the concentration of cadmium was reduced by 67.2% by CBA, 64.4% by PBA, and 58.86% by HBA through natural immobilization processes such as adsorption and desorption, ion-exchange, and electrostatic attraction. Meanwhile, PBA reduced the concentration of Cd to 17.8 mg/kg, and HBA immobilized Cd from 50.02 mg/kg to 20.57 mg/kg. The results of the amendments on cadmium immobilization in sandy soil are illustrated in figure 5. When it came to immobilization, CBA had the best rate at 56.2%. One possible explanation is the high concentration of calcium phosphate. Amendments to sandy soil result in a lower amount of Cd immobilized compared to clay and lateritic soils. Because sandy soil has very little organic matter, it has a limited cadmium immobilization capacity. Ping et al. (2016), Cheng and Hseu (2002), and Lee and Shen (2004) found that organic matter adsorbed by soil oxides can form tri-complexes with cadmium, increasing the sorption strength of the mineral. The dissolution and precipitation of comparatively insoluble octaviate (Cd (CO3)) could perhaps be responsible for the decrease in Cd content [29]. Figure 6 shows that the metal concentration in the soil was reduced from 50.02 mg/kg to 6.04 mg/kg, 8.34 mg/kg, and 10.80 mg/kg, respectively, due to the percentage immobilization of Cd by CBA, PBA, and HBA. By increasing solubility and precipitating the insoluble complex, iron oxide may have enhanced metal adsorption, leading to a drop in the concentration of bioavailable metals in the soil.

CONCLUSION

Surface morphological analysis of CBA, PBA and HBA using scanning electron microscope (SEM) revealed existence of large and high concentration of calcium, carbon and phosphorus atoms. Analysis of the soil samples reveals that they all contain low quantities of heavy metals. The percentage immobilization of the metal also increases as time increases but at a time of 150days immobilization decreases as a result of the depletion of the specific sorption sites and increase in the non-specific sorption sites. Comparative analysis of data in this investigation showed that Cow Bone Ash (CBA) Pig Bone Ash (PBA) and Horse Bone Ash (HBA) can be used to immobilize and thus remediate Clay soil, Sandy soil and Lateritic soil contaminated with Pb, Cd and Cr. The efficiency of the immobilizing agent is in the order CBA>PBA>HBA while that of the soil is in the order Laterite>Clay >sandy.

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