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Lead and Cadmium Ions Mitigation in Soil using Calcium Carbide Waste-Carbonised *Lophira Alata* Sawdust Composite (CCW-CLAS) as Immobilizing Agent

Okponmwense Moses[1*] and Queen Omone Asekhame[1]

[1]Department of Chemistry, Faculty of Physical Sciences, University of Benin, Benin City, Nigeria
*Corresponding author's email: okponmwense.moses@uniben.edu

Abstract – Soil contamination with heavy metals is a global environmental matter of interest due to its thinkable dangers to humans, environmental health, and the ecosystem preceding shortfalls in agricultural produce and dangerous health outcomes as they penetrate the food chain. This study aimed to mitigate lead and cadmium ions contaminant in soil using calcium carbide waste-carbonized *lophira alata* sawdust composite (CCW-CLAS). Contaminated soil samples were collected from Evbareke spare parts market, Benin City, Edo state Nigeria, using a grid sampling method at a depth of 0-20cm with a soil auger. 50% W/W of the powdered calcium carbide waste and *lophira alata* sawdust was placed in a ceramic crucible, put into a muffle furnace, and heated at 350ºC for 3 hours. The X-ray diffraction analysis for the calcium carbide waste-carbonized l*ophira alata* sawdust composite (CCW-CLAS) revealed that it was made up of predominantly calcite fraction (55.0%), portlandite (25.0%), coesite (7.3%), muscovite (6.7%), dolomite (4.0%) and lime (2.1%) this was supported by FTIR analysis. SEM analysis revealed that calcium carbide waste-carbonized *lophira alata* sawdust composite (CCW-CLAS) possesses a rough surface with a pore of varied size and irregular shape. The contaminated soil from the Evbareke spare parts market was heavily polluted with petroleum hydrocarbons, and the soil was highly saline with low C.E.C. The soil texture was loamy sand; as the amount of CCW-CLAS increased in the soil, the amount of cadmium and lead ions that was releasable in the soil reduced, indicating that CCW-CLAS was effective in immobilizing cadmium and lead ions. The optimum amount of the amender in the contaminated soil to immobilize lead ion was 4% w/w, and cadmium ion was 2%.

Keywords: soil, remediation, immobilization, contamination, disassembling, automobile, heavy metal.

Introduction

Soil contamination with heavy metals is a global environmental matter of interest owing to its thinkable dangers to human, environmental health, and ecosystem preceding shortfalls in agricultural produce and dangerous health outcomes as they penetrate the food chain (Guala *et al.,* 2010; Bielicka-Giełdoń *et al.,* 2013; Moses and Obasigie, 2020). The entire quantity of potentially accessible metals (quantity/capacity factor), the activity and the ionic ratios of metals in the soil solution (intensity factor), as well as the rate of metal relocation from solid to liquid phases and to plant roots (reaction kinetics), are the three factors that determine the relocation of heavy metals from soils to plants (Moradkhani *et al.,* 2013, Bakshi *et al.,* 2018).

Automobile spare parts sourcing from disassembling unserviceable vehicles is a means of getting recyclable automobile parts. However, this activity leaves a trail of contaminants in the soil made up of organic and inorganic compounds, such as heavy metals. Heavy metals are of noticeable worry owing to their toxicity, extensive supplies, non-biodegradable properties, and amassing behaviors (Akoto and Abankwa, 2014; Adeyi and Torto, 2014; Topcuoğlu, 2016). Heavy metals in soil are potentially accessible for plant uptake if the mobility Factor exceeds 10%, and high mobility factors indicate that the metals originated from anthropogenic sources (Chengo *et al.,* 2013).

Bioavailability is the quantity of ingested metal ions absorbed in soil cavities. It relies on the metals' dividing wall in the middle of the solid and solution phases (Abdu, 2010; Violante *et al.,* 2010). The major factors that influence the heavy metal mobility and bioavailability to plants are entire metals content in the soil, metals forms,

the soil pH, organic matter content, and clay fraction (Akoto and Abankwa, 2014; Okoro *et al.,* 2012; Benson *et al.,* 2013; Gawdzik *et al.,* 2015). The most active and bioavailable heavy metals in the fractionated form are water soluble, exchangeable, and carbonate-bound species, and those associated with clay minerals as relatively inactive (Abdu, 2010).

The best-demonstrated technologies (BDATs) for the remediation of heavy metal-contaminated locations, such as immobilization, soil washing, and phytoremediation procedures, are the most regularly used (Kede *et al.,* 2014). The process of decreasing metal mobility, bioavailability, and bioaccessibility of heavy metals in soil by adding immobilizing agents such as an organic and inorganic amendment to the contaminated soils is called immobilization or waste fixation. This process helps to transform heavy metals into harmless forms (Fahmi *et al.,* 2018; Khalid *et al.,* 2017; Zaghloul and Saber, 2019). The basic function of immobilizing amendments is to change the prototype soil metals to further geo-chemically stable phases through sorption, precipitation into insoluble hydroxides, carbonates or silicates, ion exchange as well as complexation processes, swap of metals into a mineral structure, physical encapsulation and other possible means (Bakshi *et al.,* 2018; Khalid *et al.,* 2017; Sarkar *et al.,* 2014). Soil amendments like clay, cement, Zeolites, minerals, phosphates, fly ash, organic composts, and microbes are the most applied (Soltan *et al.,* 2012; Wuana *et al.,* 2013). Recently, heavy metals in contaminated soils have been immobilized using cheap industrial remainders such as red mud, termitaria, and industrial eggshells (Khalid *et al.,* 2017; Ba-Naimoon and Hamid. 2016, Muhammad *et al.,* 2018).

Lead (Pb), which mounts up in the body organs, precedes poisoning (plumbism) or even death. The gastrointestinal tract, kidneys, and central nervous system are likewise influenced by lead. Children open to lead are in danger of impaired growth, low IQ, shortened attention span, hyperactivity, and mental deterioration [(Chen *et al.,* 2014; Lakherwal, 2014; Singh and Gupta, 2016; Abdel-Raouf and Abdul-Raheim, 2017)**.** Cadmium is extremely harmful to humans, plants, and animals. Detrimental consequences of cadmium in humans include renal damage, emphysema, hypertension, and testicular atrophy. The mainly serious type of Cd toxicity in humans is "Itai-itai," a disease typified by the agonizing ache in the bone (Singh and Gupta, 2016).

In order to attenuate the consequences of lead (Pb) and cadmium (Cd) presence in contaminated soil in and around the Evbareke spare parts market, this research was intended to assess the degree of lead and cadmium ions mitigation with the use of calcium carbide waste-carbonized *Lophira alata* sawdust composite (CCW-CLAS) as an immobilization materials

Materials and Methods
Study area description, soil sample collection, and pre-treatment
A collection of sixty (60) samples out of a possible sixty-four (64) sampling points of contaminated soil samples were collected from Evbareke spare parts market in Evbareke of latitude 6^0 22' 00"N and longitude 5^0 36' 00"E. The control soil was taken from Evbareke senior Secondary School at latitude 6^0 21' 33"N and longitude 5^0 37' 00"E, in Egor Local Government Area, Edo State, Nigeria, at the depths of 0-20cm using a soil auger, on a land size of approximately 450m² (30m x 15m). The grid sampling method was used for gathering the soil sample. The samples were collected at regularly spaced intervals over space or time. After removing the trashes, the soil samples were air-dried at ambient temperature, ground, and sieved to give <2mm particle size and composited as the parent soil. The remaining moisture was dried by heating at 105±5°C for 3 hours in an oven.

The soil sample was physiochemically characterized for pH, Particle size analysis – Hydrometer method, electrical conductivity, cation exchange capacity, total phosphorus, total petroleum hydrocarbon, total Organic carbon, and moisture content using the standard method (North Central Region-13, 1998). The heavy metal content in the soil was determined using sequential extraction procedures-Fractionation**,** adopted from Tessier *et al.* **(**1979). The heavy metal content in every fraction was determined using the atomic absorption spectrophotometric (AAS) VGP 210 model – Buck Scientific Equipment Inc.

Preparation of calcium carbide waste-carbonized *Lophira alata* composite (CCW-CLAS)
The calcium carbide waste was collected from a panel beating workshop at Isiohor near Nigeria Army School of Supply and Transport (NASST) in Isiohor, Benin city, Edo state. The calcium carbide waste was air-dried for 3 weeks at ambient temperature. The dried calcium carbide waste was crushed and ground into powdery form. 50% W/W of the powdered calcium carbide waste and *Lophira alata* sawdust was put in a ceramic crucible,

positioned into a muffle furnace, and heated at a temperature of 350°C for 3 hours until a dark ash composite powder was obtained.

Characterization of calcium carbide waste-carbonized-*Lophira alata* sawdust (CCW-CLAS)

The CCW-CLAS Composite prepared was characterized using Fourier Transform-Infra Red (FT-IR System, spectrum BX, PerkinElmer, England), Scanning electron microscopy (SEM; phenom pro suite desktop scanning electron microscope), and x-ray diffraction (with X-Ray diffractometer, Shimadzu 6000 model). Immobilization assessment of lead and cadmium from soil amended with CCW-CLAS composite using column test leaching method (CTLM). Chezom et al. (2013) adopted the column test leaching method with minor alterations applied to confirm the level of in-situ immobilization of lead and cadmium. The leaching technique was done in a column filled with different mass proportions of CCW-CLAS composite and the soil sample at an L/S ratio of 2:1. The column containing the amended soil sample of 50g was supported on a retort stand. The end of the column was covered with a semi-permeable membrane, and 100ml of distilled water loaded with 1% HCl (v/v) was poured into the column and allowed to stand for 1 hour, after which it was perforated so that the leachate would flow through the vertical column of the amended soil in a down-flow manner and the leachate obtained was analyzed using AAS. Table 1 shows the different mixture ratios of contaminated soil samples and CCW-CLAS composite placed in each column.

Table 1. Ratios of Soil Sample and Calcium Carbide Waste-Carbonized *Lophira Alata* Sawdust (CCW-CLAS) Composite Mixture.

| Percentage	Weight of CCW-CLAS Composite (g)	Weight of Soil (g)
2%	1	49
4%	2	48
6%	3	47
8%	4	46
10%	5	45

The relative index mobility of these metals was calculated as the mobility factor (M_F) (Osakwe and Okolie, 2015) using the following equation (1).

$$M_F = \frac{F_1 + F_2 + F_3}{F_1 + F_2 + F_3 + F_4 + F_5 + F_6} \text{ X } 100 \tag{1}$$

Results

Characterization of calcium carbide waste-carbonized-*Lophira alata* sawdust (CCW-CLAS)

The results of the characterization of calcium carbide waste-carbonized *lophira alata* sawdust (CCW-CLAS) composite for its functional group identification, crystallinity, and surface morphology were obtained using Fourier Transform-Infra Red (FT-IR), x-ray diffraction (XRD) as well as Scanning electron microscopy (SEM). The results are shown in Figures 1, 2, 3, and Figure 4, respectively:
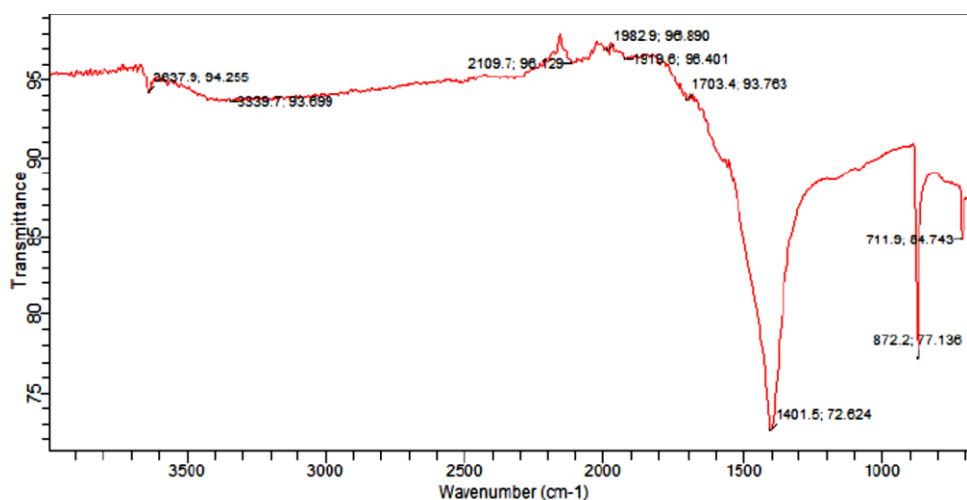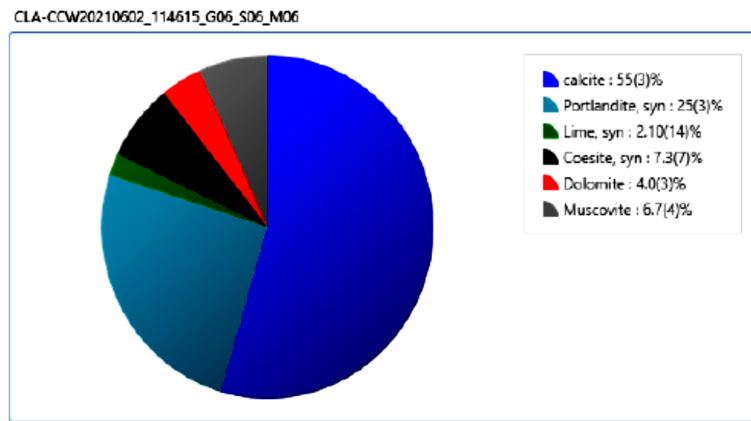

Figure 1. FTIR Spectrum for CCW-CLAS composite.
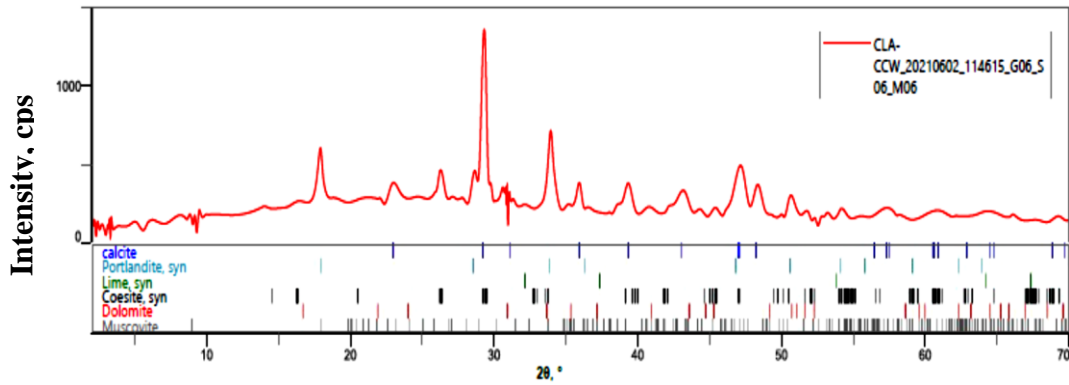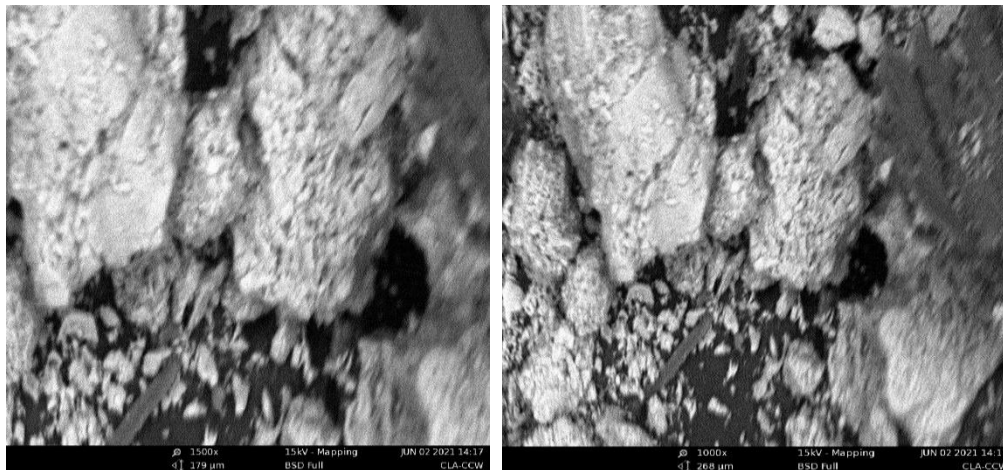
Figure 2. X-ray defractogram for CCW-CLAS composite.

Figure 3. Plot and table of the result of X-ray for CCW-CLAS composite.

a

b

Figure 4. SEM pictures of CCW-CLAS Composite.

The Results of the soil sample's physicochemical and geochemical characterization are shown in Tables 2 and 3. It expressed the quality of the contaminated soil and the control site soil in determining the level of contamination and their resultant consequences.

Table 2. Results of the physicochemical characterization of the soil sample.

Parameters	Units	Contaminated soil	Control Soil
pH	-	6.24	5.43
Electrical conductivity	µS/cm	1037.50	235.00
Total phosphorus	mg/kg	372.58	202.20
Total petroleum hydrocarbon	mg/kg	107,304.59	4.04
Cation exchange capacity	Cmol/kg	9.89	4.34
Total organic carbon	%	3.63	1.11
Moisture content	%	1.05	0.84
Particle size analysis	Clay (%)	4.38	5.88
	Silt (%)	3.60	7.60
	Sand (%)	92.02	86.52

Table 3 Results of the soil sample's geochemical (soil fractionation) characterization for lead and cadmium.

Fraction	Contaminated soil (mg/kg)		Control soil (mg/kg)	
	Lead	Cadmium	Lead	Cadmium
Soluble	0.21	0.03	BDL	BDL
Exchangeable	1.07	0.20	BDL	BDL
Carbonate-bound	7.50	0.20	0.71	BDL
Fe-Mn oxide bound	21.07	0.47	0.71	BDL
Organically bound	3.21	0.20	BDL	0.07
Residual	19.64	BDL	BDL	BDL
Total	52.70	1.10	1.42	0.70

Note: BDL=below detectable level

A comparison of the quantity of bioavailable lead and cadmium ions in the contaminated soil vis-à-vis the highest acceptable concentration (HAC) by NESREA is shown in table 4. The effect of calcium carbide waste-carbonized *Lophira alata* sawdust (CCW-CLAS) on the leaching of cadmium and lead from contaminated soil is shown in Table 6 and Figure 5.

Table 4. Bioavailable lead and cadmium of the geochemical fraction in the contaminated soil sample vis-à-vis the highest acceptable concentration (HAC) by NESREA.

Fraction	Contaminated soil (mg/kg)		Control soil (mg/kg)	
	Lead	Cadmium	Lead	Cadmium
Soluble	0.21	0.03	BDL	BDL
Exchangeable	1.07	0.20	BDL	BDL
Carbonate-bound	7.50	0.20	0.71	BDL
Total	8.78	0.46	0.71	BDL
HAC (NESREA, 2009)	10	3.0		

Note: BDL=below detectable level

Table 5. Percentage of lead in various geochemical fractions in the contaminated soil sample.

Fraction	Contaminated soil (%)		Control soil (%)	
	Lead	Cadmium	Lead	Cadmium

Soluble	0.40	2.73	0.00	0.00
Exchangeable	2.03	18.18	0.00	0.00
Carbonate-bound	14.23	18.18	50.00	0.00
Fe-Mn oxide bound	39.98	42.73	50.00	0.00
Organically bound	6.09	18.18	0.00	100.00
Residual	37.27	0.00	0.00	0.00
Mobility Factor	16.66	39.09	50.00	0.00

Table 6. The effect of CCW-CLAS composite on cadmium leaching and lead from contaminated soil.

Percentage of CCW-CLAS Composite	Weight of CCW-CLAS Composite (g)	Weight of Soil (g)	Amount of Leached lead from contaminated soil (mg/g)	
			Lead	Cadmium
2%	1.00	49.00	0.02	BDL
4%	2.00	48.00	0.01	BDL
6%	3.00	47.00	0.01	BDL
8%	4.00	46.00	0.01	BDL
10%	5.00	46.00	0.01	BDL

Discussion

Characterization of calcium carbide waste-carbonized-*Lophira alata* sawdust (CCW-CLAS)

The FT-IR spectra revealed characteristic bands at 3637.9 cm^{-1} due to –OH stretching attributed to Ca(OH)$_2$, SiOH group, alcohols (R-OH), and phenols (Ar-OH) groups. The appearance at the 3,339.7 cm^{-1} band is due to the OH-stretching band of the hydroxyl group attributed to Alcohol (ROH), phenol (ArOH); N-H stretch of primary aliphatic amines (R-NH$_2$), primary aromatic amine (Ar-NH$_2$) and amide (-CONH). The appearance of diverse amine groups in CCW-CLAS makes it an efficient encapsulating agent and an effective sorbent for metal ions via chelation. The peak at 1401.5cm^{-1} represents –OH bending attributed to tertiary alcohol and phenol; silicon compounds (-SiOH) and CO$_3^{2-}$ broad and sharp band was attributed to calcium carbonate CaCO$_3$, while the peaks at 872.2 representing CaO, CO$_3^{2-}$ the strong and sharp band was attributed to calcium carbonate (CaCO$_3$) and SiO$_3^{2-}$ attributed to calcium silicate (CaSiO$_3$). 711.9 represent CO$_3^{2-}$ the weak band also attributed to calcium carbonate (CaCO$_3$). The presence of these inorganic groups in CCW-CLAS can bring about a rise in the soil pH of the environment hence making the cadmium and lead non-bioavailable. The presence of the other functional groups will increase the heterogeneity and thereby increase their ability to retain lead and cadmium ions, thus preventing their leaching into the environment.

The diffractogram plot and table of the result of the x-ray for CCW-CLAS composite revealed that it was made up of predominantly calcite fraction (55%), which is a calcium carbonate mineral component of limestone (Figure 3); others are portlandite (25%) which is the naturally occurring form of calcium hydroxide, the calcium analog of brucite, coesite (7.3%) – a form of silicon oxide (SiO$_2$), muscovite (6.7%) – a form of hydrated phyllosilicate mineral of aluminum and potassium, dolomite (4.0%) - a type of limestone rich in magnesium and calcium carbonate (CaMg(CO$_3$)$_2$) and lime (2.10%) – this is a calcium-containing inorganic mineral composed primarily of carbonates, oxides, and hydroxides. The X-ray diffractogram confirms that the CCW-CLAS composite is made up of carbonates, oxides, and hydroxides of calcium which can raise the pH of the soil environment, thereby decreasing the availability of the cadmium and lead ions for uptake by plants to a large extent.

The SEM photograph, at 1,500 and 1,000 times magnification, showed that the CCW-CLAS composite appears porous and possesses a rough surface. The pore is irregularly shaped and varies in size. The surface area, fragmentation of the particles, and the difference in pore size describe its ability to retain cadmium ions and lead ions, thus preventing its leaching. This coincides with Ince and Ince's (2017) observation that a large surface area and a high number of pores in materials (e.g., activated carbons) have been responsible for removing heavy metals.

The physicochemical characterization of the contaminated soil from the Evbareke spare parts market revealed that the soil was heavily polluted with total petroleum hydrocarbon (TPH) (Table 2). This was an indication of anthropogenic activities in the spare parts market. Edori *et al.* (2020) asserted that the ensuing

consequence of this pollutant would bring about the relapse of the physicochemical and biochemical properties of the soil as well as limit the growth of plants. They also opined that the TPH would cause oxygen and water insufficiencies and phosphorus and nitrogen deficiency based on nutrients in the soil. The contaminated soil contains high soluble salt content (salinity) based on its electrical conductivity value compared to the soil from the control site; this indicated lead and cadmium availability. Du Laing *et al.* (2007) asserted that metal availability increases with salinity. The cation exchange capacity was rated low because its value falls between 6-12 Cmol/kg (Hazelton and Murphy, 2007), indicating that the soil has a low resistance to change in soil chemistry triggered by land use (Table 2). The value of pH obtained for the contaminated soil is 6.24 (Table 2); it is slightly acidic and could have contributed to the bioavailability of cadmium and lead ions in the contaminated soil; however, the heavy metals would have been more bioavailable in the control site soil if it had contained as high a quantity of lead and cadmium as in the contaminated soil. The particle size analysis for the contaminated soil revealed that the soil textural class was loamy sand, and the clay quantity was very low (< 5%) hence having a little or no withholding effect on the lead and cadmium ions.
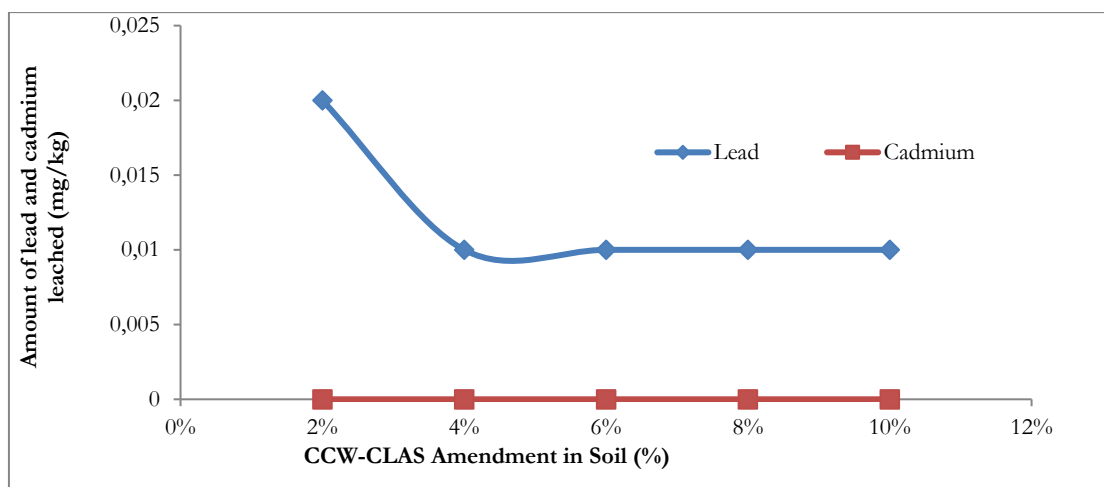

Figure 5. Effect of CCW-CLAS effect in remediating lead in contaminated soil.

Geochemical (Soil Fractionation) Characterization of the Soil Sample for Lead and Cadmium

The combined amount of lead and cadmium ions in the soluble, exchangeable, and carbonate bound fraction was less than that in the Fe-Mn oxide bound fraction (Table 3). Chengo *et al.* (2013) asserted that heavy metals in soil are potentially accessible for plant uptake if the Mobility Factor is above 10%; this indicated that the lead and cadmium ions were bioavailable since they had a mobility factor of 16.66 and 39.09%, respectively (5).

On comparing the bioavailable amount of lead and cadmium ions in the contaminated soil with the highest acceptable concentration (HAC) from NESREA (2009), it revealed that the amount of available lead and cadmium ions in the contaminated soil from the Evbareke spare parts market was lesser (Table 4). However, the total amount of lead in the contaminated soil (52.70mg/kg) exceeded the highest acceptable concentration (HAC) from NESREA, an indication that the contaminated soil was below pollution level in terms of bioavailability and well above the pollution level in term of the total amount of the heavy metals for the lead only.

The water-soluble, exchangeable, and carbonate fractions ($F_1 + F_2 + F_3$) are the main reactive, mainly mobile, and potentially accessible or bioavailable fractions because they are weakly or loosely bound to soil components (Table 5). The metals in these fractions are accessible by man via ingestion and are commonly deemed as being of anthropogenic origin.

Effect of CCW-CLAS composite on leachability of cadmium and lead from contaminated soil

Despite the low mobility factor of lead and cadmium ions in contaminated soil, CCW-CLAS composite was able to further reduce their bioavailability in the soil by in-situ immobilization remediation pattern, most likely through adsorption and complexation mechanism for the cadmium and lead ions. Thereby making it unavailable

for uptake by plants hence reducing its transmission to man and animals, indicating that the CCW-CLAS amendment has transformed most lead and cadmium ions into insoluble or unavailable forms. Ogundiran and Osibanjo (2009) asserted that if heavy metals are obtained mainly in the soluble form, they may be leached down the soil profile attaining the groundwater or taken up and stored by plants, invertebrates, animals, and man. The amount of lead ion that was releasable or made available in the soil decreased appreciably as the amount of CCW-CLAS amendment increased. However, no soluble or available form of cadmium ions was leached. When the proportion of CCW-CLAS amendment reached 4% w/w level, the number of lead ions leached remained constant at 0.01mg/kg, and the cadmium ions were below detectable level, even when the amendment dosage increased to 10% w/w CCW-CLAS. This revealed that the optimum amount for the remediation of the contaminated soil was 4% w/w (2.00g-CCW/48.00g-contaminated soil) for the lead contaminant and <2% w/w (<1.00g-CCW/49.00g- contaminated soil) for cadmium contaminant (Figure 5). Calcium carbonate may have acted as the strong adsorbent for lead and cadmium ions since it can be complex as double salts like $CaCO_3.MCO_3$ (Rostami and Ahangar, 2013) (where M stands for heavy metals) and Carbonized *lophira alata* sawdust also acted as an adsorbent; this implies that the joint consequence of the complexation, as well as adsorbent, is most likely accountable for the in-situ immobilization of the lead and cadmium ions in the contaminated soil.

Conclusion

CCW-CLAS was able to appreciably reduce the lead and cadmium ions in the soil by in-situ adsorption and complexation remediation pattern, thereby making them appreciably less available for uptake by plants hence reducing their transmission to man and animals. As the amount of CCW-CLAS amendment increased in the soil, the amount of cadmium and lead ion that was releasable or made available in the soil reduced when the amendment increased from 0 – 4% w/w and remained constant at 0.01mg/kg for lead ions released, even when the amendment dosage increased to 10%w/w CCW-CLAS, However, at the application of the amendment, the amount of cadmium leached was below the detectable level. This revealed that the optimum amount of CCW-CLAS composite for the remediation of the contaminated soil was at 4% w/w (2.00g-CCW/48.00g contaminated soil) for lead contaminant and ≤2% w/w (<1.00g-CCW/49.00g- contaminated soil) for cadmium contaminant, this observation indicated that CCW-CLAS was effective in immobilizing cadmium and lead ions. However, it immobilized the cadmium ion more than the lead ion.

Acknowledgment

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