International Journal of Human Capital in Urban Management
(IJHCUM)

Homepage: http://www.ijhcum.net/

ORIGINAL RESEARCH PAPER

Labile metal evaluation, speciation and accumulation in harvested plant from urban major dumpsites

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ARTICLE INFO

Article History:
Received 08 June 2020
Reviewed 13 August 2020
Revised 11 October 2020
Accepted 14 November 2020

Keywords:
Absorption
Contaminants
Hazardous
Ingestion
Labile metals

ABSTRACT

BACKGROUND AND OBJECTIVES: Disinterred manures from dumpsites in the cities are believed to be readily available source for soil nutrient for backyard farming. Health hazards posed on human due to labile metals contaminants are not considered or evaluated before consumption. Three major municipal dumpsites from Okitipupa (Waste Management (OKA), Igodan(OKB), and Okitipupa Oil Mill Road (OKC)) were analyzed for the concentrations, forms of labile metals in the soil and also concentration in green vegetables from the sites. The objectives of the research were to investigate the labile metal concentration, forms of the labile metals in the soil and their accumulation in plants from these major urban dumpsites.
METHODS: Dried and digestedSoil and vegetable samples from the sites were analyzed for total concentration of labile metals and their forms through speciation in the soil were equally quantified. Concentrations from sample solutions were determined by Atomic Absorption Spectrophotometer.
FINDINGS: Labile metals concentrations from the soilof Waste Management Dumpsite (OKA), IgodanDumpsite (OKB) and Okitipupa Oil Mill Road Dumpsite (OKC) indicated that Cadmium (Cd) values range from87.453mg/kg-106.500mg/kg). Copper (Cu) in the three samples ranged between 3.100-5.510mg/kg, which are significantly low and beyond the toxicity level as well as cobalt (Co). Chromium (Cr) was higher in OKA (22.980mg/kg) and OKC (10.560mg/kg) and least in OKB (2.900mg/kg).Iron was the most abundant ranging from 3690.000-6780.000mg/kg, followed by zinc ranging from385.000-2880.000mg/kg. Speciation of the labile metal indicate that the metal exist mostly in the inert fraction and easily absorbed by plant.
CONCLUSION: The concentrations of the most labile metals in soil samples were high and majorly exist in inert fraction after speciation. Also, the concentrations in the plants were almost half of the concentration in the soil which indicated that they are not desirable for human consumption due to their toxicity level.

DOI: 10.22034/IJHCUM.2021.02.07

NUMBER OF REFERENCES
33

NUMBER OF FIGURES
4

NUMBER OF TABLES
3

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Note: Discussion period for this manuscript open until July 1, 2021 on IJHCUM website at the “Show Article.

INTRODUCTION

Labile metals constitutes a major category of pollutants in land because in large enough doses it can

Prove lethal to organisms including humans. These metals can be released into the terrestrial system by both natural and anthropogenic sources and during the course of their transportation can be distributed in water bodies, suspended sediment and bed sediment. Labile metals occur in an unusual or abnormal concentration in terrestrial system as a result of pollution from domestic wastes, mining process and other industrial activities (Olajire and Imeokparia, 2000; Adefemi and Awokunmi, 2010). These metals can be transported by particulate matters to the atmosphere. There is very large set of health consequences from exposure to soil contamination depending on pollutant type, pathway off attack and vulnerability of the exposed population, lead is especially hazardous to young children and for whom there is a high risk of developmental damage to the brain, while to all population kidney damage is a risk. Although, many potential contaminants are required in trace amounts by plants for food production but they become hazardous when they occur in excess in the soil (Adefemi and Awokunmi, 2010). Labile metal toxicity can cause several diseases affecting almost all the vital organs and functions (Nwajei *et al*., 2012). Unlike organic pollutants, labile metals do not decay and hence persist in the environment. They have the potential of bioaccumulation and biomagnifications also (Fagbote and Olanipekun, 2010). Soil often forms a repository of these elements because soil particles such as clay and humus have charges that help the metal cations to bind themselves with the soil, and thus prevents their release, though temporarily. The soluble forms of labile metals are more dangerous because they are readily available to plants and animals (Srivastava and Singh, 2012).

Labile Metal Pollution in Vegetables

Swift industrialization and urbanization have contributed to the eminent levels of labile metals in the urban environment. The developing countries such as United States of America, European communities, China and India are most affected as explained by Wong *et al.,* 2003; Tripathi and Singh, 1997; Sharma *et al.,* 2009; Sharma *et al*., 2008, . Labile metals are non-biodegradable and unrelenting ecological contaminants which may be deposited on the top soil and sub soil from dumping waste and then adsorbed into the tissues of the vegetables (Singh and Agarwa, 2010).

Effects of Labile metals on the growth of plants

The effects of labile metals on plants are different in diverse growth stages of plants (Shuiping, 2003). Cd inhibits the photosynthesis and growth of rice in the early stage, then inhibits the reproductive organs' differentiation, and lastly distributes the nutrients transport and mobilization (Wang, 1996), but a stumpy concentration of Hg (10^{-5} mol/L) stimulated the growth of wheat seedlings. The reason for this may be as a result of low concentrations of Hg which increased the activities of amylase, proteinase and lipase and speed up the decomposition of endosperm and the respiration rate, so that the germination was more rapid (Ma and Hong, 1998). Cd and Pb are labile metals which are non-essential elements for plants development (Xu and Shi, 2000). The germinating ratio of barley was lower than 45 % and the growth of roots were dormant under 10 -2 mol/L Cd application (Shuiping, 2003).The seedlings of bean turned brown and died under Cd stress (Mo and Li, 1992). The target organs by Cd pollution were the roots. That the root growth of crops such as wheat (Shuiping, 2003; Hong *et al.,* 1991), maize, pumpkin (Liu and Cui, 1991), cucumber (Chen, 1990), and garlic *(Allium sativum L)* (Liu *et al.,* 2000) were inhibited.

Effects on absorption of nutrients

The hydroponical experiment of oat revealed that the absorption aptitude of K and Mg declined in suspended cultivated cells, and the absorption of Ca, Fe and Zn increased by Cd pollution. Conversely, absorption of Zn reduced in higher concentrations of Cd solution (Xu and Yang, 1995). Wang (1990) also reported that Cd drastically inhibited maize seedlings from absorbing N, P and Zn and enhanced the absorption of Ca. Cd also affected the absorption of Mn and Zn by roots of B. chinensis seedlings (Qin *et al.,* 1994), inhibited the absorption of Fe, Mn, Cu, Zn, Ca and Mg by rye grass *(Loliumperenne),* maize (*Zea mays*), shamrock (*Trifoliumrepens*) and cabbage (*B. oleracea var. capitata)* and increased the absorption of P (Yang *et al.,* 1998).

Contradictory effect of Labile Metals on Fertilization

Through continuous composting using dumpsite manure, the contents of labile metals in municipal waste did not become declined, but the bioavailable contents for plants were seen to reduce to the concentration in the waste (Shuiping, 2003). Fertilizing the highway greenbelt by the composting of sludge or dumpsite waste showed no more accumulation of heavy metals in plants (Xue *et al.,* 2000) and bioavailability contents of heavy metals were decreased by comprehensive composting. Generally, there are lower bioavailability contents of labile metals in the composted fertilizer due to more organic carbons with a higher pH (Guo-hang *et al*., 2018). In bio-solids used as fertilizer, most of the labile metals were combined with organic carbon and carbonate, the mobility was declined and also correlated to pH, elements in the soil and distinctiveness of the organic substances (Shuiping, 2003). Therefore, using plants to remediate the municipal waste solids is possible (Shuiping, 2003).

Water Contamination by Labile Metals

Water contamination by labile metals in some areas is practically unavoidable due to natural process (weathering of rocks) and anthropogenic activities (industrial, agricultural and domestic effluents). These elements, at concentrations exceeding the physiological requirement of the plants, not only could administer toxic effect in them but also could enter food chains, get biomagnified and pose a potentials threat to human health (USEPA, 2000).

The Environmental Risks

Pollution to lakes, river and the ocean by labile metals can be toxic to marine and fresh water organisms; additionally the ford chain can be affected via bioaccumulation. This initial effect to aquatic animals will eventually spread through the ecosystem making its way to the top of the food chain-which is often humans (USEPA, 2000). However**,** this research aimed at determining the concentration of labile metals from three major dumpsites in Okitipupa town in southern part of Ondo State and the rate of absorption of labile metals in green vegetable harvested after three weeks of planting obtained from these dumpsites. The objectives are to determine the concentration of labile metals in various soil samples collected from the sites, to evaluate the rate of absorption of labile metals in green vegetables harvested from different dumpsites investigated. The current study has been carried out in Okitipupa in Ondo State, Nigeria in year 2019.

MATERIALS AND METHODS

Collection of material

Soil samples were obtained from different dumpsites (Waste Management Dumpsite, Igodan Dumpsite and Okitipupa Oil Mill Road Dumpsite) as indicated in Fig.1 and shown in Figs. 2, 3 and 4 in Okitipupa town,Okitipupa local government area, which is part of Ikale geographical location in Ondo State, Nigeria. Okitipupa had geographical coordinates of $6^{0}30^{1}$ North and $4^{0}48^{1}$ East of the meridian. Okitipupa has an area of 803 meters square and population of 272,030(2011 estimation) in Ondo state, Nigeria. The green vegetable (*Amaranthus hybridus*) seeds were purchased from king's market in Akure town in Ondo state, Nigeria. The samples from the studied sites in

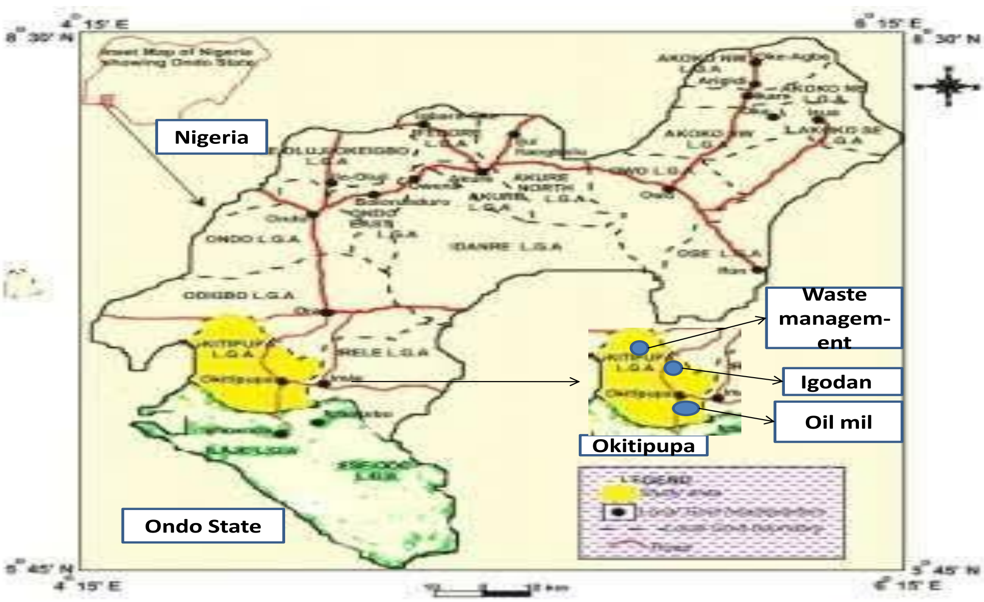

Fig. 1: Location of the sampling area in Okitipupa

Fig. 2: Waste Management dumpsite,

Fig. 3: Igodan road dumpsite,

Fig. 4: Oilmill dumpsite

Okitipupa were collected and brought to Analytical research laboratory, Rufus Giwa Polytechnic, Owo and analyzed for the total concentration of Labile metals, forms at which the metals exist and the rate of absorption in green vegetables after three weeks of growth.

Preparation of Soil and Plant Samples

Soil samples were manually dug from different point source by random sampling method in the site and kept inside a clean five litter buckets. Green vegetables were planted at the different point source after minor clearing for easy germination and harvested after three weeks. Both the soil and the vegetable were oven dried at 105^{0}C in thermosetting oven. The vegetable samples were reduced to fine powder with the aid of a mechanical mince to pass through 40 mesh sieves to increase the surface area for proper analysis. The milled powder samples were collected and stored in glass jars, tightly covered and kept for analysis.

Analytical Methods

AOAC (2000) was followed for proper digestion of dried soil and green vegetable samples for total concentration of labile metals and Sequential extraction of different forms of labile metals in the soil helps in quantifying the fractions or forms of labile metals in different phases as explained by (Tessier *et al.,* (1979) and made up to 100cm^3 mark with distilled water. The absorbance of the sample solutions were read by already standardize Atomic Absorption Spectrophotometer with appropriate lamp for the required metals.

Labile metal speciation- forms of labile metals (fractionation)

The procedure of Tessier *et al.,* (1979) was selected for this study. In this method, labile metals are separated into five operational defined fractions: exchangeable fraction, bound to carbonates, bound to iron and manganese oxide, bound to organic matter and residual fraction. The sequential extraction is as follows:

Step I, Exchangeable fraction: Following Tessier *et al.,* (1979), Samples (2g) were extracted at room temperature for 1hour with 16ml of $MgCl_2$solution (1M $MgCl_2$) at pH 7. Sediment and extraction solution were thoroughly agitated throughout the extraction. This is mainly an adsorption – desorption process. Metals extracted in the exchangeable fraction include weakly adsorbed metals and can be released by ion-exchange process. Changes in the ionic composition of the water would strongly influence the ionic exchange process of metal ions with the major constituents' of the samples like clays, hydrated oxides of iron and manganese. The extracted metals were then decanted from the residual samples for AAS analysis while the residue was used for the next extraction.

Step II- Bound to carbonates: Following Tessier *et al.,* (1979), the metals bound to carbonate phase are affected by ion exchange and changes of pH. The residue of Fraction 1 was extracted with 16ml of 1M sodium acetate/acetic acid buffer at pH 5 for 5 hours at room temperature. Significant amount of trace metals can be co-precipitated with carbonates at the appropriate pH. The extracted metal solution was decanted from the residual bitumen samples for AAS analysis. The residue was used for the next extraction.

Step III- Bound to iron and manganese oxides: Following Tessier *et al.,* (1979), the residue from fraction 2 was extracted under mild reducing conditions. About 13.9g of hydroxylamine hydrochloride ($NH_2OH.HCl$) was dissolved in500ml of distilled water to prepare 0.4M $NH_2OH.HCl$. The residue was extracted with 20ml of 0.4M $NH_2OH.HCl$ in 25% (v/v) acetic acid with agitation at 96°C in a water bath for 6hours. Iron and manganese oxides which can be present between particles or coatings on particles are excellent substrates with large surface areas for absorbing trace metals. Under reducing conditions, Fe (III) and Mn(IV) could release adsorbed trace metals. The extracted metal solution was decanted from the residual sediment for AAS analysis while the residue was used for the next extraction.

Step IV – Bound to organic matter and sulphide

Following Tessier *et al.,* (1979), the residue from fraction 3 was oxidized as follows: 3ml of 0.02M HNO_3 and 5ml of 30% (v/v) hydrogen peroxide, which has been adjusted to pH2, were added to the residue from fraction 3. The mixture was heated to 85°C in a water bath for 2hours with occasional agitation and allowed to cool down. Another 3ml of 30% hydrogen peroxide, adjusted to pH2 with HNO_3, was then added. The mixture was heated again at 85°C for 3hrs with occasional agitation and allowed to cool down. Then 5ml of 3.2M ammonium acetate in 20% (v/v) HNO_3 was added, followed by dilution to a final volume of 20ml with de-ionized water. Trace metals may be bound by various forms of organic matter, living organisms and coating on mineral particles through complexation or bioaccumulation. These substances may be degraded by oxidation leading to a release of soluble metals. The extracted metal solution was decanted from the residual bitumen samples for AAS analysis while the residue was used for the next extraction.

Step V – Residual or inert fraction: Following Tessier *et al.,* (1979), residue from fraction 4 was oven dried at 105oC. Digestion was carried out with a mixture of 5ml conc. HNO3 (HNO_3, 70% w/w), 10ml of hydrofluoric acid (HF, 40% w/w) and 10ml of perchloric acid (HClO4), 60%w/w) in Teflon beakers. Fraction 5 largely consists of mineral compounds where metals are firmly bonded within crystal structure of the minerals comprising the sediment. Analysis was carried out with AAS using GBC Avanta PM. Ver 2.02. To validate the procedure, the instrument was programmed and it carried out metal detection by displaying two absorbance readings and what was reported was the average. Blanks were also used for correction of background and other sources of error. Apart from calibration before use, quality checks were also performed on the instrument by checking the absorbance after every ten sampleruns.2 gram of the sample was extracted using 16ml of 1M $MgCl_2$solution (pH = 7.0 with stirring at room temperature for 1 hour.

RESULTS AND DISCUSSION

Tables 1 to 3 revealed the results obtained from labile metal concentration, forms in which the labile metals exist during speciation and the rate of absorption in green vegetables.

Sample Code: 1: Exchangeable Fraction, 2: Bound to Carbonates, 3: Bound to Iron or Manganese oxide, 4: Bound to Organic Mater or Sulphide, 5: Inert Fraction

OKA(1-5)= Waste Management Dumpsite

OKB(1-5)=Igodan Dumpsite

OKC(1-5)= Okitipupa Oil Mill Road Dumpsite

OKAV= Waste Management Dumpsite Vegetable

OKBV=Igodan Dumpsite Vegetable

OKCV- Okitipupa Oil Mill Road Dumpsite Vegetable

This research work was carried out to investigate the effect that the labile metals posed on human indirectly by seepage into water bodies during rainfall due to their concentration in the soil, the forms at which each metals exist in the soil during speciation and to investigate the effect of labile metals absorption in plants from the dumpsites when the soil from the sites were directly utilized for agricultural purposes. Ten labile metals concentration analyzed were shown in Table1, their forms were shown in Table 2 during speciation from1: Exchangeable Fraction, 2: Bound to Carbonates, 3: Bound to Iron or Manganese oxide, 4: Bound to Organic Mater or Sulphide, 5: Inert Fraction and the rate of absorption in green vegetables in

Table 1: Concentrations of Labile Metal from Three Dumpsites Soil in Okitipupa Region

Soil Sample Codes	Cd (mg/kg)	Cu (mg/kg)	Cr (mg/kg)	Co (mg/kg)	Fe (mg/kg)	Mn (mg/kg)	Zn (mg/kg)	Ni (mg/kg)	As (mg/kg)	Pb (mg/kg)
OKA	87.453	3.100	22.980	0.140	3690.000	210.250	2880.000	74.150	0.025	12.000
OKB	106.500	3.150	2.900	0.730	5250.000	147.800	1217.000	73.900	0.014	18.000
OKC	91.500	5.510	10.560	0.048	6780.000	37.450	385.000	46.200	0.063	12.000

OKA= Waste Management Dumpsite,OKB=Igodan Dumpsite, OKC- Okitipupa Oil Mill Road Dumpsite

Table 2: Chemical Speciation of Labile Metals from Three Major Dumpsites in Okitipupa Region

Sample Codes	Cd (mg/kg)	Cu (mg/kg)	Cr (mg/kg)	Co (mg/kg)	Fe (mg/kg)	Mn (mg/kg)	Zn (mg/kg)	Ni (mg/kg)	As (mg/kg)	Pb (mg/kg)
OKA1	0.042	0.090	0.400	0.020	1.500	0.090	0.350	0.130	0.006	0.080
OKA2	0.031	0.420	0.410	0.030	1.000	0.120	0.630	0.060	0.004	0.190
OKA3	0.210	1.090	1.000	0.010	0.700	0.170	1.180	0.320	BDL	0.050
OKA4	0.150	0.040	0.290	0.010	2.600	0.140	0.150	0.010	BDL	0.020
OKA5	87.000	1.360	20.86	0.070	3684.000	209.700	2877.940	73.620	0.015	11.65
OKB1	0.110	0.530	0.160	0.020	2.800	0.070	0.550	0.030	BDL	0.140
OKB2	0.070	0.080	0.310	0.020	3.600	0.060	0.490	0.060	BDL	0.020
OKB3	2.320	1.450	0.780	1.030	8.100	1.040	5.650	0.010	BDL	1.040
OKB4	0.510	0.920	0.300	BDL	1.800	0.120	0.500	BDL	0.001	BDL
OKB5	103.97	0.160	1.34	0.650	2503.500	146.500	1209.780	73.700	0.013	16.600
OKC1	0.160	1.380	0.390	BDL	0.800	0.150	0.240	0.040	BDL	BDL
OKC2	0.310	0.080	0.250	BDL	5.000	BDL	0.610	1.040	0.009	0.100
OKC3	0.180	1.150	0.250	0.010	3.500	0.050	0.310	0.410	0.005	0.150
OKC4	0.150	1.630	0.310	0.020	1.100	BDL	0.670	0.100	BDL	BDL
OKC5	90.710	1.25	9.340	0.018	6769.400	37.440	383.15	44.610	0.049	11.730

Note: BDL= below detection Limit

Table 3: Rate of Absorption of Labile Metals in Green Vegetables Planted on the Major Three Dumpsites in Okitipupa Region.

Sample Codes	Cd (mg/kg)	Cu (mg/kg)	Cr (mg/kg)	Co (mg/kg)	Fe (mg/kg)	Mn (mg/kg)	Zn (mg/kg)	Ni (mg/kg)	As (mg/kg)	Pb (mg/kg)
OKAV	23.660	0.2209	11.100	0.049	224.050	32.780	515.000	7.300	0.027	12.000
OKBV	28.960	0.260	1.290	0.130	197.000	18.960	319.000	18.750	0.047	18.000
OKCV	19.080	0.340	3.280	0.021	296.600	5.460	347.000	12.350	0.024	10.000

Table3.

Cadmium (Cd) from OKB (106.500mg/kg) was higher than the results obtained from OKC (91.500mg/kg) and OKA (87.453mg/kg).during speciation higher percentages were observed at the inert fraction, that is non-reactive state when Cadmium is ingested caused immediate poison and damage to the liver and kidney (Rahimzadeh *et al.*, 2017).Cadmium from OKB should be avoided because of the higher concentration to avoid liver damage.

Copper (Cu) in the three samples ranged between 3.100-5.510mg/kg, which are significantly low and beyond the toxicity level as well as cobalt (Co). Chromium(Cr) was higher in OKA(22.980mg/kg), followed by OKC(10.560mg/kg) and least in OKB(2.900mg/kg) They are metals of no advantage or benefit to humans (Marian and Ephraim, 2009) except in industrial application in plating and coating..

Iron (Fe) is the most abundance in the three dumpsites ranging from 3690.000-6780.000mg/kg from OKA to OKC respectively in ascending order. Iron is imperative in hemoglobin formation in animal and healthy development. The hazards pose by other toxic labile metals present outweigh the advantages of the consumption of the Iron present and should be discouraged. Reverse was the values obtained for Manganese (Mn) that increased from OKC to OKA (37.450 - 210.250mg/kg) as observed in the results.

Zinc (Zn) is an essential metal for the regular performance of numerous enzyme systems. Zn shortage, mostly in children, results to loss of appetite, growth impedance, weakness, and even immobility of sexual growth (Saracoglu *et al.*, 2009).Zinc was the second abundance in the dumpsites. The results obtained from these sites were OKA (2880.00mg/kg), OKB (1217.000mg/kg) and OKC (385.000mg/kg) respectively. However, noxious labile metals in the soil should be avoided in consumption of green vegetables from the sites.

Nickel (Ni) decreased from OkA to OKC (74.150, 73.900 and 46.200) in mg/kg. According to the Environment Agency (2014), more than 30mg of Nickel may cause changes in muscle, brain, lings, liver kidney and can also cause cancer, tremor, paralysis and even death. The values obtained were higher than acceptable level for Nickel consumption in human and should also be discouraged for consumption. Arsenic was generally low in the three samples. Lead values were the same in OKA and OKC (12.000mg/kg), while OKB had 18.000mg/kg. Nontoxic limit of lead, which is 2.5 mg/kg) as reported by Sharma *et al.*, (2006) and Chirenje *et al.*, (2004) was exceeded in the three dumpsites. The labile metals were present in the inert fractions in all the analyzed soil samples in the region.

These showed that the most abundant of these metals were in forms that were not desirable and not useful in human diet if consumed. Though, the absorption rate in the green vegetables as indicated in Table 3 were not up to 50% of the total concentrations in the soil, but the concentration observed in the vegetables for human consumption were exceeded with higher value observed in the OKA and OKB. According to World Health Organization (2019), all metalloid elements are poisonous compounds in which some of the labile metals belong like arsenic since as explained by World Health Organization, 2019 till date.

CONCLUSION

The human body requires a numeral of minerals in order to maintain good health, but not all are essential as some are labile metals which are sinister to human health.Soils are the major sink for labile metals released into the environment by anthropogenic activities and unlike organic contaminants which are oxidized to carbon (IV) oxide by microbial action. Most labile metals do not undergo microbial or chemical degradation, which are less toxic or biodegradable and most of these metals exist in the inert fractions in most of the soil examined and persist in the environment. Labile metals constitute an ill-defined group of inorganic chemical hazards, and those most commonly found at contaminated sites are cadmium (Cd), copper (Cu),chromium (Cr),cobalt (CO), zinc (Zn), nickel (Ni),arsenic (As), and lead (Pb). Most of these metals were fund in the residual fraction contained higher concentrations in all the sites irrespective of plant absorption, though higher values were observed in iron (Fe) and Zinc (Zn).The results obtained from the three dumpsites analyzed indicated that the concentrations of labile metals from these sites were undesirable for human utilization except iron which is required for hemoglobin formation in mammals. The concentrations of the most labile metals in soil samples analyzed were higher in the inert fractions after speciation. This showed that they abundantly reside in the soil and detrimental to human health if consumed. The most threatening were dumpsites from waste management (OKA) and Igodan (OKB), which some of the labile metals aforementioned for their toxicity were accumulated. Though, Plants from the three dumpsites should be discouraged for human consumption because their toxicity level was exceeded. This will avert the menace which the presence of these labile metals will cause to man, animals, plants and their environment.The tolerable protection and restoration of soil ecosystems contaminated by labile metals require regular characterization and remediation. This will provide adequate environmental protection and public health awareness, at both national and international levels.

AUTHOR CONTRIBUTIONS

G. Aladekoyi performed the literature review, experimental design, analyzed and interpreted the data, prepared the manuscript text, and manuscript edition. A. Akinnusotu developed the study methodology that also comprised preparing a checklist that was used in data collection. He further interpreted and analyzed the data. G. Aladekoyi and A. Akinnusotu also performed the experiments and literature review, compiled the data and manuscript preparation. Both authors edited the paper to ensure completeness and consistency with the journal's formatting guidelines.

ACKNOWLEDGEMENT

The authors would like to thank the Tertiary Education Trust Fund and The Director of The Research Unit of Rufus Giwa Polytechnic, Owo, Ondo State, Nigeria for granting the permit to undertake the study. Appreciation also goes to the Rector, Rufus Giwa Polytechnic, Owo, Ondo State who provided clearance for collecting data. Special acknowledgement correspondingly goes to Mr. Ogunmola, a native of Ilutuntun in Okitipupa Local Govt. Area who provided the survey maps that were used to determine the spatial extent of conformity with the approved land subdivision regulations.

CONFLICT OF INTREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript. In addition, the ethical issues; including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, redundancy has been completely observed by the authors

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HOW TO CITE THIS ARTICLE

Aladekoyi, G; Akinnusotu, A., (2021). Labile metal evaluation, speciation and accumulation in harvested plant from urban major dumpsites. Int. J. Hum. Capital Urban Manage., 6(2): 185-192.

DOI: 10.22034/IJHCUM.2021.02.07

url: http://www.ijhcum.net/article_46947.html