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Bioavailability of heavy metal load in soil, groundwater, and food crops *manihot esculenta* and *carica papaya* in dumpsite environment

O. M. Omorogieva¹ · J. A. Tonjoh¹Received: 3 January 2020 / Revised: 7 June 2020 / Accepted: 11 June 2020
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Abstract

The need for consciousness of the impact of heavy metal on crop production and human health risk through policy formulation necessitated the study. Screening of the total heavy metal iron (Fe), nickel (Ni), cadmium (Cd), lead (Pb), chromium (Cr), and copper (Cu) concentration in soils collected at random at a depth of 0–30 cm within and around dumpsite environment in Ikhueniro, Iguomo, and Okhuahe suburban communities, Benin City, Nigeria was carried out. Cassava tubers, Pawpaw pods (*manihot esculenta* and *carica papaya*) cultivated and groundwater within and around the dumpsite environment were also tested for the selected heavy metal. The study employed integrated scientific methods in actualizing its aim. The results show that mean values of 520.97 ± 1.66 mg/kg Fe, 39.07 ± 0.33 mg/kg Ni, 1.41 ± 0.10 mg/kg Cd, 1.92 ± 0.00 mg/kg Pb, 1.24 ± 0.02 mg/kg Cr, and 24.97 ± 5.16 mg/kg Cu were recorded for soil. On the other hand, 127.00 ± 2.01 : 22.36 ± 1.20 mg/kg Fe and 5.70 ± 2.11 : 0.98 ± 1.11 mg/kg, respectively, amongst other metals measured in cassava and pawpaw. The results were compared to Standard Organization of Nigeria (SON), National Food and Drug Administration Control (NAFDAC), and Food and Agricultural Organisation/World Health Organisation (FAO/WHO) standards; it was observed that Fe, Ni, and Pb exceeded the threshold values for metal concentration in soil and drinking water. Policy formulation restricting farming and drilling of boreholes within the locus of dumpsite environment is advocated to fostering the attainment of green environment for sustainable development and public consciousness of the detrimental effects of heavy metals on human health.

Keywords Policy formulation · Crop cultivation · Ikhueniro · Screening and public consciousness

Introduction

It is a common phenomenon to cultivate food crops within and around dumpsite environment, because the soil is assumed to be rich in organic manure needed for plant growth. However, studies have shown that soils in dumpsite environment are reservoir for heavy metals (Ogunbanjo et al. 2016; Xaypanya et al. 2018). Heavy metals are not biodegradable once absorbed and accumulated in plants and human tissues (Dinake et al. 2018; Ogbonna et al. 2020). Solid waste dumpsite as a crude and age long method of waste disposal particularly in developing countries is one of

the main sources of heavy metal due to the different types of waste and their contents usually dumped in it (Imasuen and Omorogieva 2013b, 2015; Yang et al. 2019). Open dumpsite method of waste disposal is not safe, because it lacks engineering properties and is exposed directly to the atmosphere. Consequently, leachate, heavy metal, and other deleterious contaminants emanating from the spoilt heap at the dumpsite can escape and contaminate surface and groundwater as well as absorbed by plant roots (Godwin and Oghenekohwiroro 2016; Omorogieva 2018; Sagbara et al. 2020). Through food chain, animals, particularly humans, become a victim of the laden contaminants either in food crops or water consumed by man (Selinus 2013). To convince and discourage users of dumpsite soils for crops cultivation, it is necessary to screened soils within and around dumpsite for total heavy metal concentration to know the various forms in which the concentration is mobile and available in soil for plant growth. This will assist in advising government, policy maker, and the general public on the potential dangers associated with heavy metal load in plants

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 O. M. Omorogieva
osakpolor.omorogieva@physci.uniben.edu

¹ Department of Marine Geology, Faculty of Marine Environmental Management, Nigeria Maritime University, Okerenkoko, Delta State, Nigeria



and human tissues as well as the steps needed to address the ugly situation, although metals are essential commodity needed by plants and animals for proper functioning, but become detrimental to plant growth and human health at extremely low and high doses respectively (Wasserman 2008, Mishra 2009, Taylor et al. 2010, Omorogieva 2018, Ogbonna et al. 2020). Generally speaking, assessment of the total concentration of heavy metal load in the soil is not adequate for policy formulation and decision-making, because knowledge of the total content of heavy metals present in the soil provides limited information about their potential behavior and bioavailability (Asagba et al. 2007; Kalis et al. 2007; Yanshan and Xin, 2011; Ogunbanjo et al. 2016). Screening of the total concentration of heavy metal gives detailed information necessary for decision-making and policy formulation to salvaging the erroneous belief of dumpsite soils as source of organic manure for plant yield as well as creating public awareness on its impact on animals particularly human that feeds on plants through food chain. Against this backdrop, the research was necessitated to screen the total concentration Fe, Ni, Cd, Pb, Cr, and Cu for their bioavailable forms in soil with a view of policy formulation, to fast tracking environmental culprit and create public awareness of the dangers pose by heavy metal dose in crops cultivated within and around dumpsite environment which are eventually eaten by man. In addition to the mechanism of mobility, spatial resolution, bioavailability, chemical transformation, as well as health impact on human through food chain, the study of speciation also helps to understand the most effective ways of remediation, environmental management strategies, development of predictive models, and risk assessment (Sparks, 2003 in Asagba et al. 2007, Selinus et al. 2013, Ogunbanjo et al. 2016, Song et al. 2019). The selection of these metals in the study area is based on their prevalence and high concentration often recorded in soils within and round dumpsite environment (Oviasogie and Ukpebor 2003; Mor et al. 2005; Imasuen and Omorogieva 2013a, b; Chibuike and Obiora 2014; Omorogieva 2016). The specific objectives of the study are to determine total concentration of the selected heavy metals in soil and food crops like *manihot esculenta* and *carica papaya* being the commonest food crops cultivated in the study area; assess groundwater data for heavy metal as well as to evaluate the physicochemical properties of the soil in relation to heavy metal presence. Integrated scientific approaches including Atomic Absorption Spectrometer (AAS), XRF, modify BCR sequential extraction method was applied in the study. The modify BCR sequential extraction method was adopted for speciation, because is one of the most effective and widely acceptable techniques in speciation studies in addition to the environment of study, nature of soil and the level of contamination (Tessier et al. 1979; Lo and Yang, 1998; Asagba et al 2007; Zimmerman and Weindorf 2010; Sungur et al.

2014). The overall outcome of the study is targeted towards food and groundwater safety and environmental management through policy formulation which would eventually foster sustainable environment for sustainable development as well as public awareness of the devastating effects of extreme dose of heavy metal in food crops and water consumed by man.

Materials and methods

Site description

The dumpsite measured approximately 250 by 500 m is government approved dumpsite sited at Ikhueni, a nodal and sub urban community within Benin metropolitan city, Nigeria and its surrounding communities of Okhuahe and Iguomo (Fig. 1). It lies on 5°44'N and 7°37'E, and 5°44 and 6°43'E, and share common boundaries with neighbouring states of Kogi, Ondo, Delta, and Anambra (Seibert 2000; Omorogieva 2018). The area covers a land mass of about 2033 km² with a population estimate of 121,749 and a population density of 59.89 km² according to Edo State Bureau of Statistics (2013), National Population Commission (2006), respectively. The topography of the terrain is characterized by gentle slope plunging towards the south eastern portion of the study area and gradually increases in steepness towards Okhuahe community (Omorogieva 2016). The area experiences tropical climatic weather condition characterized by heavy rainfall and sunshine with an annual rainfall of 1898.0 mm having its peak in the month of July and September, respectively (Omorogieva 2018). The soil is loamy sand texture which favours agricultural activities like Cassava and Pawpaw production. The inhabitants depend largely on groundwater from borehole and crop cultivation as their means for sustenance.

Samples' collection, preparation, and analysis

Soil

Six composite samples each were collected in the three study sites, namely, Ikhueni, Okhuahe, and Iguomo at a depth of 0–30 cm with the aid of soil auger (Fig. 1). The samples were thoroughly mixed and air dried within the sample collection bag at room temperature to avoid external influence. Each soil sample was pulverized using agate mortar and pestle, and sieved through 2.0 mm sieve. The pH value was determined by weighing 10 g of the prepared sample into 50 mL beaker containing 10 mL of distilled water. The mixture was intermittently stirred with a glass rod for 30 min.



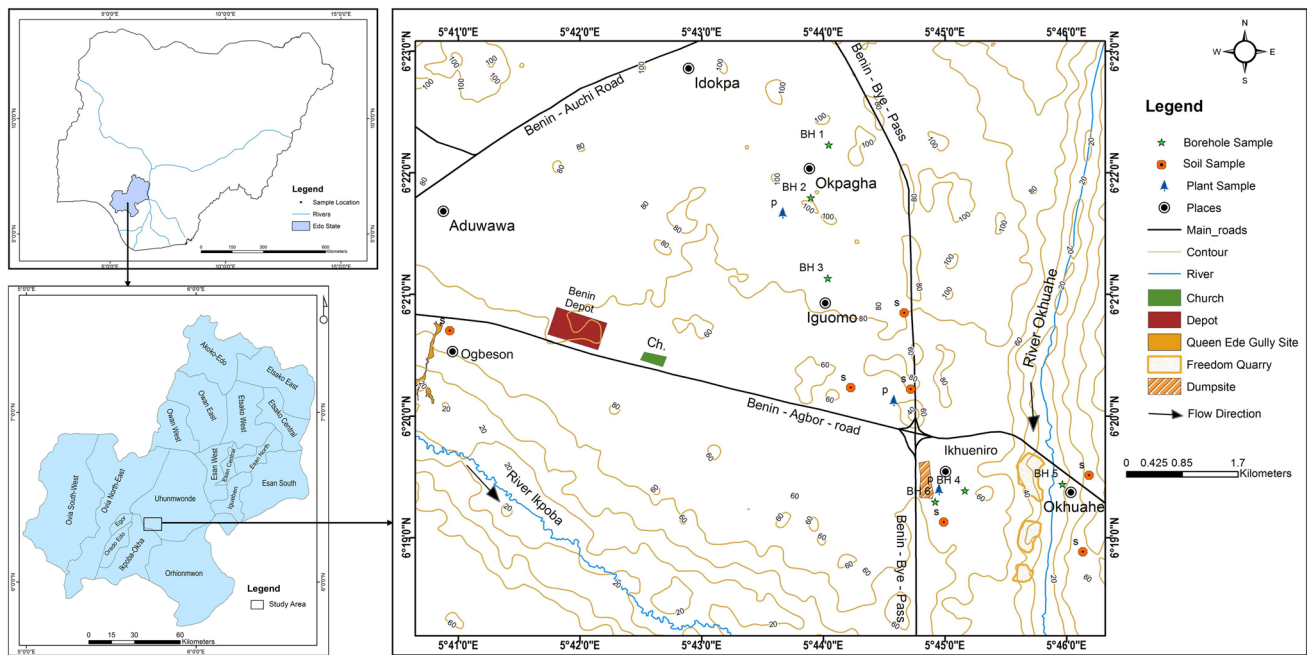


Fig. 1 Study site indicating sampling points

The pH meter earlier calibrated with buffer 4 and 7 solutions, respectively, was set appropriately at control pH 7.0 and read in a standardized electronic pH meter. The physical and chemical properties of the soils were determined according to standard methods, while the determination of particle size followed the procedure of Asagba et al. (2007) and Omorogieva (2018) with slight modification. Fifty one (51 g) of prepared sample was weighed into a 250 mL beaker containing 50 mL of calgon solution (Sodium Carbonate + Sodium Hexametaphosphate). One hundred (100 mL) of distilled water was added and stirred vigorously for 1 minute using glass rod and allowed to stand for 30 minutes. The suspension was transferred into a mechanical mixer and mixed for 15 min. The suspension was transferred into a sedimentation cylinder and makes up to mark of 1 L (1000 mL) with distilled water. The mixture was further mixed by several vertical movement of a plunger that was placed in the cylinder at a time. The first reading (R_1) was taken after 40 s by sliding the hydrometer slowly into the suspension and the temperature of the suspension was noted. The second reading (R_2) was taken after 2 h. The formulae below were used in calculating the percentages of sand, silt, and clay. Corrected HR = 40 secs.HR $\pm$ 0.3 (T – 20 °C)/W $\times$ 100/1. % Sand + Silt + Clay = 100%, % Silt = 100 – % Silt + Clay, and % Silt + Clay = Corrected HR/W $\times$ 100.

Ammonium saturated method after Donald and Quirine (2011) with a slight modification was used to determine the Cation-Exchange Capacity (CEC) which include Potassium ion (K^+), Calcium ion (Ca^{2+}), Magnesium ion (Mg^{2+}), and Sodium ion (Na^+). The procedure includes

measuring 10 g of prepared soil sample in 250 mL conical flask containing 100 mL of 1 M ammonium acetate solution (1 M NH_4OAc), and the mixture was shook with the aid of a mechanical shaker for 8 h and thereafter filtered through Whatman filter paper into a 100 mL volumetric flask and made up to 100 mL mark with distilled water. The mixture was stored in a 100 mL plastic bottle. Potassium ion (K^+) and Na^+ were read in a Jenny model flame photometer, while Ca^{2+} and Mg^{2+} were read in a Unicam series 969 model of AAS according to American Public Health Association (APHA 2005), Adeleke and Abegunde (2011). The results of pH, particle-size distribution and Cation-Exchange Capacity for the study areas are presented in Table 1. Total heavy metal concentrations in soils were determined by X-ray fluorescence method at the Geochemical Laboratory, University of New South Wales, Australia.

Table 1 pH, particle-size distribution, and CEC of investigated soils

Location	pH	% Sand	% Clay	% Silt	CEC (cmol/kg)
Ikhueniro	6.79	70.1	18.82	11.08	2.52
Iguomo	5.52	65	25	10	1.93
Okhuahe	6.50	85.01	9	5.99	2.48
Mean	6.27	73.37	17.61	9.02	2.37



Soil speciation

Bureau of Community Reference (BCR) Sequential Extraction Procedures after Asagba et al. (2007) and Ali et al. (2014) with a slight modification was carefully followed in this experiment.

Soluble fraction: One (1) gram (g) of the prepared soil sample was measured into a centrifuge tube containing 40 mL of 1 M concentration of acetic acid solution. The mixture was thoroughly mixed with a mechanical shaker for sixteen (16) hours. This was done to enhance the separation of the soil from solid phase into liquid phase. The mixture was filtered through Whatman filter paper; 20 mL of distilled water was used to wash the solid phase in the centrifuge followed by shaken of the mixture for 15 min. The mixture was further centrifuge for 20 min to facilitate the discharge into the liquid phase.

Exchangeable fraction: In the exchangeable fraction, 10 mL of 1 M $MgCl_2$ was added to the residue and agitated for 10 h in mechanical shaker.

Organically bound fraction was determined by adding 3 mL of 0.02 M HNO_3 and 5 mL 30% diluted H_2O_2 at pH 2.0 to the residue and the mixture was heated in water bath to $85\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 2 h (hr). Additional 3 mL of 30% diluted H_2O_2 was added to the mixture and heated for 3 h.

Oxidizable fraction was determined by adding 10 mL H_2O_2 solution to the residue in the centrifuge tube and shaken for 1 h. The solution was heated at $55\text{ }^{\circ}\text{C}$ and allowed to cool. The procedure was repeated by adding 10 mL H_2O_2 and further allowed to cool. Fifty (50 mL) ammonium acetate solution was further added to the mixture and was shaken mechanically for 16 h and thereafter centrifuge for 20 min.

Residual fraction The solid phase was transferred into the centrifuge tube from the fifth step. The mixture was digested with 5 mL HF and 10 mL $HClO_4$ on a hot plate until the residue dissolved. The residue was filtered and diluted with deionised water to make up to mark of 15 mL. The results of the total heavy metal concentrations in soil, speciation, and analysis of variance as well as the percentage (%) distribution of the speciated metals are summarized in Table 2.

Food crops (*Manihot esculenta* and *carica papaya*)

Six tubers of *Manihot esculenta* (Cassava) and six pods of *carica papaya* (Pawpaw) fruits were collected at a point 50 m from the centre of the dumpsite, and another set from a pristine environment about 2 km from dumpsite

environment as control. Both crops were peeled with the aid of a kitchen knife and carefully washed with distilled water to avoid external influence; the crops were sliced and wrapped separately in a well-labeled foil paper. The prepared crop samples were oven dried at a control temperature of $50\text{ }^{\circ}\text{C}$ for about 36 h. The dried crops of both crops were crushed in an agate mortar with pestle and sieved through 2 mm sieve. One (1 g) of each processed sample was measured in a crucible and placed in muffle furnace for 4–6 h at a temperature of $600\text{ }^{\circ}\text{C}$ to allow ashing of the crops samples. The ashes were allowed to cool for 2 h before adding 20% dil. HCl. The mixture was filtered through Whatman filter paper in 120 mL plastic bottle and made up to 100 mL mark with distilled water. Concentrations of the selected heavy metals were measured with Atomic Absorption Spectrometer (AAS) model 969 Unicam series with air acetylene and the results are presented in Table 2.

Groundwater sample

Groundwater samples earlier tested for in study of Omorogieva (2016) as cited in Omorogieva and Andre-Obayanju (2020) were referenced in the current study. The results obtained for the tested groundwater from surrounding boreholes are presented in Table 3.

Statistical analysis

To aid interpretation, primary data were subjected to multi-variance statistical analysis such as analysis of variance (ANOVA), correlation, and cluster analysis. Software Packages for Social Sciences (SPSS) version 16 was used to determine the ANOVA, while Statistics Software Package for Education and Data Analysis (PAST) version 3.02a as applied in Hammer et al. (2001) was used for the cluster analysis. The QQ test was used for confirmation of the data normality. The result of the confirmation shows a normally distributed dataset judging from the straight line graph obtained from the test. Figure 2 represents the cluster analysis, while Table 5 represents the correlation analysis.

Results and discussion

Soil properties

Physicochemical property of soil samples measured in the study is represented in Table 1. The pH values measured range from 6.50 to 6.79 with a mean value of 6.27, indicating that the soil is slightly acidic. Soils with slightly acidic-to-moderately alkaline pH (6.0–7.5) values are excellent for



Table 2 Analysis of variance (ANOVA) of speciated soil in the study areas

Forms	Metals	Ikhueni	Iguomo	Okhuahe	Mean Conc	% Distribution
H ₂ O soluble	Fe	33.33 ± 0.18 ^a	30.50 ± 0.17 ^b	26.49 ± 0.29 ^c	30.11 ± 0.21	5.78
	Ni	2.37 ± 0.16 ^a	2.54 ± 0.28 ^a	3.00 ± 0.06 ^a	2.64 ± 0.11	6.76
	Cd	0.04 ± 0.00 ^a	0.08 ± 0.00 ^c	0.05 ± 0.00 ^b	0.06 ± 0.01	2.26
	Pb	0.08 ± 0.00 ^c	0.03 ± 0.00 ^a	0.06 ± 0.00 ^b	0.06 ± 0.01	3.13
	Cr	0.04 ± 0.00 ^c	0.03 ± 0.00 ^a	0.03 ± 0.00 ^b	0.03 ± 0.00	2.42
	Cu	1.41 ± 0.30 ^a	3.27 ± 0.00 ^b	3.35 ± 0.00 ^b	2.68 ± 1.12	10.73
Exchangeable	Fe	39.43 ± 0.02 ^c	26.30 ± 0.17 ^a	32.67 ± 0.00 ^b	32.8 ± 2.05	6.30
	Ni	4.25 ± 0.02 ^c	3.33 ± 0.17 ^a	3.72 ± 0.01 ^b	3.77 ± 0.05	9.6
	Cd	0.08 ± 0.00 ^c	0.05 ± 0.00 ^b	0.04 ± 0.00 ^a	0.06 ± 0.01	4.26
	Pb	0.32 ± 0.01 ^c	0.02 ± 0.00 ^a	0.28 ± 0.01 ^b	0.20 ± 0.01	10.42
	Cr	0.13 ± 0.00 ^a	0.17 ± 0.04 ^a	0.31 ± 0.01 ^b	0.20 ± 0.01	16.13
	Cu	2.32 ± 0.01 ^a	2.67 ± 0.33 ^a	3.10 ± 0.06 ^b	2.69 ± 0.03	10.77
Org. bound (carbonate)	Fe	55.67 ± 0.06 ^a	40.67 ± 0.18 ^a	53.57 ± 0.32 ^b	49.97 ± 2.05	9.59
	Ni	4.35 ± 0.18 ^a	5.27 ± 0.15 ^b	4.23 ± 0.02 ^a	4.61 ± 0.02	11.79
	Cd	0.12 ± 0.00 ^c	0.10 ± 0.00 ^b	0.08 ± 0.00 ^a	0.10 ± 0.01	7.09
	Pb	0.65 ± 0.00 ^c	0.43 ± 0.02 ^b	0.32 ± 0.00 ^a	0.46 ± 0.01	23.95
	Cr	0.40 ± 0.00 ^a	0.51 ± 0.01 ^c	0.48 ± 0.00 ^b	0.46 ± 0.01	37.09
	Cu	2.55 ± 0.01 ^a	3.08 ± 0.02 ^b	4.61 ± 0.00 ^b	3.41 ± 0.00	13.65
Oxidisable	Fe	36.33 ± 0.17 ^a	20.72 ± 0.00 ^b	21.15 ± 8.93 ^b	26.06 ± 1.01	5.00
	Ni	2.15 ± 0.09 ^a	2.24 ± 0.14 ^a	3.00 ± 0.06 ^a	2.46 ± 0.03	6.29
	Cd	0.03 ± 0.00 ^a	0.05 ± 0.00 ^a	0.04 ± 0.02 ^b	0.04 ± 0.01	2.83
	Pb	0.21 ± 0.00 ^a	0.36 ± 0.21 ^a	0.29 ± 0.00 ^a	0.28 ± 0.01	15.10
	Cr	0.12 ± 0.00 ^a	0.13 ± 0.01 ^b	0.16 ± 0.00 ^c	0.13 ± 0.01	10.48
	Cu	1.67 ± 0.29 ^a	2.02 ± 0.00 ^b	2.87 ± 0.41 ^b	2.18 ± 0.11	8.73
Reducible	Fe	53.33 ± 0.17 ^b	45.12 ± 0.10 ^a	56.44 ± 0.25 ^c	51.63 ± 0.04	9.91
	Ni	3.20 ± 0.12 ^a	2.95 ± 0.19 ^a	3.10 ± 0.03 ^a	3.08 ± 1.07	7.88
	Cd	0.06 ± 0.00 ^a	0.07 ± 0.00 ^a	0.09 ± 0.01 ^b	0.07 ± 0.01	4.96
	Pb	0.50 ± 0.00 ^a	0.40 ± 0.00 ^a	0.46 ± 0.00 ^a	0.45 ± 1.00	23.43
	Cr	0.32 ± 0.00 ^c	0.27 ± 0.00 ^b	0.30 ± 0.00 ^b	0.29 ± 0.01	23.38
	Cu	2.44 ± 0.25 ^a	4.00 ± 0.00 ^b	5.47 ± 0.27 ^c	3.97 ± 0.05	15.89
Residual	Fe	318.00 ± 0.09 ^a	379.00 ± 0.13 ^b	250.00 ± 0.35 ^c	315.66 ± 0.06	60.59
	Ni	33.48 ± 0.27 ^a	14.07 ± 0.04 ^b	15.43 ± 0.25 ^c	20.99 ± 2.01	53.72
	Cd	0.72 ± 0.00 ^a	1.17 ± 0.00 ^b	1.36 ± 0.00 ^b	1.08 ± 0.00	76.59
	Pb	0.08 ± 0.00 ^a	0.67 ± 0.00 ^a	0.43 ± 0.00 ^a	0.39 ± 0.01	20.31
	Cr	0.17 ± 0.07 ^a	0.09 ± 0.00 ^a	0.10 ± 0.00 ^a	0.12 ± 1.03	9.67
	Cu	15.36 ± 0.21 ^a	17.38 ± 0.22 ^b	2.60 ± 0.35 ^c	11.78 ± 0.11	47.17
Total	Fe	536.40 ± 0.00 ^a	524.10 ± 0.00 ^b	502.40 ± 4.97 ^c	520.96 ± 0.25	33.33
	Ni	54.24 ± 0.18 ^a	30.53 ± 0.31 ^b	32.44 ± 0.25 ^c	39.07 ± 2.01	33.33
	Cd	1.05 ± 0.00 ^a	1.52 ± 0.30 ^b	1.67 ± 0.00 ^a	1.41 ± 0.21	33.25
	Pb	1.84 ± 0.00 ^a	2.04 ± 0.00 ^b	1.87 ± 0.00 ^b	1.92 ± 0.11	33.39
	Cr	1.12 ± 0.07 ^a	1.22 ± 0.00 ^b	1.38 ± 0.00 ^b	1.24 ± 0.05	33.33
	Cu	25.87 ± 0.50 ^a	32.70 ± 0.00 ^b	16.35 ± 14.97 ^c	24.97 ± 3.01	33.32

Means ± SD of the same row with dissimilar superscript lowercase alphabet (a, b, or c) are significantly different, while those with similar superscript lowercase alphabet (aa, bb, or cc) are not significantly different at $P < 0.05$

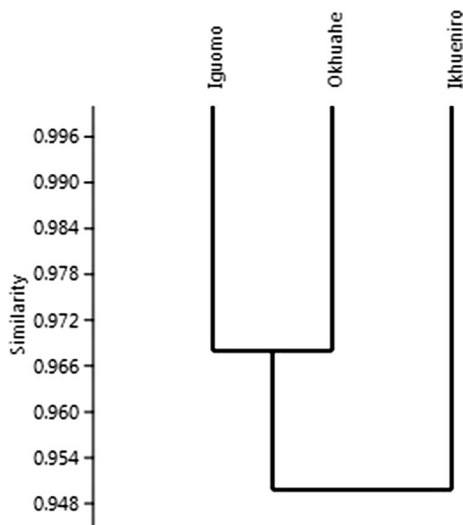
SEM Standard minimum error to declare significance and it is calculated as $\sqrt{(EMS/nr)}$ where EMS is the error mean square and nr is the number of variables



Table 3 Concentration of heavy metals in Cassava tuber and Pawpaw fruit (mg/kg)

Element	Cassava (<i>Manihot esculenta</i>)	Pawpaw (<i>Carica papaya</i>)	CP
Fe	127.00 ± 2.01	22.36 ± 1.20	7.09 ± 0.13
Ni	20.00 ± 1.10	5.22 ± 1.50	1.98 ± 0.01
Cd	ND	1.00 ± 0.01	0.50 ± 0.17
Pb	5.70 ± 2.11	0.98 ± 1.11	0.65 ± 0.45
Cr	2.00 ± 0.00	0.50 ± 0.27	0.10 ± 2.21
Cu	21.00 ± 1.31	6.56 ± 0.11	4.98 ± 0.01

ND not detected

**Fig. 2** Cluster analysis for the studied areas

the cultivation of crops like Cassava and Pawpaw (Jaoun 2002). Soil pH influences the availability of essential nutrient for plant growth; hence, soils with extreme pH values do not often support plant cultivation except in rare cases like blueberries crop which grow well in soil with moderately acidic pH (4.5–5.5). Cation-exchange capacity (CEC) which is the ability of soil to hold exchangeable cations (K^+ , Na^+ , Ca^{2+} , and Mg^{2+}) and other soil nutrients required for plant growth was relatively high across the study areas. The CEC recorded in the study ranged from 1.93 cmol/kg in Iguomo to 2.52 cmol/kg in Ikhueni with a mean value of 2.37 cmol/kg. Ikhueni has a higher value of CEC compared to Okhuahe and Iguomo. This may account for the favourable condition for the cultivation of food crops like cassava (*manihot esculenta*) and pawpaw (*carica papaya*), because soil with high percentage of clay and organic matter has the ability to attract more exchangeable ions which are positive in nature (McKenzie et al. 2004, Hazelton and Murphy 2007; Brown and Lemon 2018). The dumpsite activities,

which not limited to dumping of sewage, garden trim, poultry dung, municipal solid waste (MSW), spent oils and other petroleum wastes, could also be a contributing factor considering the value of clay and silt measured in the soil (Omorigieva 2018). Soil particle distributions in the study show a range of 65.00–85.01% with a mean value of 73.37% for sand. On the other hand, clay and silt have a range of 9.00–25.00% with a mean value of 17.61, and 5.99–11.08% with a mean value of 5.99%, respectively. Previous study showed that high percentage of sand and silt in soil impedes the growth and cultivation of crops because of their low CEC which could reduces the organic matter and the organic carbon of the soil.

Concentration and speciation of metals

Mean total concentration of heavy metal assessed in soil, bioavailable forms upon speciation, Analysis of Variance (ANOVA), and percentage distribution results are summarized in Table 2. The concentrations are recorded in mean values and are in the order of residual > organically bound (carbonate) > reducible > water soluble and exchangeable forms. The mean value of Fe in the residual form in the three study sites was found to be 315.66 ± 0.06 mg/kg (60.59%) of the total mean concentration of 520.97 ± 0.25 mg/kg (Table 2). Other heavy metals speciated in the residual forms include Ni with a mean value of 20.99 ± 0.01 mg/kg, (53.72%) of total mean concentration of 117.2 ± 0.01 mg/kg; Cd has a mean value of 1.08 ± 0.00 mg/kg (76.59%) of the total mean concentration of 4.24 ± 0.21 mg/kg; Pb has a mean value of 0.39 ± 0.21 mg/kg (20.31%) of the total concentration of 5.75 ± 0.11 mg/kg; Cr has a mean value of 0.12 ± 1.03 mg/kg (9.67%) of the total concentration of 3.72 ± 0.05 mg/kg and Cu has 11.78 ± 0.11 mg/kg (47.17%) of the total mean concentration of 74.92 ± 3.01 mg/kg. The high concentration of Fe, Ni, and Cu recorded in the residual forms may be due to the presence of acid resistance mineral and organic materials that may be present in the soil as well as the preponderance of lateritic (tropical red soil) overlying the Formation, and the coarse sandy textural characteristic of the soil in some part of the study area like Okhuahe and Iguomo (Table 1) may be responsible. Another factor may be as a result of the co-precipitation of various silica species following their adsorption into the mineral lattice as reported in the study of Manceau et al. (1996); Adano et al. (1995); Asagba et al. (2007) and Sun et al. (2018).

Previous study has shown that carbonate forms have been implicated by immobilizing most heavy metals by providing an absorbing or nucleating surface for the soil pH (Dudley et al. 1991). Asagba et al. (2007) reported 14.6% Cu and 34.0% Cd in soils around active automobile spare parts market in Benin City; a study carried out under the same environment and geological condition as the present



study. It was observed that the results obtained in the carbonate form show that Cu and Cd were higher than the values of 3.41 ± 0.00 and 0.10 ± 2.05 mg/kg recorded in current study, respectively. The differences in the results could be attributed to the direct leakages of spent petroleum products from vehicle engines as compared to the fraction that gets to the dumpsite through discarded parts as observed in the study of Imasuen and Omorogieva (2013a). Chromium and Pb in Ikhueniro were observed to be more bound in the carbonate form which could be as a result of the clay particle attached to the soil lattice, as indicated in Table 1. According to Lambert et al. (1997), carbonate is more stable in soils with moderate-to-high pH values which may be responsible for the relatively high values of Cr and Pb because of the moderate pH value of 6.27 recorded in the study. In the oxidisable form, 26.06 ± 1.01 mg/kg, 5.01% Fe, 2.46 ± 2.03 mg/kg, 6.29% Ni, and 0.04 ± 0.10 mg/kg 2.84% Cd were recorded. Others include 0.28 ± 2.20 mg/kg, 15.10% Pb, 0.13 ± 0.05 mg/kg, 10.48% Cr, and 2.18 ± 0.11 mg/kg, 8.77% Cu. In an earlier study by Jaradat et al. (2006), 13% Pb, 44% Cu and 13% Cd were found in the oxidisable form, while Asagba et al. (2007) reported 29% Pb, 24% Cd, and 22.3% Cu. The results obtained in the current study are similar to those of Asagba et al. (2007) and Jaradat et al. (2006). The reason could be as a result of the similarity in the environment of study and soil type as factor for choice of SEP which may also determine the corresponding result (Ali et al. 2014).

In the exchangeable form, Fe has the highest value of 32.80 ± 2.05 mg/kg of the total mean value of 520.97 ± 0.25 mg/kg amounting to 6.30%. Ni has a value of 3.77 ± 0.05 mg/kg (9.65%) and Cd 0.06 ± 3.01 mg/kg (4.26%) was recorded, respectively. Others include Pb (0.20 ± 2.20 mg/kg, 10.42%), Cr (0.20 ± 1.02 mg/kg, 16.13%), and Cu ($2.69 \pm$ mg/kg, 10.77%). The aqueous phase is the most available form for plant as nutrient, because the metal species are found in soluble form in the soil. This form is very important because of the high mobility of the metal species due to the availability of water as a medium of mobility (Li et al. 2001). In the current study, metals found in water soluble form consist of a small fraction of the total metals in the soil with Fe measuring 30.11 ± 0.21 mg/kg (5.78%) of the total mean value of 520.97 ± 0.25 mg/kg; Ni (2.64 ± 0.11 mg/kg, 6.76%), Cd (0.06 ± 2.01 mg/kg, 4.26%), respectively. Other metals assessed include Pb (0.06 ± 1.20 mg/kg, 3.13%), Cr (0.03 ± 0.00 mg/kg, 2.42%), and Cu (2.68 ± 1.12 mg/kg, 10.73%). Mobility and immobility of heavy metals along with their availability in soil largely depend on the type of binding forms in which the metal species occur. Water soluble, exchangeable, oxidisable, organically bound, and the reducible constitute the most available forms in soil, while the reducible constitute the less available pool. The speciation result demonstrates that

water soluble, exchangeable, reducible, organically bound, and oxidisable are the mobile fractions which are bioavailable for plant as nutrient. Heavy metals in uncontaminated soils and sediments are mainly immobile as they are bound to silicates and primary minerals. Conversely, heavy metals in contaminated soils are more mobile and bound to other phase rather than silicate minerals (Rauret, 1998, Ali et al. 2014).

Geobotanical evaluation

To assess the impacts of the heavy metal contaminant in plants and groundwater, first, geobotanical study was embarked on. It involves the assessment of the selected heavy metal in Cassava (*Manihot esculenta*) and Pawpaw (*carica papaya*) which is the commonest crops cultivated in the area. The results obtained shows that 127.00 ± 2.01 mg/kg and 22.36 ± 1.20 mg/kg of Fe were found in Cassava and Pawpaw, respectively (Table 3). Cadmium was not detected in Cassava tuber, but was detected in 1.00 ± 0.01 mg/kg in Pawpaw fruit. The results of other heavy metal measured in both crops are presented simultaneously as follows: Ni (20.00 ± 1.10 : 5.22 ± 1.50 mg/kg), Pb ($5.7.00 \pm 2.11$: 0.98 ± 1.11 mg/kg), Cr (2.00 ± 0.00 : 0.50 ± 0.27 mg/kg), and Cu (21.00 ± 1.31 : 6.56 ± 0.11 mg/kg), respectively. The values recorded for control for each of the metals Fe, Ni, Cd, Pb, Cr, and Cu were 7.09 ± 0.13 mg/kg, 1.98 ± 0.01 mg/kg, 0.50 ± 0.17 mg/kg, 0.65 ± 0.45 mg/kg, 0.1 ± 2.21 mg/kg, and 4.98 ± 0.01 mg/kg, as shown in Table 1. From the control results, it was observed that the dumpsite greatly influences the presence of heavy metal in the soil. The high concentration recorded in the study is capable of causing damage in plant and animal tissues when consume at high dose (Kamran et al. 2013; Toth et al. 2016). Plants grown in the Zn and Cu polluted soils have the ability to absorb metals in their roots (Stalikas et al. 1997). This might be responsible for the high values of metal concentration recorded in Cassava and Pawpaw fruits grown around the dumpsite environment. Imasuen and Omorogieva (2013a) reported that Cassava and Plantain grown around contaminated site such as dumpsite and automobile workshop are contaminated with heavy metal such as Pb, Cd, Ni, and Cr, which is in support of the current study. Nickel, Pb, Cr and Cu were observed to be more concentrated in Cassava than the bioavailable forms in soil. This could be as a result of the affinity of Cassava in adsorbing nutrient from the soil as previously reported by Stalikas et al. (1997). The guideline set by National Food Drug and Administration Control (NAFDAC), Food and Agricultural Organisation/World Health Organisation (FAO/WHO), Ministry of Environment Finland, and the European Commission/CODEX was referenced in this study for the purpose of policy formulation (Table 6). The results



Table 4 Physicochemical characteristics of groundwater from surrounding boreholes in the study area (Omorogieva and Andre-Obayanju 2020 in Omorogieva 2016)

S/n	Parameters	Units	GW1	GW2	GW3	GW4	GW5	RW	WHO (2011)	SON (2006)
1	Temp	⁰ C	28	27.5	27	26	28.07	28	20	20
2	pH		6.8	6.33	5.71	5.78	6.2	6.11	6.9	6.5
3	Pb	mg/L	0.04	0.03	0.01	0.001	0.05	0.07	0.01	0.01
4	Cr	mg/L	ND	0.03	0.12	ND	ND	0.02	0.05	0.05
5	Cu	mg/L	1.2	ND	ND	0.001	ND	0.001	2	1
6	Zn	mg/L	5		0.56	0.38	0.15	0.21	3	5
7	Cd	mg/L	ND	ND	ND	ND	ND	0.001	0.003	0.003
8	Ni	mg/L	0.64	0.05	0.09	0.73	0.08	0.11	NA	0.02
9	Fe	mg/L	0.9	0.2	1.43	0.38	0.3	0.43	0.3	0.3

NA Not available, ND not detected, ⁰C degree celsius, SON standard organization of nigeria

Table 5 Correlation matrix of heavy metals in soil (mg/kg)

	Fe	Ni	Cd	Pb	Cr	Cu
Fe	1					
Ni	0.8121	1				
Cd	0.8486	0.5752	1			
Pb	0.8962	0.8712	0.8941	1		
Cr	0.9702	0.9258	0.8033	0.9472	1	
Cu	0.8210	0.6055	0.9931	0.9173	0.8016	1

Bold Strongly correlated

Table 6 National and international guidelines for metals in food and vegetable

Metals (mg/kg)	Current study for Cassava	Current study for Pawpaw	WHO/FAO	NAFDAC	EC/CODEX	Normal range in plants
Cd	NA	1	1	NA	0.2	< 2.4
Cu	21	6.56	30	20	0.3	2.5
Pb	5.7	0.98	2	2	0.3	0.50–30
Zn	ND	NA	60	50	< 50	20–100
Fe	127	22.36	48	NA	NA	400–500
Ni	20.00	5.22	NA	NA	NA	0.02–50
Cr	2.00	0.50	NA	NA	NA	NA

Modify after FAO/WHO (1976) cited in Paluwa et al. (2012)

NA = Not Available, ND = Not Detected

recorded in the current study show that metal concentration in the bioavailable forms (water soluble, reducible, oxidisable, organic bound, and exchangeable phases) of Fe, Pb, Cr, Ni, Cu, and Cd in Cassava and Pawpaw assessed was either equivalent the tolerable limits or far exceeds the tolerable limit of 2.00 mg/kg and 48 mg/kg for Cassava as in the case of Pb and Fe with a value of 5.70 ± 2.11 mg/kg and 127.00 ± 2.01 mg/kg, respectively (Table 6).

Groundwater evaluation

To further buttress the claim of the dumpsite as the source of contaminants and impact on the geoenvironment, data

set of groundwater study culled from Omorogieva (2016) and cited in Omorogieva and Andre-Obayanju (2020) was adopted and compared to established standard by the Standard Organization of Nigeria (SON 2006) and WHO (2011). The results obtained showed that groundwater data from boreholes within and around the dumpsite environment-tagged GW1–GW5 and a river water from nearby river-tagged RW earlier presented in Table 4 were under serious threat of Fe, Pb, and Cr and, consequently, not fit for drinking unless appropriate treatment is administered. Accumulation of heavy metal in human system is detrimental to human health; for example, Ni which is available in soil through the application of fertilizer,



burning of fossil fuel sewage sludge, and polymer wastes can cause respiratory failure, cancer, birth defects, and blood pressure at high dose (Dai et al. 2017). Additionally, Pb, Cr, and Fe sourced from solid and industrial waste activities, weathering processes, burning, and vehicular emissions has also been reported to cause respiratory tract defeat (asthma cough short breath), sperm damage, cancer, pregnancy and respiratory disorder, brain damage, cardiovascular disease, as well as gene mutations. Iron overload can also result in severe disease such as fatigue, weight lost, joint pains, heart, and liver failure according to Agency for Toxic substance and Disease Registry (ATSDR 2012), Hime et al. (2015), Isley et al. (2017), and Sagbara et al. (2020). Furthermore, it was evidence from the control results recorded for food crops (Cassava and Pawpaw) that dumpsite soils are repository of deleterious contaminants (Table 3).

Correlation coefficient of the soil matrix in Table 5 showed a range of $R^2 = (0.57-0.99)$ at two-tailed; 95% confident level revealed that metal assessed were strongly correlated indicating a similar source of origin. Cadmium and Cu have the highest correlation coefficient of **0.9931**, followed by Cr and Fe with coefficient value of **0.9702**. The strong positive relationship between the heavy metals in soil and the food crops studied couple with the clusters analyses further confirmed the dumpsite as the main source of the heavy metal contamination in the soil, plants, and groundwater resources, as presented in Table 5 and Fig. 2, respectively. ANOVA results earlier presented in Table 2 also show that concentrations of the selected heavy metals are significantly different from one community to another which further buttresses the claim.

Contamination and/or pollution associated with deleterious metals like Pb, Cr, Cd, Ni, and Fe among others as a result of environmental mismanagement is a serious global concern, because they are none biodegradable, highly toxic to plants and animals when accumulated in their tissues (Afroze and Sen 2018). Consequently, heavy metal removal from polluted water is a global debate by scientist due to growing population and limited fresh water resources. Currently, there are numerous methods such as chemical precipitation, the use of agricultural byproducts, chemical coagulation, ion exchange, electrochemical, the use of activated carbon, use of natural zeolite, membrane processes., and ultrafiltration in removing heavy metals from polluted water (Ince and Ince 2019; Alalwan et al. 2020). However, the most recommended techniques are based on the availability of raw materials and cost-effectiveness of the entire process. For example, the use of natural zeolite, biomass (agricultural byproducts), and activated carbon is very effective and cheap to run. Consequently, these methods are often recommended for the removal of heavy metal from polluted water.

Conclusion

Due to global increase in population, the quest for clean water and food is on the increase. As a result, it is necessary to device efficient means of managing water resources and soils impacted with heavy metals because of their detrimental impacts on human health through food chain. In the current study, it was observed that the total concentration of heavy metal assessed was in the order of $Fe > Ni > Cu > Pb > Cd > Cr$. On speciation, the forms were in the order of residual > organically bound (carbonate) > reducible > water soluble > exchangeable > oxidisable) fractions. The summation of the bioavailable forms (organically bound/carbonate, reducible, water soluble, oxidisable, and exchangeable) for plant absorption in soil as nutrient and in groundwater obtained from boreholes was significantly high. Soils impacted with heavy metals are not advisable for crops cultivation, because plant roots can absorb and accumulate heavy metal in their systems which are eventually consumed by man as source of food for survival. Since heavy metals are not biodegradable, it accumulates in both tissues of plants and animals particularly man. Excess metal load in plant and animal tissues can be detrimental to plant growth and human health respectively. In addition to accumulation in plant and animal tissues, heavy metal can leached from unprotected dumpsite and contaminate groundwater as demonstrated in this study. This can lead to environmental degradation and shortage of natural resources like land for agricultural activities and water resources for human consumption. Practice such as Innovative and cost-effective waste management integrated into the residential waste disposal system, through educational training at grassroots as well as private–public partnership are imperative to minimize heavy metals contamination of soil, food, surface, and groundwater resources. In view of the foregoing, it is advisable that open dumpsite practice should be discouraged through legislative measures and public awareness by government agencies as well as concerned organisations, especially when the dumpsite is domicile within and around human environment, because heavy metal accumulation over a considerable period of time in food crops cultivated in dumpsite environment and groundwater from nearby boreholes can significantly affect human health through food chain. It is, therefore, necessary to have thorough understanding of the bioavailable forms in which metal occurs in soil and its impact on crop production and water resources to facilitate policy formulation gear towards sustainable environment, food, and water resource safety.

This research focused on the bioavailability of toxic metal like lead, cadmium, chromium, nickel, copper, and iron in soil within and around open dumpsite environment. This is because the total concentration of metal in soil is not adequate for policy formulation and does not assist in applying the right technique for environmental management. Hence,



the need for speciation to know the fractions those metals are available in soil media for plant absorption. The research will help to advice farmers who farm close to dumpsite believing that the soil is rich in natural manure against the backdrop of laden deleterious metal. Consequently, the study will assist in adopting the best technique in environmental management as well as assisting the host community, government, and industry stakeholders in making informed evidence-based decisions with respect to public and environmental health.

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Compliance with ethical standards

Conflict of interest There is no conflict of interest in this study. Dr. O.M Omorogieva designed the research in collaboration with Prof. E.G Imeokparia and Prof. O.I Imasuen. Dr. O. M Omorogieva and Mr. A.J Tujoh carried out the sampling, while Prof. David Cohen and Mr. Mike Erhayimwen did the analysis. Dr. O.M Omorogieva wrote the manuscript and Mr. J.A Tonjoh did the interpretation of data set, wrote the manuscript, and accepted to publish the article in International Journal of Environmental Sciences and Technology hosted by Springer nature.

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