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Sequential extraction and bioavailability of some heavy metals on dumpsite soils in a semi-urban community

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ABSTRACT

BACKGROUND AND OBJECTIVES: Solid wastes are composed of organic and inorganic pollutants (heavy metals) that can contaminate soil, underground and surface water; resulting in serious health challenges to humans. The bioavailability of cadmium, chromium, copper, lead and nickel on dumpsite soils, were investigated during the wet and dry seasons of 2018 by a sequential extraction method (Tessier).

METHODS: The soils were sampled from eight points within each of two dumpsites in Ilisan-Remo, Ogun State. Top soil of 0–15 cm depth was sampled, air-dried and sieved. The extracted soil samples were analysed for heavy metal concentrations using flame atomic absorption spectrometer.

FINDINGS: In the dumpsite soils examined, cadmium, chromium, copper and lead were majorly bound to the residual fractions at the first and second dumpsites, respectively for both seasons with mean concentrations (mg/kg) of: Cd – 0.65 ± 0.12 and 1.20 ± 0.07 , Cr – 36.83 ± 5.70 and 26.83 ± 3.57 , Cu – 28.37 ± 3.69 and 8.04 ± 0.32 , Pb – 12.40 ± 2.34 and 14.11 ± 2.44 , but Ni was found mainly in the Fe-Mn oxide fraction during the wet season, with mean concentrations (mg/kg) of 6.22 ± 1.33 and 8.24 ± 0.78 , for dumpsites A and B, respectively. The values obtained for these metals were higher during the dry season than a wet season and there were no metals in the mobile fractions for wet season. However, cadmium and lead were found in the carbonate fraction during the dry season, resulting in their bioavailability in the soils.

CONCLUSION: The speciation results revealed that heavy metals were more bioavailable in the residual fractions. The absence of mobile fractions in the soils indicated that metals are not bioavailable for plants uptake at present condition; hence, the metals obtained are of geogenic rather than anthropogenic origin.

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INTRODUCTION

One of the biggest challenges faced by cities (both small and large) in developing countries is managing solid wastes (Guerrero *et al.*, 2013). Unfortunately, increase in population, urbanization, flourishing economy, and higher standard of living make the amount of solid wastes being generated to skyrocket in these countries (Minghua *et al.*, 2009). Solid wastes are substances which are non-fluid, and are apparently useless with no instant economic use; whether as a raw material, end product, expired products, or remnants, and certainly must be discarded (Eni *et al.*, 2011; Longe and Balogun, 2010). Anthropogenic activities like agricultural, domestic, industrial, and hospital activities, generate solid wastes. Following Udiba *et al.*, (2015) definition, wastes are grouped based on their origin (industrial, construction, and domestic, commercial or institutional), their contents (metal, plastic, organic substances, glass and paper etc.) or their plausible hazards (flammable, radioactive, toxic, non-toxic and infectious). Solid wastes management occurs through several methods. In major cities of Nigeria, solid wastes disposal takes place on landfills and open dumpsites. Wastes dumped at these dumpsites usually biodegrade and generate some leachates with time, which become the source of water and soil pollutions (Agbor *et al.*, 2013). Municipal wastes compose of a heterogeneous mixture of materials like metals, plastic, textiles, and so on. Meanwhile, a serious problem posed by these municipal wastes (due to their contamination on soil and water), is that it consequently put human health and their environments at greater risk of toxicity (Drechesel and Kunze, 2001). Solid wastes comprise organic and inorganic pollutants, which contaminate soil and water (underground and surface), leading to various health challenges (Srivastava, 2014). Metal toxicity depends on its mobility, transformation, potential bioavailability as well as their specific forms or binding state. In sediment and soil, total metal concentration can be used to characterize pollution intensity. However, heavy metal speciation with selective agents for extraction provides information about the key reactions controlling the metal behaviour in different matrices, and this helps to check the environmental impact of polluted sediment and soil (Ogunfowokan *et al.*, 2013; Salomons *et al.*, 1995). More so, speciation gives vital

and accurate information on how metals are mobile and bioavailable for plant metabolism (Nagajyoti *et al.*, 2010; Shuman, 1991). Mobility is the capability of metals to flow within fluids after decomposition. Prediction about quantitative element mobility is quite difficult in the environment. Mobility is considered empirically by making comparison among elemental behaviors, within changing environmental conditions. Chemical fractionation is largely influenced by several factors like pH, redox state, and so on. Thus, sequential extraction of metals helps to differentiate the relationship of the individual metal with the soil matrix; to establish metal bioavailability and its potential threat to the environment. Bioavailability is the tendency of metals to be soluble, and it increases in the order: residual < organic < Fe-Mn oxide < carbonate < exchangeable < water soluble (Ma and Rao, 1997). The fraction of elements from an ingested material like food, soil or water, which are absorbed by organisms, is termed as bioavailable fraction (Ng *et al.*, 1998). Bioavailability and mobility of metals rely on the various forms in which the metals are linked with the soil (Jaradat and Momani, 1998). One approach in determining the bioavailability and the potential danger is through sequential extraction of metals into different fractions, in order to estimate the labile fractions in atmospheric particulate matter, soils and sediments (Filgueiras *et al.*, 2002; Gleyzes *et al.*, 2002; Ross, 1994; Smichowski *et al.*, 2005; Tack and Verloo, 1995). One of the most important aspects of health risk assessment programs is the bioavailability of metals from sites that have been contaminated (Ng *et al.*, 1998). Trace element species are well understood in terms of distribution of metals, their bioavailability in soil, accumulation of these metals by organisms, and their toxic effects to humans (Bernhard *et al.*, 1986). Consequently, identifying speciation of elements for proper understanding of environmental processes is quite imperative. Soils contain different components of solid and soluble substances, which are organic and inorganic in nature (Xian, 1989). Thus, in order to gain better understanding on the chemical behavior of potentially harmful heavy metals in the environment, it is imperative to assess their bioavailability and mobility (Ng *et al.*, 1998). Several research studies have reported the total metal concentration, toxicity, mobility, and bioavailability of heavy metals in dumpsite soils from

several cities in Nigeria (Ogunfowokan *et al.*, 2013; Osakwe *et al.*, 2012; Yusuf, 2007). However, there is little or no data about bioavailability of heavy metals, through speciation on dumpsites soils in small towns like Ilisan Remo in Ogun State. Therefore, the aim of this study is to extract species of heavy metals using Tessier's extraction method. It further focuses on how to identify the bioavailability of these metals in major dumpsite soils. We carried out the study during the wet and dry seasons of 2018, in Ilisan Remo, Ogun State, Nigeria.

MATERIALS AND METHODS

Study area

Ilisan Remo is a settlement situated in Ikenne Local Government Area of Ogun State, South-Western, Nigeria. It is located within Latitude $6^{\circ} 54' 0''$ N to $6^{\circ} 57' 0''$ N and Longitude $3^{\circ} 42' 0''$ E to $3^{\circ} 39' 0''$ E (Fig. 1), in the rain forest climatic region of the country. Two major dumpsites and one control site (which was about 1 km from the dumpsites, with very minimal anthropological activities) within the town were used for the study. The first dumpsite (tagged dumpsite A)

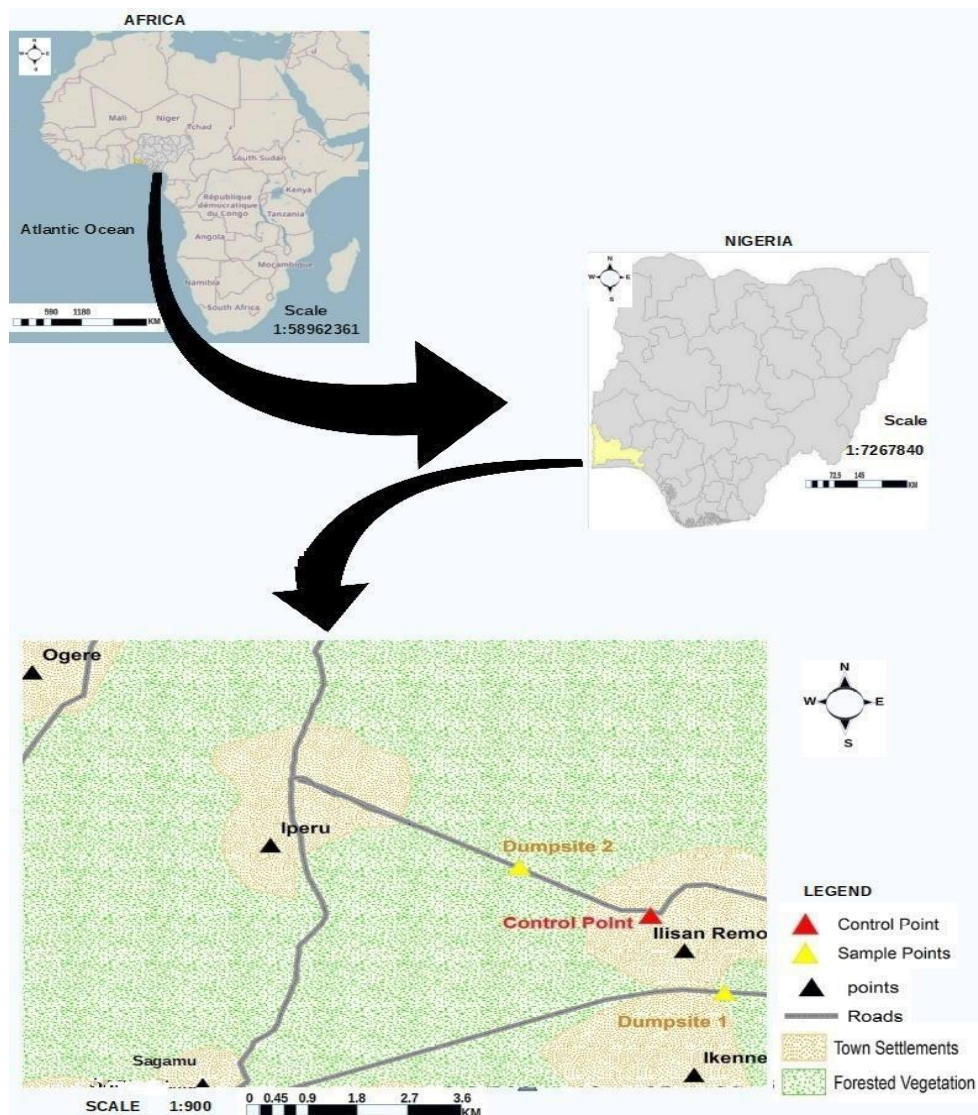


Fig. 1: Geographic location of the study area in Ogun State, South-Western, Nigeria

is located along old Ilisan-Ikenne road, whereas the second dumpsite (tagged dumpsite B) is located at Ilisan-Iperu road.

Sample Collection

Soils were sampled during the dry and wet seasons between August and December, 2018 from two major dumpsites and one control site. They were sampled from the top soil (0 – 15 cm depth) using a soil auger from eight points on each sampling site, with composite and representative samples obtained using the quartering method.

Determination of physicochemical properties of dumpsite soils

pH

Following Folsom method, the pH of soil was ascertained using water (Folsom, 1981). One gram representative soil sample was dissolved in 20 mL of distilled water. The mixture was stirred continuously for 30 minutes with a glass rod and the pH reading was taken. Calibration of pH meter was carried out with buffer 4, 7 and 9 solutions. The electrode was cleaned with distilled water and was used to take the pH reading, by immersing into the soil-water mixture. The pH was obtained as pH (H₂O).

Total organic carbon

The rapid wet oxidation procedure was used in determining the total organic carbon (Walkley and Black, 1934). Half gram of the soil was measured into a 250 mL beaker; 10 mL of 1 M K₂Cr₂O₇ solution was added and shaken carefully to properly moisten the sample. Twenty milliliters of concentrated H₂SO₄ was added and the whole mixture was left to cool for 30 minutes. One hundred milliliters of distilled water was thereafter added, then few drops of the Ferroin indicator and the mixture was titrated against 0.5 M FeSO₄ solution till a wine-red endpoint was obtained. A blank titration was carried out similarly. The percentage organic carbon was calculated using Eq. 1 and the percentage organic matter was further extrapolated with Eq. 2.

$$\% \text{ Organic carbon} = \frac{(B-T) \times 0.5 \times 0.003 \times 1.33 \times 100}{\text{Weight of sample}} \quad (1)$$

$$\% \text{ Organic matter} = \% \text{ Organic carbon} \times 1.729 \quad (2)$$

Where, B = titer value for blank, T = titer value for

sample, F = factor of correction = 1.33, and 0.5 M is of the ferrous sulphate.

Total nitrogen

The total nitrogen was measured using Macro-kjeldahl method (Black, 1965). Half gram soil sample weighed into a kjeldahl flask, 10 mL of concentrated H₂SO₄ and a selenium tablet were added and the mixture was heated for about 2 h until the solution became clear. The digest was distilled into a 2% boric acid solution containing a mixed indicator. Ammonia was trapped out of the whole digest by using 20 mL of 40% NaOH solution. The liberated ammonia was released into boric acid in receiver flask, which turns light green. Distillation continued until about 50 mL of distillate was collected into receiver flask. The solution obtained from the distillation was titrated against 0.1 M HCl until the initial color (pink) was detected at the end point.

Particle size determination

Particle size of the soil sample was estimated using the Hydrometer method and further classified using the Textural Triangular Diagram (Bouyoucos, 1962). Fifty grams of soil sample was weighed and transferred to a dispersing cup, then 100 mL of 5% dispersing solution was added and mixed for 60 s. The suspension was carefully transferred to a 1000 mL graduated cylinder, filled to mark with deionized water and the suspension was left to stand overnight to equilibrate. For 40 s reading, the hydrometer was inserted and the reading was taken after 40 s to measure the amount of silt (Eq. 3) plus clay suspended. After 6 h, the reading was taken again to know the amount of clay (Eq. 4) in the suspension. Finally the percentage of sand was determined using Eq. 5.

$$\% \text{ silt} = \frac{\text{corrected hydrometer reading at 40 s} \times 100}{\text{Weight of sample}} \quad (3)$$

$$\% \text{ clay} = \frac{\text{corrected hydrometer reading at 6 h} \times 100}{\text{Weight of sample}} \quad (4)$$

$$\% \text{ sand} = 100\% - (\% \text{ silt} + \% \text{ clay}) \quad (5)$$

Sequential extraction

Metal extractions were carried out using the method of Ma and Rao (1997), which is a modified

Tessier method (Tessier *et al.*, 1979) for speciation studies. For the sequential extraction processes, 1 g of the sieved soil samples was used. The top soils of the dumpsites for dry and wet seasons were investigated using a total of eight representative samples.

Water soluble fraction

Ten milliliters deionized water was poured in a test tube with 1 g of air-dried soil sample, and was mixed continuously for 1 h using a mechanical shaker. After an hour of shaking, it was taken through centrifugation, and the supernatant obtained was filtered (using Whatman filter paper No 42) into a 25 mL volumetric flask, and filled up to mark using distilled deionized water before heavy metal analysis.

Exchangeable fraction

Eight milliliters of 1 M MgCl₂ at pH 7 was used to leach the residue from soluble fraction (Fraction 1) at room temperature with continuous shaking for 1 h. The resulting solution was centrifuged at 4000 g for 10 mins and the supernatant (fraction 2) was carefully filtered (using Whatman filter paper No 42) into 25 mL volumetric flask, and made up to mark with distilled deionized water for metal analysis.

Carbonate fraction

The exchangeable fraction residue was further extracted to obtain carbonate fraction (fraction 3) using 8 mL of 1 M NaOAc solution of pH 5.0 with CH₃COOH at 96 °C and continuously agitated for 1 h, before centrifuging at 4000 g for 10 min. The supernatant was filtered (using Whatman filter paper No 42) and filled up to 25 mL mark with distilled deionized water before metal analysis.

Fe-Mn oxides (reducible) fraction

Fraction 3 residue was carefully extracted using 20 mL of 0.04 M NH₂OH.HCl in 25% (v/v) acetic acid at 96 °C with intermittent shaking for 5 h before being centrifuged at 4000 g for 10 min. The supernatant was filtered (using Whatman filter paper No 42) and made up to 25 mL mark using distilled deionized water, before being analysed for metal concentrations.

Organic matter (oxidisable) fraction

To the residues from Reducible fractions, 3 mL of 0.02 M HNO₃ and 5.0 mL of 30% H₂O₂ (adjusted to pH 2 with HNO₃) were added before heating

at 85 °C for 2 h, with intermittent shaking. Then, another 3 mL portion of 30% H₂O₂ was added to the resulting mixture, and maintained at 85 °C for 3 h with occasional shaking. On cooling, 5 mL of 3.2 M NH₄OAc was added to the sample, diluted with distilled deionized water to 20 mL and shaken ceaselessly for half an hour. Ammonium acetate was added to prevent the metals extracted from adhering into the oxidized sediment. The supernatant was filtered (using Whatman filter paper No 42) into 25 mL standard flask and filled up to mark with distilled deionized water, before metal analysis.

Residual fraction

Forty milliliters of aqua regia was used in digesting the residues from oxidisable fraction. The digested residue was allowed to cool, filtered (using Whatman filter paper No 42) into a 25 mL measuring flask, and filled to mark with distilled deionized water.

Besides, blanks were also prepared for the different fractions and were taken through similar protocol as carried out on the soil samples.

Analysis of heavy metals

Pure analytical grade reagents were used in this study and were checked for possible trace metal contamination. All glassware for metal analysis had been previously soaked in 10% HNO₃ (v/v) for 24 h to remove all embedded metals, and then rinsed with distilled deionized water before use.

The sequentially extracted soil samples were analysed for heavy metal concentrations with the use of air/acetylene flame atomic absorption spectrophotometer (210 VGP model, East Norwalk, Connecticut, USA) at the Department of Agronomy, University of Ibadan, Nigeria.

Quality control was done by running the analyses in triplicate, with blanks also processed for each of the sequential steps, analysed for heavy metals and used for calculation of metal contents. The percentage recoveries of the metals in the sequential extraction methods were between 90 and 102%. The parameters of operation for the heavy metals were set according to manufacturer's recommendations. Analyses of metal concentrations in the fractions and blank digests were carried out at specific wavelengths: Cd – 228.9 nm, Cr – 357.9 nm, Cu – 324.8 nm, Pb – 283.3 nm and Ni – 341.5 nm.

Table 1: Physicochemical parameters of dumpsite soils for wet season

Sites	WET			
	pH	TN (%)	OC (%)	OM (%)
Dumpsite A	6.60 ± 0.14 ^a	0.42 ± 0.03 ^a	1.65 ± 0.04 ^b	2.85 ± 0.02 ^b
Dumpsite B	5.95 ± 0.21 ^b	0.33 ± 0.03 ^b	1.92 ± 0.11 ^a	3.32 ± 0.19 ^a
Control	6.30 ± 0.10 ^b	0.24 ± 0.02 ^c	1.62 ± 0.28 ^b	2.80 ± 0.05 ^b

NB: values are shown as mean ± Standard Deviation (SD); n=2 and values with different letters on the same column are significantly different at $p < 0.05$
 TN- Total Nitrogen, OC- Organic Carbon, OM- Organic Matter, Dumpsite A- old Ilisan- Ikenne road, Dumpsite B- Ilisan-Iperu road

Table 2: Physicochemical parameters of dumpsite soils for dry season

Sites	DRY			
	pH	TN (%)	OC (%)	OM (%)
Dumpsite A	7.35 ± 0.21 ^a	0.29 ± 0.02 ^a	2.34 ± 0.13 ^b	4.06 ± 0.11 ^b
Dumpsite B	7.50 ± 0.12 ^a	0.24 ± 0.03 ^a	2.73 ± 0.08 ^a	4.72 ± 0.14 ^a
Control	7.60 ± 0.10 ^a	0.18 ± 0.01 ^b	2.29 ± 0.13 ^b	3.97 ± 0.24 ^b

NB: values are shown as mean ± Standard Deviation (SD); n=2 and values with different letters on the same column are significantly different at $p < 0.05$
 TN – Total Nitrogen, OC – Organic Carbon, OM – Organic Matter, Dumpsite A – old Ilisan-Ikenne road, Dumpsite B – Ilisan-Iperu road.

Statistical analysis

Data were analysed using statistical package for social sciences (SPSS) version 22.0 and were reported as mean ± standard deviation (SD). Means were compared using analysis of Variance (ANOVA) at 5% significance level, and were separated using least significant difference (LSD).

RESULTS AND DISCUSSION

Soil Characteristics

pH

Soil pH has a significant outcome on metal dynamics. This is due to the mechanism of metal retention in soil, which includes precipitation and adsorption based on hydrogen ion activity (Abdulhamid et al., 2015). The mean pH values of the dumpsites soils ranged from 5.95 ± 0.21 to 6.60 ± 0.14 for wet season and 7.35 ± 0.21 to 7.50 ± 0.12 for dry season, while the control site had a mean pH of 6.30 ± 0.10 for wet season and 7.60 ± 0.10 for dry season (Table 1 and Table 2). These values show that some of the soils were slightly acidic whereas others were slightly alkaline. The values reported from this study are within the range of those previously

reported – Yahaya et al., (2009) – 6.3-7.1, Iyaka and Kakulu (2009) – 5.0-7.5 and Parth et al., (2011) – 5.7-8.9. At low pH (acidic), metals are more soluble and bioavailable in the soil. The range of values obtained from the study site soils during wet season will favour plant uptake of heavy metals and hence there is possibility of toxicity problem (Osakwe et al., 2015). The higher pH of the study site soils, especially during the dry season, could be due to the presence of liming materials and microbial activities, which are sources of calcium carbonate (Ideriah et al., 2006; Yahaya et al., 2009).

Total nitrogen

The mean percentage (%) of total nitrogen ranged from 0.33 ± 0.03 (dumpsite B) to 0.42 ± 0.03 (dumpsite A) for wet season and 0.24 ± 0.03 to 0.29 ± 0.02 for dry season in the dumpsite soils, while the control site soil was 0.24 ± 0.02% for wet season and 0.18 ± 0.01% for dry season.

The results were similar to 0.12 – 0.21% reported by Akinbile (2012) for % N of soil samples within a landfill in Akure, Nigeria and 0.05 – 0.98% reported by Ideriah et al., (2017).

Organic carbon

The mean percentage (%) organic carbon (OC) of soil samples ranged from 1.65 ± 0.04 to 1.92 ± 0.11 for wet season and 2.34 ± 0.13 to 2.73 ± 0.08 for dry season at the dumpsites. The mean values for the control site soil had OC ranging from $1.62 \pm 0.28\%$ for wet season to $2.29 \pm 0.13\%$ for dry season. The OC values obtained were lower than 12–18% reported (for organic soils), by Troeh and Louis (2005). They were however similar to those reported by Abdulhamid *et al.*, (2015) – 0.95–2.25% and Akinnusotu and Arawande (2016) – 1.00–1.93%. On another hand, they were higher than values reported by Ideriah *et al.*, (2017) – 0.63–1.05%.

Organic matter

The organic matter (OM) value of the dumpsite soils ranged from 2.85 to 3.32% and 4.06 to 4.72% for wet and dry seasons, respectively while the control site soils had OM of 2.80% and 3.97% for wet and dry seasons, respectively. These values show that the dumpsite and control site soils had high organic matter according to the classification by Brady and Weil (2008), who classified OM level of soil into <0.4% OM as very low, 1.0 – 1.5% OM as moderate and >2.0% OM as high. The result obtained from this study could be attributed to deposition and decomposition of large amount of organic wastes and sewage sludge at the dumpsites. Organic matter is important in soil structure, water retention, formation of complexes and cation exchange (Alloway and Ayre, 1997). These values agree with those reported by Akinnusotu and Arawande (2016) – $0.69 \pm 0.01\%$ to $3.35 \pm 0.02\%$. However, they are higher than 0.025 to 0.707% reported for OM by Abdulsalam *et al.*, (2011) for soil from a dumpsite in Lokoja, Nigeria. These values are lower than those reported by Tefera *et al.*, (2018) – $13.83 \pm 0.48\%$ to $16.60 \pm 0.90\%$ and Olayinka *et al.*, (2014) – 14.78%.

Particle size of soil

The dumpsite soils particle size and texture are represented in Table 3. The soil particle size distribution were 80.8% sand, 17.2% clay, and 2% silt for the dumpsite soils and 82.8% sand, 15.2% clay and 2% silt for control site soil. The soil textural class was loamy sandy for the two dumpsite soils and the control site soil for wet and dry seasons. The particle size distribution showed that the soil had higher sand composition than clay and silt in all the sites. The low clay percentage implies a low cation exchange capacity, since clay in the soil determines its water retention capacity. More so, the volume of water is directly proportional to the pore spaces in any given soil. The sandy nature of the soil could be due to break down of humus from the soil (Tefera *et al.*, 2018). Similar soil texture (loamy sandy) and particle sizes (83% sand, 11% clay and 5% silt) have been previously reported by Obianefo *et al.*, (2017).

Metal concentration using sequential extraction

Tables 4 and 5 show the result of cadmium, chromium, copper, lead, and nickel concentrations, in each fraction of soils, for both wet and dry seasons. The total concentrations of most of the heavy metals in the soils collected from the two dumpsites were significantly higher ($p < 0.05$) than those found in the soil obtained from the control site.

Cadmium

Cadmium had a highest mean value of 3.77 ± 0.47 mg/kg (73.2% - Fig. 2) associated with the carbonate fraction of soil from dumpsite A, during dry season (Tables 4 and 5). This is in agreement with the study carried out by Yusuf (2007). The next predominant fraction of cadmium was found with Fe-Mn fraction during wet season, with concentrations of 1.72 ± 0.25 mg/kg at dumpsite A and 2.18 ± 0.32 mg/kg at dumpsite B (Table 4). On another hand, only

Table 3: Particle size distribution and textural class of studied site soils

Site	Season	% sand	% clay	% silt	Textural class
Dumpsite A	Wet	80.8	17.2	2	Loamy-sandy
Dumpsite B		80.8	17.2	2	
Control		82.8	15.2	2	
Dumpsite A	Dry	80.8	17.2	2	Loamy-sandy
Dumpsite B		80.8	17.2	2	
Control		82.8	15.2	2	

Dumpsite A – old Ilisan-Ikenne road, Dumpsite B – Ilisan-Iperu road.

Table 4: Heavy metal concentrations (mg/kg) of sequential extraction (wet season)

	Cd	Cr	Cu	Pb	Ni
SITE A					
Water soluble	ND	ND	ND	ND	ND
Exchangeable	ND	ND	ND	ND	ND
Carbonate bound	ND	ND	ND	ND	ND
Fe-Mn Oxide	1.72 ± 0.25	4.55 ± 0.85	5.36 ± 0.84	20.04 ± 1.85	6.22 ± 0.63
Organic matter bound	ND	ND	17.06 ± 1.95	8.28 ± 0.41	ND
Residual	0.65 ± 0.12	26.16 ± 1.61	3.67 ± 0.51	12.40 ± 1.34	ND
Sum of extracted metals	2.37 ± 0.37 ^b	30.71 ± 2.46 ^a	26.09 ± 3.30 ^a	40.72 ± 3.60 ^a	6.22 ± 0.63 ^b
SITE B					
Water soluble	ND	ND	ND	ND	ND
Exchangeable	ND	ND	ND	ND	ND
Carbonate bound	ND	ND	ND	ND	ND
Fe-Mn Oxide	2.18 ± 0.32	5.39 ± 0.63	6.29 ± 0.41	16.67 ± 1.96	8.24 ± 0.78
Organic matter bound	ND	ND	5.76 ± 0.57	8.28 ± 0.41	ND
Residual	1.20 ± 0.07	23.10 ± 1.97	3.00 ± 0.29	14.11 ± 1.44	ND
Sum of extracted metals	3.38 ± 0.39 ^a	28.49 ± 2.60 ^a	15.05 ± 1.27 ^b	39.06 ± 3.81 ^a	8.24 ± 0.78 ^a
CONTROL					
Water soluble	ND	ND	ND	ND	ND
Exchangeable	ND	ND	ND	ND	ND
Carbonate bound	ND	ND	ND	ND	ND
Fe-Mn Oxide	ND	4.05 ± 0.31	2.43 ± 0.38	ND	4.11 ± 0.36
Organic matter bound	ND	ND	ND	3.16 ± 0.47	ND
Residual	1.88 ± 0.14	15.80 ± 2.05	1.98 ± 0.28	11.40 ± 2.10	ND
Sum of extracted metals	1.88 ± 0.14 ^b	19.85 ± 2.36 ^b	4.41 ± 0.66 ^c	14.56 ± 2.57 ^b	4.11 ± 0.36 ^c

Values represent mean ± standard deviation (SD); ND – not detected. Values of the same total heavy metal with different superscripts are significantly different from each other at $p < 0.05$.

dumpsite A had a mean value of 1.38 ± 0.21 mg/kg cadmium associated with Fe-Mn fraction, during dry season. Percentage of cadmium in Fe-Mn fraction was in the range of 26.8 to 72.5% at both dumpsites. This was followed by the residual fraction with cadmium concentration of 0.65 ± 0.12 mg/kg at dumpsite A and 1.20 ± 0.07 mg/kg at dumpsite B. The percentage of cadmium in the residual fraction was 27.4% and 35.5% at dumpsites A and B, respectively, during wet season (Fig. 2). The result obtained in this fraction agreed with those presented by [Naji et al., \(2010\)](#) and [Anegebe et al., \(2014\)](#). Cd was not detected in water soluble and exchangeable fractions; hence it would not be bioavailable for plants uptake. The order of cadmium in the different fractions was: Fe-Mn oxide > residual for wet season and carbonate > Fe-Mn oxide for dry season.

Chromium

High level of chromium was mostly found in the residual fraction with mean values of 26.16 ± 1.61 mg/kg at dumpsite A, 23.10 ± 1.97 mg/kg at dumpsite B and 15.80 ± 2.05 mg/kg at the control site during wet season (Table 4). However, during dry season, the mean levels (mg/kg) at dumpsite A, dumpsite B and control site were 36.83 ± 2.70 , 26.83 ± 1.57 , and 14.07 ± 1.89 , respectively (Table 5). This was followed by Fe-Mn oxide fraction with concentrations (mg/kg) of 4.55 ± 0.85 , 5.39 ± 0.63 , and 4.05 ± 0.31 at dumpsite A, dumpsite B and control site, respectively during wet season only (Table 4). The result obtained in this study, was of similar trend with studies carried out by [Tukura et al., \(2007\)](#) and [Iwegbue et al., \(2009\)](#). Chromium in the residual fraction is less mobile compared with Cd and are often connected with the geogenic phase.

Table 5: Heavy metal concentrations (mg/kg) of sequential extraction (dry season)

	Cd	Cr	Cu	Pb	Ni
SITE A					
Water soluble	ND	ND	ND	ND	ND
Exchangeable	ND	ND	ND	ND	ND
Carbonate bound	3.77 ± 0.47	ND	6.35 ± 0.35	70.45 ± 4.07	ND
Fe-Mn Oxide	1.38 ± 0.21	ND	1.50 ± 0.12	30.51 ± 3.36	ND
Organic matter bound	ND	ND	28.37 ± 2.69	21.80 ± 1.89	ND
Residual	ND	36.83 ± 2.70	13.24 ± 0.98	18.20 ± 1.91	5.33 ± 0.47
Sum of extracted metals	5.15 ± 0.68 ^a	36.83 ± 2.70 ^a	49.46 ± 4.14 ^a	140.96 ± 11.23 ^a	5.33 ± 0.47 ^a
SITE B					
Water soluble	ND	ND	ND	ND	ND
Exchangeable	ND	ND	ND	ND	ND
Carbonate bound	1.86 ± 0.22	ND	11.98 ± 0.45	19.68 ± 1.69	ND
Fe-Mn Oxide	ND	ND	ND	ND	ND
Organic matter bound	ND	ND	10.72 ± 0.73	ND	ND
Residual	ND	26.83 ± 1.57	3.20 ± 0.21	64.00 ± 4.16	ND
Sum of extracted metals	1.86 ± 0.22 ^b	26.83 ± 1.57 ^b	25.90 ± 1.49 ^b	83.68 ± 5.85 ^b	ND ^b
CONTROL					
Water soluble	ND	ND	ND	ND	ND
Exchangeable	ND	ND	ND	ND	ND
Carbonate bound	0.59 ± 0.14	ND	2.23 ± 0.08	ND	ND
Fe-Mn Oxide	ND	ND	ND	ND	ND
Organic matter bound	ND	ND	1.17 ± 0.00	ND	ND
Residual	ND	14.07 ± 1.89	1.87 ± 0.11	71.82 ± 4.74	ND
Sum of extracted metals	0.59 ± 0.14 ^c	14.07 ± 1.89 ^c	5.27 ± 0.19 ^c	71.82 ± 4.74 ^b	ND ^b

Values represent mean ± standard deviation (SD); ND – not detected. Values of the same total heavy metal with different superscripts are significantly different from each other at $p < 0.05$.

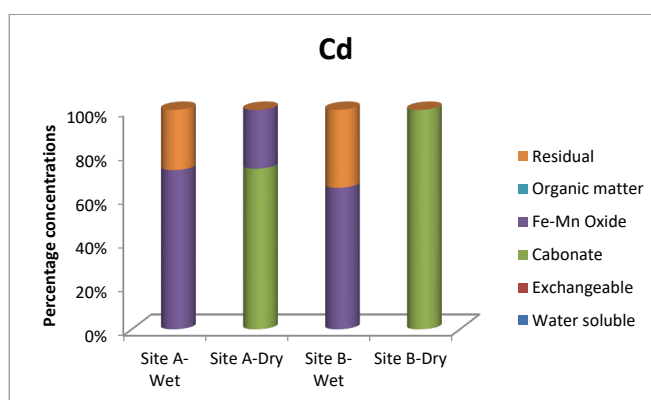


Fig. 2: Percentage concentrations of Cd in the soil Fraction for wet and dry seasons

Chromium was predominantly in the residual fraction, having a percentage of 100%, during dry season (Fig.

3). The availability of chromium is in the order of residual > Fe-Mn oxide for wet and dry seasons.

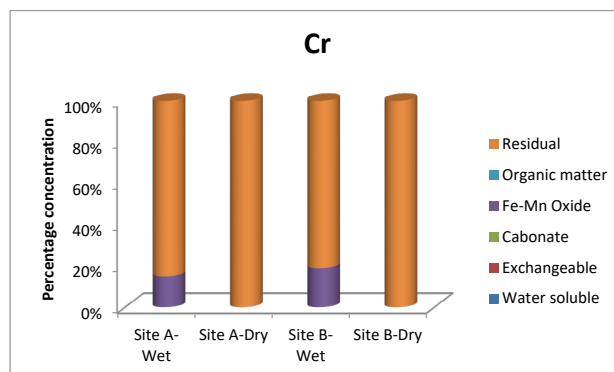


Fig. 3: Percentage concentrations of Cr in the soil for wet and dry seasons

Copper

A high level of copper was found in most of the organic matter fractions with a mean concentration of 17.06 ± 1.95 mg/kg at dumpsite A, 5.76 ± 0.57 mg/kg at dumpsite B for wet season, whereas, during dry season, copper had a concentration of 28.37 ± 2.69 mg/kg and 10.72 ± 0.73 mg/kg at dumpsite A and dumpsite B, respectively (Tables 4 and 5). These high contents of copper in organic matter fractions were also reported by Harrison *et al.* (1981), Ma and Rao (1997) and Segarra *et al.*, (2008). Copper has a high concentration owing to Cu-organic complexes being formed. Moreover, the high copper level in this fraction would bring about its immobility in soils, because copper is bound so tightly to organic matter to the extent that its bioavailability from organic soils can be very minimal (Kotoky *et al.*, 2003). Similarly, in the residual fraction, a high level of copper was obtained with concentrations (mg/kg) of 3.67 ± 0.51 , 3.00 ± 0.29 and 1.98 ± 0.28 at dumpsite A, dumpsite B and control site, respectively during wet season. And during dry season, the mean concentrations (mg/kg) were 13.24 ± 0.98 , 3.20 ± 0.21 , and 1.87 ± 0.11 at dumpsite A, dumpsite B and control site, respectively. This was comparable to those reported by Adano *et al.*, (1996) and Anegebe *et al.*, (2018). High content of copper in this fraction may be as a result of mineral and organic materials, which are acid resistant, present in the dumpsite soils. In addition, because the soil is sandy in nature, co-precipitation of copper with other silicate materials could have taken place (Manceau *et al.*, 1996; Manceau *et al.*, 1999). The content of copper in the Fe-Mn Oxide fraction was 5.36 ± 0.84 mg/kg at dumpsite A, 6.29 ± 0.41 mg/kg at dumpsite B, and 2.43 ± 0.38 mg/kg at control site for wet season, while during dry season, dumpsite A had a mean value of 1.50 ± 0.12 mg/kg. The copper levels (mg/kg) bound to carbonate were

6.35 ± 0.35 , 11.98 ± 0.45 , and 2.23 ± 0.08 at dumpsite A, dumpsite B and control site, respectively. Site A has the very high percentage of copper, 65.4% and 57.4%, in organic matter fractions for wet and dry seasons, respectively (Fig. 4).

Lead

The major fraction of lead was detected in the residual fraction with a mean value of 12.40 ± 1.34 mg/kg at dumpsite A, 14.11 ± 1.44 mg/kg at dumpsite B for wet season as well as 18.20 ± 1.91 mg/kg at dumpsite A and 64.00 ± 4.16 mg/kg at dumpsite B for dry season (Tables 4 and 5). The next was found in the carbonate fraction for dry season with mean concentration of 70.45 ± 4.07 mg/kg at dumpsite A and 19.68 ± 1.69 mg/kg at dumpsite B. Meanwhile, the high level of Pb in this fraction poses a threat to the ecosystem because carbonate dissolves readily with changes in soil characteristics like pH (Osakwe *et al.*, 2012). The next fraction with lead was Fe-Mn oxide at dumpsite A with a mean concentration of 20.04 ± 1.85 mg/kg, and 16.67 ± 1.96 mg/kg at dumpsite B for wet season, while in the dry season Pb had a concentration of 30.51 ± 3.36 mg/kg only at dumpsite A. Moreover, the association of lead and its high contents with Fe-Mn oxide is ascribed to stable complexes formation (Lopez-sandez *et al.*, 1996). A similar mean value of lead in Fe-Mn oxide fraction was previously reported (Karczewska, 1996; Ramos *et al.*, 1997). This was followed by affinity of lead with organic fraction in dry season, with a value of 21.80 ± 1.89 mg/kg at dumpsite A, and 8.28 ± 0.41 mg/kg for wet season in both dumpsite soils. This pattern of result was also reported by Tyler (1978) and Livette *et al.*, (1979). The occurrence of lead in different fractions is as follows: Fe-Mn oxide > residual > organic matter fraction for

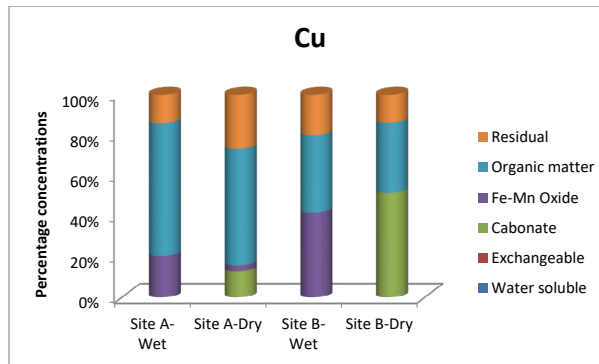


Fig. 4: Percentage concentrations of Cu in the soil for wet and dry seasons

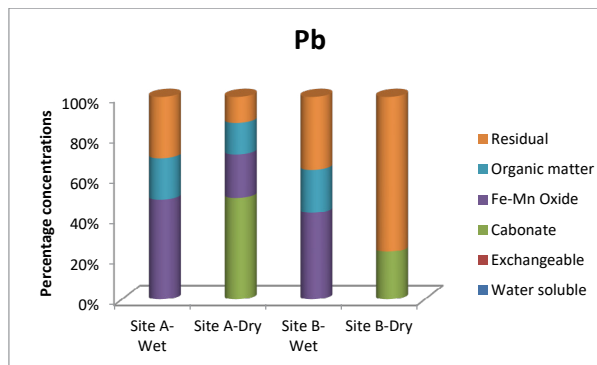


Fig. 5: Percentage concentrations of Pb in the soil for wet and dry seasons

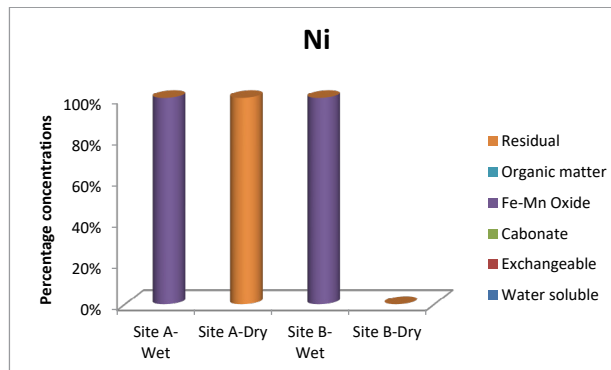


Fig. 6: Percentage concentrations of Ni in the soil for wet and dry seasons

the wet season. The levels of lead were highest in carbonate and residual fractions for dumpsite A and dumpsite B, respectively for the dry season (Fig. 5).

Nickel

Nickel was predominantly found in Fe-Mn oxide

at dumpsite A, dumpsite B, and control site with concentrations (mg/kg) of 6.22 ± 0.63 , 8.24 ± 0.78 , and 4.11 ± 0.36 , respectively for wet season and Nickel was associated with only residual fraction on dumpsite A during dry season, with mean concentration of 5.33 ± 0.47 mg/kg (Tables 4 and 5). This was agreeable with

the work reported by Olafisoye et al., (2016). Nickel was not found in other fractions and was also not found in dumpsite B, during dry season; hence Nickel is not bioavailable for plant uptake (Fig. 6).

CONCLUSION

Bioavailability of heavy metals extracted using sequential extraction was carried out in this study. The soil characteristics were also determined as studying of physicochemical properties of soil is quite crucial for management of soil and growth of plants. From this study, the results obtained show that the soils were slightly acidic (during wet season) and slightly alkaline (during dry season), loamy sandy, and were found to be higher in organic matter content. The soil acidic nature is not so good for agricultural activities; but the organic content showed clearly that it can be a good source of manure for plant cultivation. The speciation results revealed that heavy metals were found more in the residual fractions for both seasons. Metals associated with these fractions are the least extracted; less mobile and not bioavailable. The water soluble, exchangeable and carbonates which are the mobile fractions were not detected especially during wet season, hence the dumpsites do not have metals which could be bioavailable and mobile. More so, the dumpsites do not pose any environmental problem as at the time of sample collection. The high concentration and mobility of copper, cadmium and lead in dry season, could bring about their bioavailability in the soil, which could pose a risk to food chain through plants uptake. Conclusively, metals were bound mainly to the residual fraction; hence these metals are not mobile and bioavailable as at the time of sample collection. However, the dumpsites should be continuously monitored by appropriate Government agency for strict control of wastes being disposed.

AUTHORS' CONTRIBUTIONS

Oluwatosin S. Shokunbi, collected the soil samples that were mainly analyzed by her, compiled and revised the manuscript. D.O. Jegede supported during sample collection, sequential extraction and design of graphical abstract. O.O. Ajayi supervised the study and reviewed the manuscript. Olutayo S. Shokunbi assisted during sample collection, recovery experiment, and did the final review of manuscript. All authors approved the manuscript for publication.

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CONFLICT OF INTEREST

The authors declare that there is no conflict 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, and redundancies have been completely observed by the authors.

ABBREVIATIONS

$^{\circ}\text{C}$	Celsius degree
%	Percentage
ANOVA	Analysis of variance
Cd	Cadmium
CH_3COOH	Acetic acid
Cr	Chromium
Cr	Chromium
Dumpsite A	Ilisan-Ikenne road
Dumpsite B	Ilisan-Iperu road
FAAS	Flame atomic absorption spectroscopy
Fe-Mn	Iron-manganese
FeSO_4	Ferrous sulphate
Eq	Equation
Fig.	Figure
g	Gram
h	Hour
HCl	Hydrochloric acid
HNO_3	Nitric acid
H_2O_2	Hydrogen peroxide
H_2SO_4	Sulphuric acid
$\text{K}_2\text{Cr}_2\text{O}_7$	Potassium dichromate
LSD	Least significant difference
M	Molarity
MgCl_2	Magnesium chloride
mg/kg	Milligram per kilogram
mL	Milliliter
n	Number of samples
nm	Nanometer
NaOAc	Sodium acetate
NaOH	Sodium hydroxide

ND	Not detected
NH ₂ OH.HCl	Hydroxylamine hydrochloride
NH ₄ OAc	Ammonium acetate
Ni	Nickel
OC	Organic carbon
OM	Organic matter
Pb	Lead
s	Seconds
SD	Standard deviation
SPSS	Statistical package for social sciences
TN	Total nitrogen
v/v	Volume per volume

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