### SPECIATION OF SELECTED TRACE METALS IN SOIL SAMPLES FROM DUMPSITES IN LAFIA, NASARAWA STATE, NIGERIA

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Abstract: Speciation of lead (Pb), copper (Cu), zinc (Zn), manganese (Mn), iron (Fe), cadmium (Cd), and chromium (Cr) were carried out on soil samples from dumpsites in Lafia, Nasarawa State, Nigeria using Tessier et al., (1979) method. The results for various fractions obtained from the Jos road dumpsite are as follows; for samples taken from 0-5cm Jos Road, the ranges of the fractions are 0.56 to 21.56%, 0.34 to 40.66%, 1.34 to 29.18%, 0.06 to 45.91%, and 0.37 to for exchangeable, Fe-Mn oxide, organic, carbonate and residual 12.95% fractions respectively. For the samples taken from 10-15cm at Jos Road dumpsite the ranges of the fractions are 0.42 to 13.63%, 0.03 to 18.48%, 1.01 to 25.34%, 0.03 to 35.19% and 0.27 to 9.31% for exchangeable, Fe-Mn oxide, organic, carbonate and residual fractions respectively. The results of the fractions obtained from Emir Palace dumpsite are as follow. For samples taken from 0-5cm the ranges of the fractions are 0.25 to 18.34%, 2.73 to 15.58%, 4.02 to 23.28%, 0.07 to 45.25% and 1.57 to 37.43% for exchangeable, Fe-Mn oxide, organic, carbonate and residual fractions respectively. For samples taken from 10-15cm at Emir Palace dumpsite ranges of the fractions are 0.42 to 12.62%, 0.80 to 11.59%, 2.16 to 17.33%, 9.86 to 34.48%, and 0.99 to 32.99% for exchangeable, Fe-Mn oxide, organic carbonate and residual fractions respectively. The concentration of these selected metals decreases as the depth of the soil increases. All the selected metals are available in both dumpsite which shows that dumpsite pollute the soil by releasing metals into it.

Keywords: Speciation, Dumpsite, Metals and Soil

# INTRODUCTION

In recent years, concerns over the possible health and ecological effects of accumulation of heavy metals contaminants in the soil and water have prompted numerous surveys (Tovide *et al.,* 2011). The accumulation of heavy metals and metalloids in soil, water and plants especially cadmium, chromium, copper, lead, nickel and zinc poses many risks to humans and ecosystem health (Amacher, M.C 1996). One of the major challenges to human health and environmental quality is contamination of the environment by potential toxic element and this is of major concern to the environmental scientist (Olajire *et al.,* 2003, Saken *et al.,* 2010).

In particular 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 (Tokalioglu *et al.*, 2003).

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Once there is an introduction of metal contaminants into the environment, it can exist in various chemical forms and the form in which these metals exists is much more important as the degree of metal toxicity has been closely related to their chemical form (Adekola *et al.*, 2012). The occurrence of heavy metals in soil can be of geogenic or natural and anthropogenic origins. The anthropogenic sources include mining, smelting, fossil fuel combustion and various industrial activities.

Many researchers (Chandra *et al.*, 2005, Chen *et al.*, 2005, Fayun *et al.*, 2008, Olajire *et al.*, 2003) ascertained industrial activities to be the major sources of soil heavy metal contamination. Also heavy metals like iron, tin, copper, manganese and vanadium occur naturally in the environment and could serve as plant nutrient depending on their concentrations. Mercury, lead, cadmium, silver, chromium and many others that are indirectly distributed as a result of anthropogenic (human) activities could be very toxic at low concentration (Abeh *et al.*, 2007). Accumulation of trace metals especially heavy metals in the soil has a potential to restrict the soils function, cause toxicity to plants and contaminate food chain. In recent years it has also been found that heavy metals from point source and non-point sources impair water system causing lesions and deformation in fish (Henry F, 2004).

The presence of toxic metals such as lead and cadmium in the environment has been a source of worry to environmentalists, government agencies and health practitioners. This is mainly due to their health implication since they are non-essential metals of no benefit to humans (Marian and Ephraim, 2009).

Pollution of the natural environment by heavy metals is a universal problem because these metals are indestructible and most of them have toxic effect in living organisms, when permissible concentration levels are exceeded (Mwolana *et al.*, 2011). Apart from industrialization, the pollution of soil can also be from exhaust from heavy vehicles along the roadside, this has be proved by some researchers (Joshi *et al.*, 2010) found that the concentration of metals such as lead, copper, zinc and cadmium decreases rapidly within 10 to 50m from the roadside.

# MATERIAL AND METHODS

# Study Area

Lafia is the capital of Nasarawa State as indicated in Fig. 1 lies between latitude8° 30′ 21′ north and longitude 8° 30′20′ East and Nasarawa State is located centrally in the middle belt region and lies between latitude 7° 45′ and 9° 25′N of equator and between longitude 7° and 9° 37′E of the Greenwich meridian. It shares boundary with Kaduna State in the north, Plateau State in the east, Taraba and Benue State in the south, while Kogi and Federal Capital Territory flank it in the west (Marcus and Binbol, 2007).

Journal of Science and Multidisciplinary Research Volume 5, No. 2, 2013

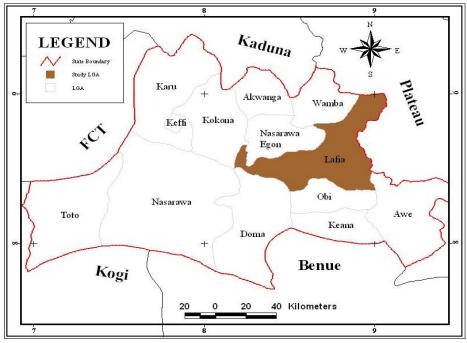


Fig. 1: Showing the Study Area

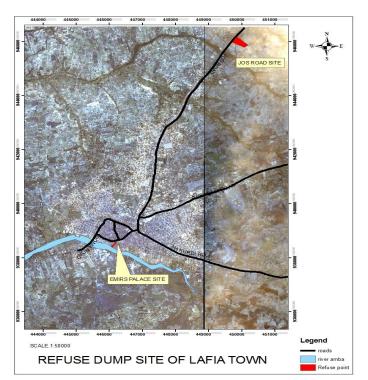


Fig. 2: Topography of Lafia Showing the Two Dumpsite Where the Samples were Taken

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Fig. 3: Emir Palace Dumpsite



Fig. 4a: Jos Road Dumpsite Front View



Fig.4b: Jos Road Dumpsite Side View

# SAMPLE COLLECTION AND ANALYSIS

Soil samples were collected from the two dumpsites indicated in figure 2 using a plastic spoon in three different locations and mixed to form composite sample at the depth of 0-5cm and 10-15cm into a polyethylene bags previously soaked in dilute nitric acid for 24hrs, rinsed with distilled water and dried. This was then transported to postgraduate chemistry laboratory and air dried for two weeks at room temperature and grinded with mortar and pestle. The grinded soil sample was sieved using 0.2mm sieve size and bottled in a polyethylene bottle for further treatment.

Tessier *et al.*, (1979) sequential method was used for the extraction except for total metal and residual fraction which aqua regia was used (Maina *et al.*, 2012) to determine the fractionation of the studied metals.

### Determination of Total Metal

This was done using 1.00g each of the dried sieved soil sample from each of the dumpsite digested with a mixture of 15ml concentrated hydrochloric acid and 25ml trioxonitrate (V) acid for 2hrs at 100°C. The mixture was filtered and made up to 50ml volume and analyzed with AAS model 7000 series east and west analytical instrument.

# SEQUENTIAL EXTRACTION

- 1. **Exchangeable Fraction**: A 1.00g triplicate dried sieved soil sample from each dumpsite was shaken with 20ml of 1M MgCl<sub>2</sub> at pH 7 for 1hr filtered and analyzed
- 2. **Carbonate Fraction:** The residue of the soil sample from fraction 1 was shaken with 20ml CH<sub>3</sub>COONa at room temperature for 5hrs at pH 5, then filtered and analyzed.

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- 3. **Fe-Mn Oxide/Reducible Fraction:** The residue of soil sample from fraction 2 was digested with a mixture of 10ml 0.04MNH<sub>2</sub>OH.HCl and 10ml of 25% CH<sub>3</sub>COOH at 96°C for 6hrs, then filtered and analyzed.
- 4. **Organic Fraction:** The residue of the soil sample from fraction 3 was transferred into 250ml beaker and 9ml of 0.02MHNO<sub>3</sub> and 15ml of 30% H<sub>2</sub>O<sub>2</sub> were added, the mixture was heated for 5hrs at 85°C on a water bath after 2hrs of heating another 15ml of 30% H<sub>2</sub>O<sub>2</sub> was added. This was then filtered and analyzed.
- 5. **Residual Fraction:** The residue from the soil sample of fraction 4 was digested with aqua Regia (7.5ml of 37% HCl and 2.5ml of HNO<sub>3</sub>) at 85°C for 1hr and then filtered and analyzed.

# **RESULTS AND DISCUSSION**

### Table 1.0: The Result of Total Metal Concentration is Shown Below in Mgkg<sup>1</sup>.

Sample	Pb	Cu	Zn	Fe	Mn	Cd	Cr
Dumpsite A	1.306 ± 0.05	0.071 ± 0.01	0.950 ± 0.05	0.730 ± 0.05	0.100 ± 0.01	0.250 ± 0.01	0.370 ± 0.02
Dumpsite B	$0.650 \pm 0.03$	0.214 ± 0.01	$3.850 \pm 0.04$	$0.320 \pm 0.01$	$0.320 \pm 0.02$	ND	0.400 ± 0.01

Note:	Dumpsite A	=	Jos Road dumpsite
	Dumpsite B	=	Emir Palace dumpsite
	ND	=	Not detected.

Dumpsite A which is the Jos road dumpsite has a higher value of lead (Pb), this can be attributed to exhaust from vehicle since the dumpsite is located by the roadside which is in agreement with Godwin, O. *et al.*, 2012. Cadmium (Cd) was not detected in Emir Palace dumpsite probably because the concentration is below the detection limit of the instrument.

Fraction	Depth	Pb	Cu	Zn	Fe	Mn	Cd	Cr
Exchangeable	0 – 5 cm	2.860 ± 0.09	0.100 ± 0.01	1.500 ± 0.07	0.078 ± 0.01	0.350 ± 0.02	0.017 ± 0.001	0.470 ± 0.15
	10- 15cm	2.460 ± 0.40	0.090 ± 0.01	1.260 ± 0.01	0.059 ± 0.02	0.280 ± 0.01	ND	ND
Fe-Mn oxide	0- 5cm	3.201±0.50	0.580 ± 0.02	2.760 ± 0.02	3.570 ± 0.06	4.134 ± 0.80	0.010 ± 0.01	0.140 ± 0.002
	10-15cm	1.28 ± 0.60	0.260 ± 0.01	2.400 ± 0.70	2.570 ± 0.04	1.879 ± 0.75	0.009 ± 0.001	ND
Organic	0 – 5 cm	3.128 ± 0.90	0.295 ± 0.05	2.940 ± 0.12	4.101 ± 0.06	0.876 ± 0.20	0.040 ± 0.01	0.440 ± 0.03
	10 – 15 cm	1.438 ± 0.67	$0.200 \pm 0.02$	2.70 ± 0.20	3.561 ± 0.08	0.719 ± 0.30	$0.030 \pm 0.01$	0.269 ± 0.01
Carbonate	0 – 5cm	1.759 ± 0.80	0.435 ± 0.07	0.646 ± 0.05	0.008 ± 0.001	0.288 ± 0.05	1.370 ± 0.04	0.368 ± 0.05
	10 – 15 cm	1.015 ± 0.50	$0.300 \pm 0.05$	0.460 ± 0.05	0.004 ± 0.001	0.261 ± 0.05	1.050 ± 0.02	ND
Residual	0 – 5a cm	0.610 ± 0.20	0.059 ± 0.02	0.061 ± 0.01	0.052 ± 0.01	0.940 ± 0.30	0.312 ± 0.02	0.281 ± 0.01
	10 – 15 cm	0.304 ± 0.05	ND	0.040 ± 0.01	0.050 ± 0.01	0.440 ± 0.12	0.146 ± 0.07	0.202 ± 0.01

Table 2a: Concentration (mgkg<sup>-</sup>) of Sequential Fraction of Metals in Dumpsite A

ND: Not Detected

Fraction	Depth	Pb	Cu	Zn	Fe	Mn	Cd	Cr		
Exchangeable	0 – 5 cm	15.84	4.31	10.16	0.56	3.44	0.57	21.66		
-	10 – 15 cm	13.36	3.88	8.53	0.42	2.75	ND	ND		
Fe – Mn oxide	0 – 5 cm	17.72	25.01	18.69	25.41	40.66	0.34	6.45		
	10 – 15cm	7.09	11.21	16.25	18.29	18.48	0.30	ND		
Organic	0 – 5cm	17.32	12.72	19.91	29.18	8.62	1.34	20.28		
	10 – 15cm	7.96	8.62	18.29	25.34	7.07	1.01	12.40		
Carbonate	0 – 5cm	9.74	18.76	4.37	0.06	2.83	45.91	17.00		
	10 – 15cm	5.62	12.94	3.12	0.03	2.57	35.19	ND		
Residual	0 – 5cm	3.38	2.54	0.41	0.37	9.25	10.46	12.95		
	10 – 15an	1.68	ND	0.27	0.36	4.33	4.89	9.31		

#### Table 2b: Percent (%) of Metal Sequential Fraction of Dumpsite A

ND: Not Detected

#### Table 3a: Concentration (MgKg<sup>1</sup>) of sequential fractionation of metal in Dumpsite B

Fraction	Depth	Pb	Cu	Zn	Fe	Mn	Cd	Cr
Exchangeable	0 – 5cn	3.21 ± 0.08	0.13 ± 0.07	10.50 ± 0.30	0.10 ± 0.06	1.32 ± 0.04	0.93 ± 0.03	0.76 ± 0.04
	10 – 15cn	$1.14 \pm 0.03$	$0.11 \pm 0.06$	7.65 ± 0.07	ND	$0.23 \pm 0.05$	$0.64 \pm 0.03$	0.72 ± 0.03
Fe–Mn oxide	0 – 5cm	$3.64 \pm 0.02$	1.62 ± 0.01	3.90 ± 0.20	3.09 ± 0.06	2.01 ± 0.09	0.79 ± 0.02	1.42 ± 0.20
	10 – 15cm	ND	1.20 ± 0.07	2.80 ± 0.10	1.08 ± 0.07	0.22 ± 0.01	ND	1.24 ± 0.60
Organic	0 – 5 cm	2.95 ± 0.06	1.04 ± 0.05	33.30 ± 0.60	4.20 ± 0.30	5.73 ± 0.07	0.32 ± 0.05	0.48 ± 0.02
Fraction	10 – 15cm	ND	$0.56 \pm 0.08$	24.80 ± 0.30	3.41 ± 0.07	3.37 ± 0.12	0.30 ± 0.01	$0.35 \pm 0.08$
Carbonate	0 – 5cm	21.20 ± 0.01	11.72 ± 0.90	30.00 ± 0.50	0.03 ± 0.01	5.49 ± 0.06	1.22 ± 0.03	2.07 ± 0.03
Fraction	10 – 15cm	18.13 ± 0.04	8.93 ± 0.12	25.95 ± 0.03	ND	3.09 ± 0.60	0.50 ± 0.07	1.99 ± 0.10
Residual	0 – 5cm	6.36 ± 0.34	$0.59 \pm 0.09$	2.25 ± 0.08	15.07 ± 0.10	$3.64 \pm 0.30$	0.32 ± 0.10	0.98 ± 0.20
Fraction	10 -15cm	$3.03 \pm 0.50$	ND	1.92 ± 0.04	13.28 ± 0.13	2.36 ± 0.20	0.05 ± 0.01	0.69 ± 0.10
ND. Net de	La sta d							

ND: Not detected

### Table 3b: Percent of Trace Metal Sequential Fraction of Dumpsite B (%)

Depth	Pb	Cu	Zn	Fe	Mn		Cr
0 – 5cn	5.40	0.50	7.34	0.25	4.81	18.34	7.10
10 – 15cn	1.92	0.42	5.35	ND	0.84	12.62	6.73
0 – 5cm	6.12	6.25	2.73	7.68	7.35	15.58	13.27
10 – 15cm	ND	4.63	1.96	2.68	0.80	ND	11.59
0 – 5 cm	5.00	4.02	23.28	10.43	20.87	6.31	4.49
10 – 15cm	ND	2.16	17.33	8.47	12.27	5.92	3.27
0 – 5cm	35.65	45.25	20.97	0.07	19.99	24.06	19.35
10 – 15cm	30.49	34.48	18.14	ND	11.25	9.86	18.60
0 – 5cm	10.70	2.28	1.57	37.43	13.26	6.31	9.16
10 -15cm	5.10	ND	1.34	32.99	8.59	0.99	6.45
	$\begin{array}{c} 10 - 15 \text{cm} \\ 0 - 5 \text{cm} \\ 10 - 15 \text{cm} \\ 0 - 5 \text{cm} \\ 10 - 15 \text{cm} \\ 0 - 5 \text{cm} \\ 10 - 15 \text{cm} \\ 0 - 5 \text{cm} \\ \end{array}$	$\begin{array}{c cccc} 0 & - 5 cn & 5.40 \\ 10 & - 15 cn & 1.92 \\ 0 & - 5 cm & 6.12 \\ 10 & - 15 cm & ND \\ 0 & - 5 cm & 5.00 \\ 10 & - 15 cm & ND \\ 0 & - 5 cm & 35.65 \\ 10 & - 15 cm & 30.49 \\ 0 & - 5 cm & 10.70 \\ \end{array}$	0 - 5cn 5.40 0.50   10 - 15cn 1.92 0.42   0 - 5cm 6.12 6.25   10 - 15cm ND 4.63   0 - 5 cm 5.00 4.02   10 - 15cm ND 2.16   0 - 5 cm 35.65 45.25   10 - 15cm 30.49 34.48   0 - 5 cm 10.70 2.28	0 - 5cn 5.40 0.50 7.34   10 - 15cn 1.92 0.42 5.35   0 - 5cm 6.12 6.25 2.73   10 - 15cm ND 4.63 1.96   0 - 5 cm 5.00 4.02 23.28   10 - 15cm ND 2.16 17.33   0 - 5 cm 35.65 45.25 20.97   10 - 15cm 30.49 34.48 18.14   0 - 5 cm 10.70 2.28 1.57	0 - 5cn 5.40 0.50 7.34 0.25   10 - 15cn 1.92 0.42 5.35 ND   0 - 5cm 6.12 6.25 2.73 7.68   10 - 15cm ND 4.63 1.96 2.68   0 - 5 cm 5.00 4.02 23.28 10.43   10 - 15cm ND 2.16 17.33 8.47   0 - 5 cm 35.65 45.25 20.97 0.07   10 - 15cm 30.49 34.48 18.14 ND   0 - 5 cm 10.70 2.28 1.57 37.43	Depth Pb Cu Zn Fe Mn   0 - 5cn 5.40 0.50 7.34 0.25 4.81   10 - 15cn 1.92 0.42 5.35 ND 0.84   0 - 5cm 6.12 6.25 2.73 7.68 7.35   10 - 15cm ND 4.63 1.96 2.68 0.80   0 - 5 cm 5.00 4.02 23.28 10.43 20.87   10 - 15cm ND 2.16 17.33 8.47 12.27   0 - 5 cm 35.65 45.25 20.97 0.07 19.99   10 - 15cm 30.49 34.48 18.14 ND 11.25   0 - 5 cm 10.70 2.28 1.57 37.43 13.26	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$

ND: Not detected

#### SPECIATION

The five sequential extraction methods (Tessier *et al.*, 1979) was used for chemical fractionation (Speciation) of the selected trace metals in the two dumpsites soil samples from Lafia, Nasarawa State and the results are shown in table 2 to 3 which is the mean values and their percentages in table 2a and 3b. One of the common trends discovered in this work was that the concentration of the various metals decreases as the depth of the soil increases. This phenomenon was observed earlier by some researchers (Adekola *et al.*, 2012, Fagbote and Olanipekum, 2010).

#### Exchangeable (MgCl<sub>2</sub>Extractable)

From Table 2 the result of exchangeable fraction from Jos road dumpsite has a higher value of lead (2.86mgkg<sup>-1</sup>), this agreed with work of other researcher (Godwin, O. *et al.*, 201). This concentration is also associated to the presence of dumpsite along the road which other

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workers reported that the concentration of metals such as Pb, Cu, Zn, and Fe increases due to exhaust from motor vehicles. The increase concentration of these Jos road dumpsite are in order Pb>Zn>Mn>Cu>Cr>Fe>Cd. As the depth increases cadmium and chromium were not detected. From Table 3 The Emir Palace dumpsite has the highest value of zinc (10.50mgkg<sup>1</sup>) followed by lead (3.21mgkg<sup>1</sup>). From these result it is obvious that shallow rooted crops can easily pick up some of these metals and accumulate in these crops since their root is mostly at the surface of the soil.

# Fe-Mn Oxide (0.04 MNH<sub>2</sub>OH.HCL + 25% CH<sub>3</sub>COOH)

Jos road dumpsite showed the higher values of Mn (4.134mgkg<sup>-1</sup>) and iron (3.570mgkg<sup>-1</sup>), this agreed with the work of Uba *et al.*, (2008) who attributed higher values to the precipitation of amorphous hydrous oxides of manganese during the aging of dumpsites (Staelens et al, 2000). For Emir Palace dumpsite the order is Zn>Pb>Fe>Mn>Cu>Cr>Cd which is also in agreement with the work of others (Fagbote and Olanipekun, 2010) who also discovered zinc to be higher in the Fe-Mn oxide fraction.

### **Organic Fraction (H<sub>2</sub>O<sub>2</sub> + HNO<sub>3</sub> Extractable)**

Jos road dumpsite showed the highest value of extractable metal to be iron (4.101mgkg<sup>1</sup>) followed by lead and zinc (3.128mgkg<sup>1</sup> and 2.940mgkg<sup>1</sup>) while Emir Palace dumpsite has the highest value to be zinc (33.30mgkg<sup>1</sup>). The order is Zn>Mn>Fe>Pb>Cu>Cr>Cd. Other workers such as Fagbote and Olanipekun (2010) obtained higher levels of manganese and zinc associated with this fraction.

### Carbonate Fraction (1M CH<sub>3</sub>COONa + CH<sub>3</sub>COOH Extractable)

Jos road dumpsite has the order of the availability of these metals as Pb>Cd>Zn>Cu>Cr>Mn>Fe and for Emir Palace dumpsite the order is Zn>Pb>Cu>Cr>Cd>Fe. Cadmium and iron have a very low values in this fraction, this agreed with the work of Abdul-Salam et al (2011) which showed only relatively lower amount of iron in this fraction.

# **Residual Fraction (Aqua Regia Extractable)**

For Jos road dumpsite, majority of the trace elements are found in this fraction and in the order of Mn>Pb>Cd>Cr>Zn>Cu>Fe. For the Emir Palace dumpsite has the highest value of iron (15.07mgkg<sup>-1</sup>) others available along with iron in the order Fe>Pb>Mn>Zn>Cr>Cu>Cd. Other workers such as (Effiong and Gilbert, 2012) also reported higher value of iron and bioavailability of all other metals in this fraction. Wufem *et al.*, (2009) also reported similar trend in the residual fraction and iron having one of the highest content.

# CONCLUSION

The concentration of these selected metal varied with the dumpsite, the primary objective of this study was to determine the chemical availability of metal in these soil from the dumpsites in Lafia, Nasarawa State. The total metal concentration of Pb, Cu, Zn, Mn, Fe, Cd and Cr were determined and are all available in Jos road and Emir Palace dumpsites exception of cadmium which was not detected in in the Emir Palace dumpsite.

Five successive selective chemical fractions were used in the analysis to determine metals easily exchangeable, associated with Fe-Mn oxide, organic matter, carbonate form and residual fractions. In both dumpsites studied the residual showed availability of all the selected metals

(Pb, Cu, Zn, Fe, Mn, Cr and Cd). It was also shown that these dumpsites contaminate soil and their concentration decrease with increasing depth down the soil.

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**Reference** to this paper should be made follows: Ibrahim, E.G. *et al.*, (2013), Speciation of Selected Trace Metals in Soil Samples from Dumpsites in Lafia, Nasarawa State, Nigeria. *J. of Sciences and Multidisciplinary Research*, Vol. 5, No. 2, Pp. 63 – 73.