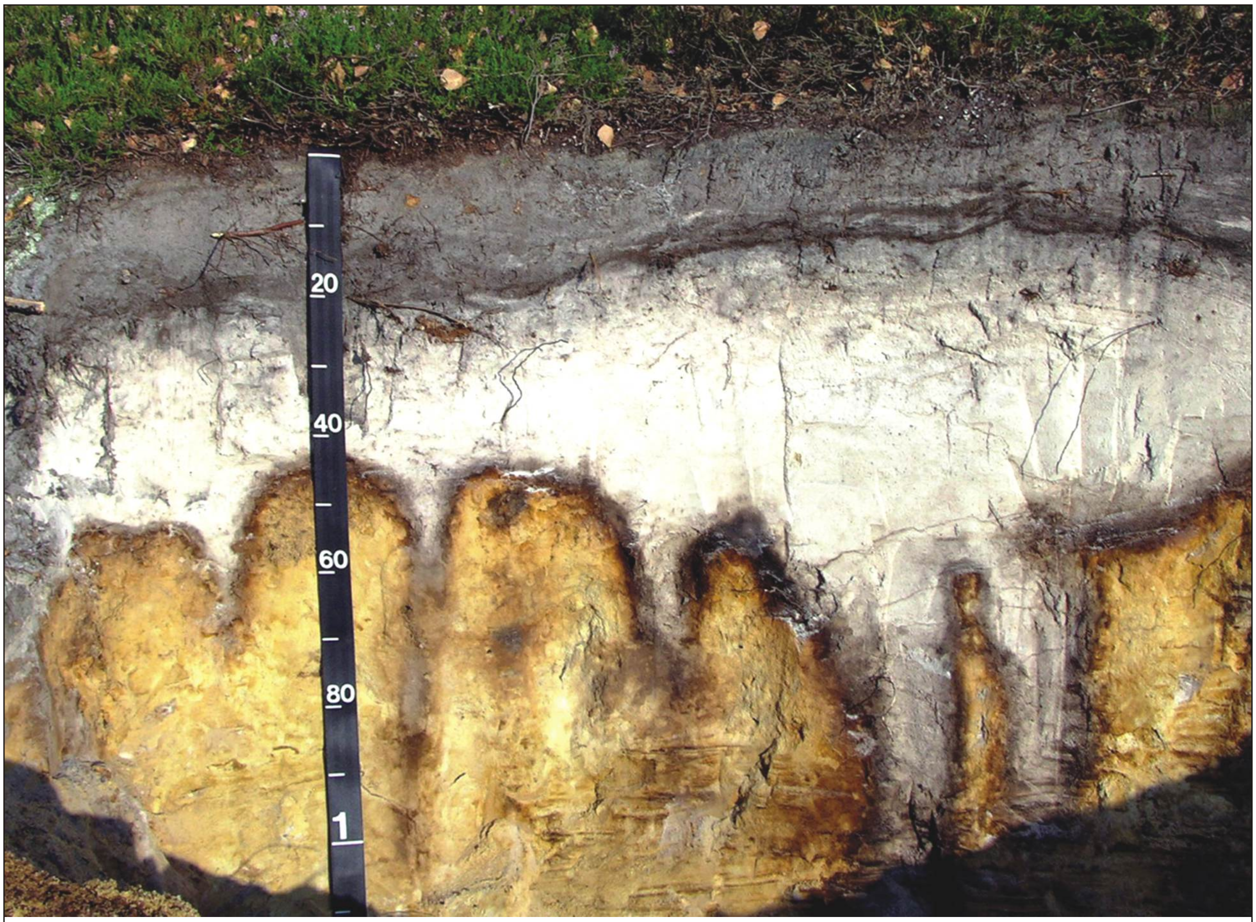

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Relation Between Aqua-regia Extractable Heavy Metals in Soil and *Manihot utilissima* Within a Municipal Dumpsite

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Abstract: The relation between aqua-regia extractable heavy metals in *Manihot utilissima* grown in a municipal dumpsite in Nigeria was investigated. Analysis has revealed significantly higher levels of Cd, Cr, Fe, Mg and Pb in the crop plant and soil from the dumpsite than values recorded fro the background soil (control). Arsenic, which was absent in the soil and plant from the background soil, was detected in soil samples from the dumpsite but not accumulated by the cassava plant cultured in the waste-dump. Heavy metals levels in soil and plants also varied with the sampling grids (locations). However, there was no clear relationship in heavy metals concentration between the cultured plants and corresponding soil from the dumpsite. Although, the levels of (Cd, Cr, Pb) detected in soil and *Manihot utilissima* are within the regulated limits, the deterioration of the dumpsite soil and environs ecoquality is desirable due to daily input of wastes and sludge. Therefore consumption of crop plants grown in dumpsites may be of serious health risk with the presence and plausible accumulation of metals such as Pb, Cr, Cd and As in the soil and plant. The frequency distribution characteristics and relative standard deviations were studied.

Key words: Heavy metals, *Manihot utilissima,* dumpsite-soil, correlation, contamination

Introduction

Heavy metals are environmental pollutants (Onyari *et al.,* 1991; Gratani *et al.,* 1992) and could be increasingly introduced anthropogenically as co-products, by-products and finished products into dumpsite soils (Shuibu and Ayodele, 2002). Several studies have been carried out on heavy metals content of soils (Yahya, 1994; Udosen *et al.,* 1990; Benson, 2004; Zauyah *et al.,* 2004). Heavy metals in soils are principally associated with geochemical cycles and biological processes, and could be greatly influenced by man-mediated activities such as industrial activities, agricultural practices and wastes disposal (Udosen *et al.,* 1990; Eja *et al.,* 2003; Benson, 2004; Zauyah *et al.,* 2004). Heavy metals in soils may be introduced anthropogenically through the use of organic and chemical pesticides and fertilizers (Zauyah *et al.,* 2004), mining and smelting of metalliferous ores, electroplating and gas exhaust (Kabata-Pendias and Pendias, 1989; Lasat, 2000) and may as well be inherited from parent materials. Heavy metals are of significant environmental concern owing to their relative toxicity and bioaccumulation potentials (Yusuf *et al.,* 2003).

Dumpsite soils are known to contain different kinds and levels of heavy metals, depending on the peculiarities of the neighbourhood (Harrison and Chirgawi, 1989; Udosen *et al.,* 1990;

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Odukoya *et al.,* 2000). Metal availability and mobility in soils also depend on soil factors such as pH, cationic exchange capacity and species of plant and rate of absorption/accumulation of toxicants (Kabata-Pendias and Pendias, 1984). Moreover, plants growing in contaminated soils can accumulate trace metals from the soil. Micieta and Murin (1998) reported that Pinus species could accumulate as much as 75% of the contaminated soil toxicants into its system. Similar observations on grasses from dumpsites have been reported (Olajire and Ayodele, 1996)

This study seeks to investigate the extent of heavy metals contamination of soils within a waste dumpsite and assess the level of metals uptake by *Manihot utilissima* a crop plant grown at the dumpsite.

Materials and Methods

Samples of *Manihot utilissima* (cassava) and culture soils were collected from a dumpsite at Uyo, Nigeria, and from a farmland, which served as the background site. The study area lies on latitude 4°59' N and longitude 7°54' E. A total of fifteen plant and culture soil samples were collected from five different sampling grids (three samples per grid) at the dumpsite, into pre-cleaned polyethylene bags, the samples were air-dried for 15 days to remove moisture and later homogenized into five composite samples.

Precisely, 0.5 g, of dried, comminuted and sieved plant and soil samples, were separately and accurately weighed, and digested in 250 mL digestion flask using 15 mL of aqua-regia, obtained by the method of Radojevic and Baskin (1999). The concentrations of aqua-regia extractable heavy metals were further released by heating and treating the filtrate with aliquots of 0.25 M HNO_3. The resultant clear solutions were transferred into 50 mL standard flasks and made up to mark with 0.25 M HNO_3. The concentrations of arsenic (As), cadmium (Cd), chromium (Cr), lead (Pb), iron (Fe) and magnesium (Mg) in the digested samples were determined using flame atomic absorption spectrophotometer Pye Unicam 939/959 model.

The average values of three determinations per sample were recorded. Pearson's correlation coefficient determined by Analyse-It statistical software was employed to establish the relationship between heavy metal concentrations in soils and *Manihot utilissima.*

Results and Discussion

The ranges of aqua-regia extractable concentration of heavy metals in soil samples from the dumpsite -soil were As, <0.001-0.003 $\mu g\ g^{-1}$; Cd, 7.15-12.00 $\mu g\ g^{-1}$; Cr, 45.11-70.18 $\mu g\ g^{-1}$; Fe, 815.00-950.10 $\mu g\ g^{-1}$; Mg, 58.18-71.19 $\mu g\ g^{-1}$; Pb, 93.50-126.05 $\mu g\ g^{-1}$. Heavy metals ranges in *Manihot utilissima* grown in the dumpsite-soil were; Cd, 0.03-0.05 $\mu g\ g^{-1}$; Cr, 3.16-5.00 $\mu g\ g^{-1}$; Fe, 630.10-742.00 $\mu g\ g^{-1}$; Mg, 1047.50 -1705.00 $\mu g\ g^{-1}$; Pb, 0.34-0.71 $\mu g\ g^{-1}$ (Table 1). Arsenic was not detected in the cultured plant despite its presence in the soil. These values were significantly higher than values recorded for the background soil and plant samples, respectively (Table 2). The enhanced levels of heavy metals in the dumpsite-soil and plants could be attributed to huge amount of domestic, commercial and industrial solid wastes of variable composition disposed of at the dumpsite. Aerial deposition of pollutants is also a likeable source.

The magnesium content in *Manihot utilissima* ranged between 1047.50 and 1705.00 $\mu g\ g^{-1}$. The plant accumulated more Mg than Cd, Cr, Fe and Pb at both the dumpsite and background soils. Magnesium is one of the macronutrients required for plant growth. The enhanced concentration found

Table 1: Concentration of heavy metals in soil and *Manihot utilissima* from dumpsite

Parameter	DS_1 (μg g^{-1})	DP_1 (μg g^{-1})	DS_2 (μg g^{-1})	DP_2 (μg g^{-1})	DS_3 (μg g^{-1})	DP_3 (μg g^{-1})	DS_4 (μg g^{-1})	DP_4 (μg g^{-1})	DS_5 (μg g^{-1})	DP_5 (μg g^{-1})
As	0.001	0.00	<0.00	0.00	0.002	0.00	0.001	0.00	0.003	0.00
Cd	10.15	0.05	8.30	0.04	9.75	0.03	12.00	0.05	7.15	0.06
Cr	69.82	3.16	63.50	5.00	70.18	4.00	50.60	3.18	45.11	3.75
Pb	126.05	0.34	95.00	0.50	93.50	0.65	101.35	0.40	120.00	0.71
Fe	910.00	714.00	815.00	672.40	930.70	686.70	950.10	630.10	818.00	742.00
Mg	65.00	1110.15	60.00	1047.50	70.35	1705.00	71.19	1084.10	58.18	1186.74

DS -Dumpsite soil, DP -*Manihot utilissima* cultured at dumpsite

Table 2: Concentration of heavy metals in soil and *Manihot utilissima* from background soil (control)

	CS_1 (ug g^{-1})	CP_1 (ug g^{-1})	CS_2 (ug g^{-1})	CP_2 (ug g^{-1})	CS_3 (ug g^{-1})	CP_3 (ug g^{-1})	CS_4 (ug g^{-1})	CP_4 (ug g^{-1})	CS_5 (ug g^{-1})	CP_5 (ug g^{-1})
As	0.00	ND	0.00	ND	0.00	ND	0.00	ND	0.00	ND
Cd	2.55	ND	3.00	ND	2.60	ND	2.65	ND	3.10	ND
Cr	26.53	1.06	25.90	1.08	27.00	1.50	28.00	1.18	25.70	1.30
Pb	5.63	0.86	6.18	0.93	5.62	0.73	6.35	0.88	6.00	0.70
Fe	350.00	650.00	510.15	615.10	430.00	700.00	370.00	595.00	495.00	618.00
Mg	35.50	785.00	40.10	690.00	40.10	815.18	35.30	711.50	36.00	765.00

CS -Control soil CP -*Manihot utilissima* cultured at background soil

Table 3: Statistical data on heavy metals in soil and *M. utilissima* from the dumpsite environment. Concentrations in μg g^{-1}

Variable	As (DS)	As (DP)	Cd (DS)	Cd (DP)	Cr (DS)	Cr (DP)	Pb (DS)	Pb (DP)	Fe (DS)	Fe (DP)	Mg (DS)	Mg (DP)
No. of samples	5	5	5.00	5.000	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
Arithmetic mean	0.001	0.00	9.47	0.046	59.84	3.82	107.18	0.52	884.60	689.04	64.94	1226.69
Median	0.001	-	9.75	0.050	63.50	3.75	101.35	0.50	910.00	686.70	65.00	1110.10
SD	0.0011	-	1.85	0.011	11.43	0.75	14.92	0.16	63.76	42.38	5.88	272.22
SE	0.0005	-	0.83	0.005	5.11	0.34	6.67	0.07	28.52	18.95	2.63	121.74
Minimum	0.001	-	7.15	0.030	45.11	3.16	95.00	0.34	815.00	630.10	58.18	1047.50
Maximum	0.003	-	12.00	0.060	70.18	5.00	126.05	0.71	950.10	742.00	71.19	1705.00
Range	0.003	-	4.85	0.030	25.07	1.84	32.55	0.37	135.00	112.00	13.01	657.50
95% CI of Mean	0.000	-	7.17	0.032	45.65	2.88	88.66	0.32	805.43	636.42	57.64	888.69
	0.003		11.77	0.060	74.03	4.75	125.70	0.72	963.77	741.66	72.25	1564.69
IQR	0.001	-	1.85	0.010	19.22	0.82	25	0.25	112	41.60	10.35	102.64
C.V.%	81	-	20	25	19	20	14	30	7	6	9	22

DS -Dumpsite soil DP -*Manihot utilissima* cultured at dumpsite

Table 4: Kurtosis, skewness, percentile and results of Kolmogorov-Smirnov test (K-SP) on metal concentrations in dumpsite soil and *M. utilissima*

Heavy metal		K-SP coefficient	Kurtosis	Skewness	K-S probability	Percentiles 25th	50th	75th
Percentiles								
As	DS	0.62	-0.18	0.40	>0.15	0.001	0.001	0.002
	DP	-	-	-	-	-	-	-
Cd	DS	0.42	-0.32	0.16	>0.15	8.30	9.75	10.15
	DP	0.62	-0.40	-0.18	>0.15	0.04	0.05	0.05
Cr	DS	0.58	-2.43	-0.51	>0.15	50.60	63.50	69.82
	DP	0.53	0.91	1.08	>0.15	3.18	3.75	4.00
Pb	DS	0.66	-2.65	0.54	>0.15	95.00	101.35	120.00
	DP	0.51	-2.33	0.16	>0.15	0.40	0.50	0.65
Fe	DS	0.66	-3.00	-0.39	>0.15	818.00	910.00	930.00
	DP	0.38	-0.09	-0.25	>0.15	672.40	686.70	714.00
Mg	DS	0.58	-2.71	-0.06	>0.15	60.00	65.00	70.35
	DP	0.93	4.29	2.05	0.04	1084.10	1110.10	1186.74

in *Manihot utilissima* is however, not high enough to cause phytotoxicity. Plant toxicity usually occurs at Mg levels above 7000 mg kg^{-1} (Alloway, 1995) and the ability of the crop plant to utilize Mg for growth may also serve as a regulatory physiological mechanism against excess Mg accumulation in the plant tissue.

Iron concentration in the dumpsite-soil (884.76 $\mu g\ g^{-1}$) was higher than the values recorded for the background soil (431.03 $\mu g\ g^{-1}$) (Table 3). Iron concentration in the *M. utilissima* samples cultured in dumpsite-soil were generally high and ranged between 630.10 and 742.00 $\mu g\ g^{-1}$. The mean concentrations of Cd, Cr and Pb were within the recommended concentration range in plants, 0.1-2.4, 0.03-14 and 0.2 -20 mg kg^{-1}, respectively (Alloway, 1995; Radojevic and Baskin, 1999). The presence and detection of these trace elements in the crop plant may be attributed to possible absorption from soil solution through the roots of *M. utilissima*.

The mean concentrations of heavy metals in the dumpsite-soil were $1.4x10^{-3}$, 9.47, 59.84, 107.18, 884.60 and 64.94 $\mu g\ g^{-1}$ for As, Cd, Cr, Pb, Fe and Mg, respectively (Table 3). However, arsenic was not detected in the background soil and plant samples. The mean concentrations of As, Cr and Pb were within the normal range in soils while Cd concentration exceeded both the normal (0.01-2.0 $\mu g\ g^{-1}$) and critical (3.0-8.0 $\mu g\ g^{-1}$) soil concentrations (Radojevic and Baskin, 1999; Alloway, 1995). The high level of cadmium in the dumpsite-soil could be attributed to anthropogenic sources such as spent batteries, sewage sludge, paints, plastics and metal plating. Many people could be at risk of adverse health effects including gastrointestinal, hematological, musculoskeletal, renal, neurological and reproductive dysfunction as a result of consuming crops contaminated with Cd (ATSDR, 1999).

The distribution of heavy metals between the different grids at the dumpsite and background soils and plant were highly variable. Fe was predominantly detected more than other heavy metals, in both background and dumpsite soils. *Manihot utilissima* cultured in dumpsite and background soil accumulated very high concentration of Fe and Mg (Table 1 and 2).

The linear correlation coefficient between identical pairs of heavy metal in *M. utilissima* and dumpsite soil gave -0.20, 0.19, 0.19,-0.53 and 0.44 for Cd, Cr, Pb, Fe and Mg, respectively. These results indicate insignificant relationship between the respective heavy metal concentration in the soil and accumulation by the plant. The uptake of metals by plants could have been influenced by other factors such as nature of soil, type of plant, climate and agricultural practice.

Frequency Characteristics of Heavy Metals Contents in Soil and M. utilissima of Dumpsite Environment

The sample skewness , kurtosis and probabilities of Kolmogorov-Smirnov test of raw data of extractable heavy metal concentrations of soil and *M. utilissima* of the dumpsite environment in Uyo, Nigeria, are listed in Table 4. Percentiles of 25, 50 (median) and 75% of concentrations are also listed.

In the research, the threshold of significance probability is maintained at $p = 0.05$. Thus, if the K-SP = 0.05, the normal distribution hypothesis is accepted. From Table 4, all the data representing the extractable heavy metal levels in both soil and *M. utilissima* from the dumpsite system followed normal distribution except for Mg (K-SP = 0.04) in the dumpsite plant. It is the basic hypothesis for many statistical methods that data should follow normal distribution (Zhang *et al.*, 1995). Comparatively, concentrations of heavy metals in the dumpsite soil have lower kurtosis than levels in *M. utilissima* and very improved skewness except Cr and Mg. This implies that Mg with the highest kurtosis (4.29) and skewness of 2.05 was the most mobile metal contaminant in the dumpsite soil. Chromium was also highly mobile with kurtosis and skewness values of 0.91 and 1.08, respectively. The least mobile metals in the dumpsite soils were Fe, Pb and Cd. These metals exhibited very low kurtosis and skewness values. Environmental factors such as pH, cationic exchange capacity, etc. might have contributed to their low mobility in the soil and the subsequent phytoextraction by *M. utilissima.*

Relative Standard Deviations (RSD) of Heavy Metal Contents in Soil and M. utilisimma

For an environmental study, one of the best ways of evaluating the variations of variables is the use of relative standard deviations (Zhang *et al.,* 1995). In the dumpsite soils, As has the largest RSD. value of 81.44%. This was followed by Cd and Cr with relatively low RSD values of 19.54 and 19.09%, respectively. Iron had the least value of 7.22%. The diversity property of the heavy metals in the dumpsite soil is primarily due to the differences in the natural conditions of the substrate. Similarly, in the *M. utilissima* cultured in the dumpsite soil, Cd, Cr, Pb, Fe and Mg exhibited relatively low RSD. values, indicating rather a small variation of the metal concentrations in the plant. The trends in variability in soil and *M. utilissima* were As>Cd>Cr>Pb>Mg>Fe and Pb>Cd>Mg>Cr>Fe>As, respectively.

Table 4 presents the computed statistical data on the measured heavy metals concentrations in soil and *M. utilissima* at the dumpsite environment.

Conclusions

The results of the present study have revealed high levels of heavy metals in *Manihot utilissima* and soil from the dumpsite. The frequency characteristics of the metal levels in the dumpsite plant and soil highlighted the low mobility or otherwise of some metals. However there was no clear relationship in heavy metal levels between cultured plant and corresponding soil obtained from the dumpsite. Although the levels of some heavy metals are within regulated limits, deterioration of the dumpsite soil and environs eco-quality can be expected with increase input of wastes and sludge. Cultivation of perennial plants in dumpsites should be totally avoided as they have the capability of gradually accumulating heavy metals including dangerous metals such as Pb and Cd.

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