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DETERMINATION OF SOME HEAVY METALS IN DUMPSITE SOIL AND Abelmoschus esculentus GROWN NEAR DUMPSITES IN KAFANCHAN METROPOLIS, KADUNA STATE, NIGERIA

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Abstract

The seasonal variations in the concentrations of Cd, Cr, Cu, Ni and Pb in the soils and Abelmochus esculentus fruit grown near 5 dumpsites in Kafanchan metropolis, Nigeria were investigated during the wet and dry seasons of 2015 using atomic absorption spectrophotometry. The soils were sandy loam and alkaline, with the cation exchange capacity, soil particle size distribution, pH, nitrogen, phosphorus and organic matter being higher in the dry season than in the wet. In the dry season, Cd in the soils range from 11.38 - 30.67 mg/kg, Cu 106.52 - 158.42 mg/kg, Ni 52.09 - 119.69 mg/kg, Pb 94.19 -308.35 mg/kg and Zn 98.43 - 332.83 mg/kg. The metals in the soil were more in the residual fraction (wet season 3.18 – 24.03 mg/kg; dry season 4.08 – 132.37 mg/kg); while the water soluble fraction had the least concentration (wet season 0.55 - 17.35 mg/kg; dry season 0.00 – 25.50 mg/kg). The soil pollution load index in the wet season was 1.95; contamination factor was 0.90 – 4.55 and the geo-accumulation index was -0.51 -1.11. The mean Cd, Cr, Cu, Ni and Pb in the A. esculentus ranged from 8.51 - 14.36; 7.98 -14.29; 7.35 - 18.80; 1.33 - 16.75; and 4.08 - 12.63 respectively. The plant had relatively low transfer factor for the metals. This study showed that the soils within the vicinity of the dumpsites were polluted by heavy metals, this calls for proper waste management practices and policy implementation.

Key Words: Amaranthus esculentus, Residual fractions, Pollution load index, Waste management

Introduction

Heavy metal concentrations in soil are associated with biological and geochemical cycles and are related to actions such as agricultural practices, industrial activities and waste disposal methods (Eja *et al.*, 2003). The presence of heavy metals at trace level and essential elements at elevated concentration do cause toxic effects if exposed to human population (Fong *et al.*, 2008). The accumulation of heavy metals in agricultural soils is of increasing concern due to food safety issues and potential health risks, as well as its detrimental effects on soil ecosystems (Qishlaqi and Moore, 2007).

The presence of dumpsites within living areas in developing nations results from inadequate waste handling facilities, coupled with poor regional and urban planning, lack of enforcement of relevant laws and edicts on waste disposal and lack of organized landfill sites (Abdus-Salam *et al.*, 2011). The burning of wastes gets rid of the organic materials and oxidizes the metals. These metals get dissolved in rain water and are then leached into soil, and then absorbed by growing plants, thereby entering the food chain (Benson and Ebong, 2005).

The order of bioavailability of the metals in the soils of some dumpsites in Zaria - Nigeria was Cd > Pb > Zn > Mn > Cu (Uba *et al.*, 2008). *Amaranthus spinusis* planted near dumpsites in Kaduna – Nigeria contained As in the range 0.001 - 0.030; Cd 0.019 - 0.250; Hg 0.144 - 0.590 and Mn 0.167 - 0.690 mg/kg in the wet season. The levels of these metals in *Amaranthus spinusis* and the soils did not vary significantly across the season (P > 0.05) (Omoniyi *et al.*, 2013).

This work reports the total concentration of Cd, Cu, Ni, Pd and Cr in

dumpsite soils and *Abelmochus esculentus* planted on the dumpsite soil in Kafanchan metropolis, Kaduna State, Nigeria; in order to assess the pollution indices and the transfer factor of the metals in the plant.

Materials and Methods Study Area and Sample Collection

Kafanchan in Kaduna State, Nigeria, is located on 9° 34' 36" N and 8° 16' 19" E. It has a mean temperature of 28°C and an estimated population of about 83,092. The study was carried out in five (5) solid waste dumpsites and a control (CTR) -5.0 km from Railway quarters) in Kafanchan metropolis, Nigeria. The sites are Garara Street dumpsite (GRR) which is about 30 years old with area 34231 m^2 ; Abuja Street dumpsite (ABJ) with area 58371 m^2 ; Albarka Street (ABR) dumpsite with area 200644 m²; Railway quarters (RLQ) dumpsite with area 420416 m²; Garaje dumpsite (GRJ) with area 14231 m² (Figure 1).



Figure 1: Map of the Study area

Samples were obtained by dividing each dumpsite into four quadrants in a diagonal basis following the methods of Nuonom *et al.* (2000). The dumpsite soils were collected with the aid of a clean stainless steel spoon at a depth of 10 cm from the top soil. The sample collection was in June 2015 (wet season) and November 2015 (dry season). The fruit of *Abelmoschus esculentus* (okra) were collected from farms 5 m from the dumpsite) and the control sites in the dry season.

Determination of Physico-chemical Parameters of the Dumpsite Soil

The soil sample was homogenized and air dried in circulating air in an oven at 30°C overnight and then passed through a 2 mm sieve (Awofolu, 2005).

The pH of 2.0 g of the dried soil mixed with 20.0 cm³ of distilled water was determined with a Hanna pH meter (model H12211). The electrical conductivity of the soil was determined with a conductivity meter using the modified method of Kumar *et al.* (2008), and Kalra and Maynard (1991).

The determination of soil type was carried out using the method of Gee and Or, 2002. Chloride (Cl⁻) contents were determined by titrating the filtrate of 5.0 g of the soil sample in 50 cm^3 of distilled water with 0.01 moldm⁻³AgNO₃ solution using 2 cm³ of 0.1 moldm⁻³ K₂CrO₄ solution as indicator until a muddy yellow colour was observed. Available nitrogen nitrate (NO_3-N) was determined using the methods described by Nnaji et al. (2011). Available phosphate – phosphorous $(PO_4^{3-}-P)$ was determined by the method of Agbenin (1995) using 15 cm³ of 1 M NH₄F and 25 cm^3 of 0.50M KCl.

The cation exchange capacity (CEC) of the soil samples were determined by the BaCl₂ Compulsive Exchange Method as described by Ross and Ketterings (1996). Organic matter in the soil samples was determined using the methods reported by Dutta *et al.* (2010) using 1 g of the dumpsite soil and 50 cm³ of 0.4M ferrous ammonium sulphate solution. Colour change from bluish green to brilliant green signalled the end point.

Metal Analysis

Quality assurance of the atomic absorption spectrophotometer: Quality assurance of the atomic absorption spectrophotometer was done by spiking the pre-digested samples of plant and soil with multi-element standard solution (10 $cm^{3}of 5 mg/l Cd, Cu, Ni, Pb and Cr)$ as reported by Awofolu (2005).

Digestion of soil samples: Each 1.0 g soil sample was digested in a Teflon cup with 30.0 cm^3 aqua regia (HCl: HNO₃). 3:1) on a thermostat hot plate at 150°C; after about 2 h of digestion 5.0 cm^3 concentrated HF was added and the mixture was heated further for 30 min. The digest was then made up to the 50 cm³ mark in a volumetric flask. The concentrations of Cd, Cu, Pb, Cr and Ni in the digest were determined by an atomic absorption spectrometer (Model-990) National Fertilizer PG at Development Centre, Kaduna, Nigeria.

Digestion of the fruit of Abelmoschus esculentus samples: The Abelmoschus esculentus fruit was dried and pulverised; these were digested according to method described by Awofolu (2005); using 5 cm³ concentrated HNO₃ and 2 cm³ HClO₄ at low heat on a hot plate. The levels of Cd, Cu, Pb, Cr and Ni in the digest were determined using atomic absorption spectrometer.

Sequential Fractionation of Heavy Metals in the Dumpsite Soil

Chemical fractionation of Cd, Cu, Pb, Cr and Ni was carried out on the dumpsite soils using the method described by Finzgar et al. (2007). This method modified the conventional method developed by Tessier et al. (1979). Six (6) geochemical fractions were determined: F1 (Fraction soluble in water), 1.0 g of the air-dried dumpsite soil sample was mixed with 10 cm^3 of deionized water with continuous agitation using a mechanical shaker for 1 hour, and then centrifuged. The supernatant was then decanted and made up to 50 cm^3 with deionized For water. F2 (Exchangeable Fraction), the residue from F1 was leached at room temperature with 10 cm³ of 1 M MgNO₃ at pH 7.0 with continuous agitation for 1 h and then The supernatant was centrifuged. decanted and made up to 50 cm^3 with deionized water prior to analysis. MgNO₃ displace ions electrostatically bound in the soil matrix. For F3 (Acid extractable - carbonate bound Fraction), the residue from F2 was leached at room temperature with 10 cm³ of 1M NaOAc (pH 5 adjusted with HOAc) with continuous agitation for 5 h and then centrifuged.

The supernatant was decanted and made up to 50 cm³ with deionized water prior to analysis. FIV (Reducible – Fe – Mn Oxides and Hydroxide Fraction), Residue from F3 was leached with 20 cm³ of 0.1M NH₂OH.HCl (pH adjusted with 25%v/v HOAc) at 96°C for 6 h with occasional agitation and then centrifuged. The supernatant obtained was decanted and made up to 50 cm³ with deionized water prior to analysis.

For F5 (Oxidizable - organic matter bound fraction), 3 cm³ of 0.02M HNO₃ and 30 %v/v H₂O₂ was added to the residue from F4. The mixture was then heated to 85°C in a water bath for 3 h. After cooling, 5 cm^3 of 1.0M NaOAc was then used for the extraction with occasional agitation for 3 h at 85°C. The mixture was centrifuged and the supernatant obtained was decanted and made up to 50 cm^3 in a volumetric flask with deionized water prior to analysis. For F6 (Residual and inert fraction), the residue from F5 was digested with a mixture of 8 cm³ of 5:1 mixture of concentrated HF and HClO₄ in acid digestion Teflon cup. Then the mixture was dry-ashed for 2 h and evaporated to dryness. The residue was then diluted to 50 cm^3 in a volumetric flask with deionized water prior to analysis. All extracts were analysed for the concentrations of Cd, Cr, Cu, Ni and Pb by atomic absorption spectrometry

Determination of Pollution Indices of the Dumpsite Soils

Contamination factor (CF) was determined using the expression: CF= Cm/Bm

Where Cm = mean concentration, Bm = background concentration of the metal directly determined from a geologically similar area (control sample) (Lar and Usman, 2014; Tijani *et al.*, 2004).

Geo-accumulation index (GeoI) of the soil was calculated using:

$Geol = \ln \left(Cm/1.5 \times Bm \right)$

Cm = mean concentration; Bm = background concentration of metal and 1.5 is a factor for possible variation in the background concentration due to lithologic differences (Lokeshwari and Chandrappa, 2006).

The transfer factor (TF) of the metals (Cd, Cr, Cu, Ni and Pb) from the dumpsite soil to the plant was calculated using the formula: $TF = C_{plant}/C_{soil}$

C_{plant} and C_{soil} are the concentrations of the toxic metal in the plants and soils (Lokeshwari respectively and Chandrappa, 2006).

The study evaluated the pollution load index (PLI) of the dumpsite soils with the expression:

$PLI = \left[\pi^n t(C_f)\right]^{1/n}$

Where C_f is the contamination factor of each metal obtained by the ratio of concentration of each metal in soil or plant to that of the metal in background soil or plant; π is the geometrical mean operator; n is the number of metals investigated and *i* represents each metal.

Statistical Analysis

Α two-factor ANOVA with replication was carried out at α level of 0.05 for the dumpsite soil samples using Microsoft Excel® 2013 to test seasonal effect on the level of Cd, Cr, Cu, Ni, and Pb in the dumpsite soil and Abelmoschus esculentus samples from the various dumpsites. And to determine the significant difference in the levels of heavy metals in the soil samples. The study also assesses the interaction between the levels of the metals by season.

A single-factor ANOVA was used to determine the difference in the levels of the heavy metals in Abelmoschus *esculentus* samples of the various sampling sites.

Results and Discussion

Physicochemical Parameters of the **Dumpsite** Soil

The soil was predominantly sandy loam. The sand content ranged from 78.0 to 84.0%. Excessive drainage of sandy soil can encourage leaching (Osazee et al., 2013). Table 1 indicated that the dumpsite soil samples had pH 7.10 to 8.90, similar to other studies (Uba et al., 2008; Elaigwu et al., 2007; Gupta and Sinha, 2006). However, the electrical conductivity of the soil vary across the sites.

Table 1: Physicochemical Parameters of the Dumpsite Soil in the Wet Season

Location		pН	EC	NO ₃ ⁻	PO_4^- (mg/kg)	Cl ⁻ (mg/kg)	CEC	OM (%)
			(µS/cm)	(mg/kg)			(Cmol/kg)	
ABR	Dry Season Wet Season	8.32±0.01	0.87 ± 0.04	0.03±0.02	75.17±0.02	6.32±0.01	8.00±0.01	3.85 ± 0.02
GRR		7.10±0.01	0.50 ± 0.01	0.07 ± 0.01	52.80±0.03	7.08±0.03	8.70±0.04	4.67±0.14
GRJ		7.22 ± 0.02	1.38 ± 0.12	0.04 ± 0.03	42.53±0.01	2.75±0.01	9.50±0.01	5.88 ± 0.03
ABJ		8.00 ± 0.02	0.53 ± 0.05	0.03±0.01	105.60±0.03	12.54±0.02	9.50±0.02	6.80 ± 0.02
RLQ		8.90±0.01	2.05±0.03	0.04 ± 0.02	61.14v0.02	6.98±0.01	8.50±0.03	5.40 ± 0.02
CTR		7.40 ± 0.02	0.11±0.01	0.02±0.03	18.03±0.01	1.73±0.01	6.00±0.01	2.83 ± 0.04
ABR		7.56±0.02	0.90 ± 0.01	0.04 ± 0.01	57.11±0.5	5.72±0.01	8.03±0.04	4.15±0.04
GRR		6.67±0.01	0.42 ± 0.01	0.08±0.03	56.05±0.03	8.18±0.03	9.51±0.02	5.02 ± 0.02
GRJ		7.20 ± 0.02	1.20 ± 0.02	0.05 ± 0.01	48.68±0.21	4.63±0.01	9.85±0.02	6.28±0.01
ABJ		7.01±0.01	0.40 ± 0.02	0.03±0.01	102.45±0.03	8.24±0.02	10.30±0.06	6.21±0.12
RLQ		7.98 ± 0.02	1.75±0.03	0.05 ± 0.02	63.00±0.02	2.54±0.01	9.81±0.02	6.01±0.02
CTR		7.30 ± 0.02	0.17±0.03	0.02 ± 0.01	19.23±0.01	2.12±0.01	6.11±0.03	2.81±0.02

EC: Electrical conductivity; CEC: Cation exchange capacity; OM: Organic Matter

High electrical conductivity of soils is an indication of significant presence of ions.

NO₃⁻N contents of the soil in the wet season ranged from 0.03 mgkg⁻¹ at ABR and ABJ to 0.07 mgkg⁻¹ at GRR, while in the dry season it was 0.03 mgkg⁻¹ at ABJ to 0.08 mgkg⁻¹ at GRR with the control sites recording 0.02 mg/kg for both the wet and dry seasons. The nitrate contents obtained was lower than the range 3.476 to 4.522% reported by Osazee *et al.* (2013).

The extractable phosphate of the dumpsite soils ranged from 42.53 mg/kg to 105.60 mg/kg during the wet season and 48.68 to 102.45 mg/kg in the dry season (Table 1). The organic matter (OM) content of the dumpsite soil was highest in the ABJ sample and lowest in the ABR sample in the two seasons. The Cl⁻ content of the dumpsite soils ranged from 2.75 mg/kg GRJ dumpsite to 12.54 mg/kg ABJ dumpsite in the wet season. The highest cation exchange capacity (CEC) was at ABJ (10.30±0.06 Cmol/kg) in the dry season and was least in the control sites.

Total Heavy Metal Concentration of the Dumpsite Soil

As depicted by Figure 2 and 3, Cd in the wet season had the range 21.86 -58.27 mg/kg, and 24.60 - 13.00 mg/kg in the dry season. The highest concentration of Cd was recorded in ABJ dumpsite (58.27 mg/kg). The relatively high concentration of cadmium in the study areas could be linked to the location of the dumpsite and the nature of the waste been dumped. Cadmium concentrations in the soils were above the standard limits (0.01 mg/kg) set by Federal Environmental Protection Agency (FEPA) 1991 and (3.0 mg/kg) set by European Commission (1986).

The level of Cr in the dumpsite soils ranged from 25.86 mg/kg - 44.69 mg/kg in the wet season and 20.34 - 52.89mg/kg in the dry season. Cr concentration in the refuse soils were generally above standard limits of 0.03 mg/kg set by FEPA (1991) and 50 mg/kg set by European Commission (1986). From the results, the level of Cu was generally higher than all other metals studied. The highest level was 161.30 mg/kg in GRR dumpsite during the dry season. Copper in the soil was above the standard limits 140 mg/kg set by of European Commission (1986). The soil also contained Ni in amounts above the threshold limit.

Lead in the soils ranged from 23.62 - 56.63 mg/kg in the wet season and 42.70 - 72.13 mg/kg in the dry season. The soil had Pb in amounts above the 0.05 mg/kg limit recommended by FEPA (1991). The soil metal content follow the ranking Cr > Ni > Pb > Cd > Cu in the wet season and Cu > Ni > Pb > Cr > Cd in the dry season.



Figure 2: Concentration of metals in the soil samples during the wet season



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Figure 3: Concentration of metals in the soils samples during the dry season

Concentration of heavy metals in the A. esculentus fruit collected in the dry season

Cadmium concentration in Α. esculentus fruit planted on the dumpsite soil in the dry session ranged from $8.53 \pm$ 0.03 mg/kg (ABR) to 21.15 ± 0.12 mg/kg (ABJ); being above the FAO/WHO (2001) limit of 0.2 mg/kg. However, Fatoba et al. (2012) reported 0.01 - 0.03 mg/kg Cd in okra irrigated with industrial waste effluents in Kwara State, Nigeria, and a highest amount of 0.08 mg/kg was reported by Amusan et al. (2005) for okra fruit obtained from dumpsites in Obafemi Awolowo University, Ile-Ife, Nigeria. In this study the highest Cr in the fruit was 14.29 ± 0.01 mg/kg (GRJ) and the lowest was 7.98 ± 0.01 mg/kg (GRR). Chromium content in the plant was generally above the limit of 2.30 mg/kg set by FAO/WHO (2001). The highest level of Cu in the fruit (18.80 ± 0.01) mg/kg) was in the GRJ samples; Cu level was below the 73.3 mg/kg limit set by FAO/WHO (2001). Cu in this study was higher than the values reported by Akpofure (2012) for Hibiscus esculentus grown on abandoned dumpsites in Effurun, Nigeria.

The level of nickel in the *A*. esculentus fruit was highest in the ABJ (16.75±0.06 mg/kg) and lowest in the RLQ samples (1.33±0.02 mg/kg); the amounts were below limit of 67.90 mg/kg set by FAO/WHO (2001). Nickel in plants is highly mobile and is likely to accumulate in both leaves and seeds (Sengar *et al.*, 2008). Lead concentration in the fruit ranged from 4.08 ± 0.25 mg/kg (GRR) to 12.63±0.04 mg/kg (ABJ). Pb content in the fruit was above the set limit of 0.3 mg/kg set by FAO/WHO (2001).

Chemical Fractionation of the Heavy Metals in the Dumpsite Soils

The ranking of Cd in the various fractions in the wet season was residual > water soluble oxidisable > > exchangeable > acid soluble > reducible; for Cr - Residual > exchangeable > acid soluble > water soluble > reducible > oxidisable: Cu - Residual > water soluble > acid soluble > exchangeable > reducible > oxidisable; Ni - Residual > water soluble > reducible > acid soluble > exchangeable > oxidisable and for Pb -Residual > oxidisable > water soluble > acid soluble > reducible > exchangeable.

Since the highest concentrations of all the metals were in the residual fraction, the metals would be released into the environment only under very harsh conditions because they are bound firmly within a mineral lattice that restricts the bioavailability of this metal (Abu-Kukati, 2001). The non-residual fractions constituted 75% Cd, indicating its ready availability for release. This finding conforms to the report of Gupta and Sinha (2006) and Obasi et al. (2013) for the soil from a tannery in Uttar Pradesh, India and for dumpsites in Okigwe, Nigeria respectively. The bioavailable Cd (F1 - F3) were relatively high ($\geq 40 \%$), thus Cd in the soil is bioavailable for plant uptake (Uba et al., 2008).

The highest bioavailability values for Cr were 70% at GRJ and 59% at ABR in the wet and dry seasons respectively. The bioavailability of copper was 11% (GRR) to 47% (ABR) in the wet season and 44% (ABJ) to 53% (GRJ) in the dry season. So, Cu will be readily bioavailable to the environment. However, some of the dumpsite had percentages of the total extractable fractions in the mobile phase (F1+F2+F3+F4+F5 >28%), indicating that these metals are readily bio-available to the environment (Uzairu *et al.*, 2013).

The highest extractable fractions of Ni was 70.69 mg/kg at GRJ and the lowest was 31.45 mg/kg at RLQ in the wet season. The percentage bioavailable nickel in the dumpsite soils were relatively high in ABR (57%) in the dry season and low in GRJ (13%) and ABJ (34%) in the wet season samples. This indicates that Ni would be readily bioavailable to the environment. The highest bioavailable Pb was 85% at GRR and 56% at GRJ in the wet and dry season respectively.

There was a significant difference in the concentrations of the metals studied

across the dumpsites and the seasons (two-way ANOVA, P < 0.05). However, there was no significant difference in the levels of the metals in *A. esculentus* fruit samples (P > 0.05). The most significant correlation existed for Cu_{soil} vs Pb_{soil} (r = 0.909), Ni_{soil} vs Pb_{soil} (r = 0.944), Cu_{soil} vs Ni_{soil} (r = 0.914), Ni_{soil} vs Cu_{plant} (r = 0.776), Cr_{soil} vs Ni_{plant} (r = 0.757). The positive correlation between the metals across the sites suggests a common source of pollution.

Contamination Factors

Pollution load index (PLI) for the soils in the wet and dry season were 1.73 and 1.95 respectively. When PLI value is below or close to one, it indicates heavy metal loads at the baseline, while values above one indicate heavy metal accumulation or pollution in plant or soil from the test site (Agunbiade and Fawale, 2009). Contamination factor (CF) of the dumpsite soils indicated that they were considerably contaminated in the wet season by Ni at GRJ (CF 3.33), and also by Pb at ABJ (CF 4.14), GRJ (CF 3.61) and at GRR (CF 4.55); while in the dry the soil was considerably season contaminated by Cu at all the dumpsites with exceptions at RLQ (CF 2.20); and was moderately contaminated by all the five heavy metals in both seasons. The soil had low contamination by Cd and Cu at GRR (0.90) and RLQ (0.96) in the wet season; while in the dry season the CF for Cd were: at ABJ (0.71), GRJ (0.76), RLQ (0.75),and GRR (0.59).The contamination of the soil by the metals studied decreased in the order: Cu>Ni>Cr>Pb>Cd in the dry season, and was Pb>Ni>Cd>Cr>Cu in the wet season.

The transfer factor (TF) of copper from the soils to plants was highest at GRJ (0.86). The TF of Ni was 0.19 -0.25, 0.23 - 0.73 for Cd and 0.08 - 0.17for Pb. Factors such as pH, exchange binding capacities, climate change and morphology of the plant might contribute to low transfer factor in dumpsite soil (Udosen et al. 2006). Pb and Cu had the highest I-geo of 1.11 and 1.35 at GRR in the wet and dry season while Cd had the least I-geo in both seasons. The study indicated that the contamination status of dumpsite soils range from the uncontaminated to slightly contaminated/

Conclusion

physiochemical The parameters indicated that the dumpsite soils are rich in plants nutrients. The heavy metal concentration in the dumpsite soil in Kafanchan metropolis varied significantly by sampling sites and metal type. The heavy metals were retained predominantly by the residual fraction (F6), indicating that these metals would be potentially unavailable in most of the dumpsites. In addition, the non - residual fraction (NRF) retained higher amount of Cd, Cr, Cu, Ni, and Pb in most of the dumpsites, hence indiscriminate dumping of refuse should be discouraged especially household near and commercial areas. These results imply that pollution of an environment by dumpsites has human health and ecological risks and that plants that are cultivated on dumpsite or the use of dumpsite as soil manure resulted to significant bioaccumulation of the heavy metals studied by sampling site (P <0.05). Therefore, dumpsites in Kafanchan metropolis, that was studied is a potential sources of heavy metal in plants and the

environment; this suggests the use of incinerators for waste collection and enlightenment on the risks of growing crops on dumpsites.

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