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Assessment of levels, Bioavailability and Fractionation of Lead and Cadmium in soils of abandoned Municipal waste open dumpsite proposed for Food crop cultivation

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Abstract

This study was carried out to assess the levels and speciation of heavy metals (Pb and Cd) in soils of an abandoned open central municipal solid waste dumpsite at Ugwuaji in Enugu, Nigeria. Samples of soils were collected from the site of the old dumpsite. To obtain the levels of the metals, the samples were cleaned, dried and pulverized. Wet digestions were carried out on all the samples using nitric- perchloric acids, followed by the evaluations of heavy metal contents using Flame Atomic Absorption Spectrophotometer (FAAS). The heavy metal concentrations in the soils (mg/kg) ranged from 20.825 to 62.501 and 7.250 to 11.806 for Pb and Cd respectively, which were all higher than their levels in the control samples, but were within the recommended limit by WHO and CCME except for Cd. Metal fractionations were carried out using the Tessier's protocol. The results showed that 62% and 29% of the total Cd and Pb content of the soil were bioavailable respectively. The results raise a lot of concern as the residents are using the abandoned site for cultivation of edible vegetables and other perennial crops.

Keywords: Dumpsite, Ugwuaji, lead, cadmium, speciation, bioavailability.

Introduction

Refuse dumpsites most of the times contain toxic materials which are capable of polluting surrounding soils especially if the dumpsites are not adequately shielded from the surrounding area. There is indiscriminate dumping of municipal solid wastes (MSW) in developing countries today scarcity of funds and resources for proper disposal, scarcity of skilled man-power among others. In most towns in Nigeria, dumping of municipal waste in open is common practice. Invariably, these causes adverse impacts on the environment. In fact nearly all human activities generate wastes which do pose serious risk to the environmental components such as the groundwater resources, air, soils and plants that grow on them. These wastes from different sources end up mostly at these dumpsites and pollute the soil of the area[1,2]. The resultant effects of such environmental pollution on the affected soil are very numerous. For instance, electronic wastes, painting wastes, used batteries etc., are usually dumped along with municipal solid wastes and can raise the heavy metals in the dumpsites[3]. Various studies have been carried out on heavy metals such as As, Cd, Ni, Cr, Zn, Co, Mn, Fe, and Cu in MSW dumpsites[2,4,5].

It is a common practice to abandon wastes for a reasonable long period of time especially for wastes deposited along roadsides considered to be away from residential areas. Heavy metals eventually enter into the soil from the metal related wastes such as metal scraps, agricultural and industrial wastes consequently, since the assorted wastes are not being screened before deposition. Among the heavy metals most commonly found at contaminated sites are Lead (Pb) and Cadmium (Cd)[6]. It has been reported that soils are the major sinks for heavy metals released into the environment by anthropogenic activities[1]. Various studies have been carried out on heavy metals such as As, Cd, Ni, Cr, Zn, Co, Mn, Fe, and Cu in MSW dumpsites[2,4,7].

The management of waste in Enugu City is a complex and difficult task. The more popular method of disposal of wastes have been to first collect them from their sources, and then deposit them in the waste bins or sites placed in strategic areas of the city by the government. The Enugu State Waste Management Agency (ESWAMA) collects all the deposited wastes from the various dumping site in the city and dump them in the major open municipal dumpsite, during dry season, the wastes were set ablaze. Prior to now waste speciation was none existent. When a new dumpsite is approved, the old one is left for other activities such as agriculture, sports and even residential buildings. The soil profile comprises of distinct layers (called horizons) differing in colour, texture, and structure, within the soil profile. Pb and Cd among other heavy metals are found concentrated in the surface horizon as a result of cycling through vegetation, atmospheric deposition and adsorption by the soil organic matter. However, recently polluted soils often have high concentration of metal pollutants in top soil until anthropogenic processes affect redistribution within the profile[5].

Metal speciation is a technique used to estimate the heavy metal geochemical partitioning in the soil. In a contaminated soil, heavy metal specification strongly influences the bioavailability, mobility and toxicity of metals[8], since the soil metals can exist in solution, organic matter, occluded into soil oxide mineral, on exchange sites or formed in the lattice structure of secondary and primary soil mineral[9]. Sequential extraction method is one of the several techniques used to speciate metals in the soil and has been globally accepted[10]. Several procedure of metal sequential extractions have been proposed, such as the Tessier sequential extraction procedure[11], where a 5-staged extraction procedure was developed to evaluate the fractions of Pb, Cd, Fe, Mg, Cu, Zn and Ni in river sediments. Other procedures developed include Sposito et al[12], BCR (community bureau of reference recently known as the standards, measurements and testing program of the European commission)[13] and others.

The reported work studied levels and speciation of Lead and Cadmium in the soils of an abandoned open municipal solid waste dumpsite (Ugwuaji abandoned dump site) to assess their bioavailability to bioata potentially available on the site.

Materials and Methods

Study area: This area of study was an abandoned municipal solid waste dumpsite along Enugu/Port Harcourt road, Enugu state (Ugwuaji dumpsite). Enugu is located between longitude 7°6'E and 7°54'E and latitudes 5°56'N and 6°52'N. The state has borders with Abia and Imo State to the South, Ebonyi State to the east, and Benue State to the north-east, Kogi State to the north-west and Anambra State to the west. Enugu falls within the humid tropical rainforest belt of the southern Nigeria and the soil is mineral rich hydro-orphic whose morphology is influenced by seasonal water logging caused by underlying impervious shale. The rainfall average is 1412 millimeters per month, with the lowest rainfall in February and the temperature ranges between 20.3°C and 32.16°C throughout the year. The old dumpsite is and its environs are shown in Figure-1.

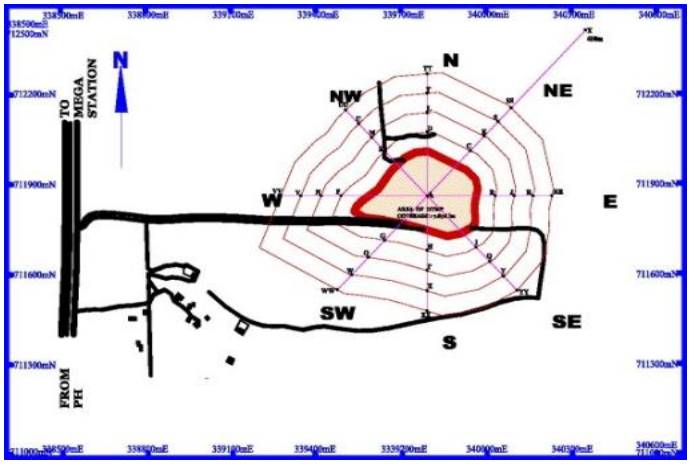

Figure-1: Map of the abandoned waste dumpsite at Ugwuaji and environs.

Soil sampling: Five sampling stations were created for the study and at each station; five (5) sampling points were created randomly but equidistant from each other at the apices of the station and the center. At each sampling point, a mini profile pit was dug to clearly reveal the soil profile to depth of 0-15cm. The soil samples were collected at the specified depth. A mass of 500g of soil was collected from each site clean plastic shovel. After collection, a composite was made of the soils collected at each station. In the course of this study, a total of 25 composites soil samples were collected from the five (5) sampling sites/stations created and on two (2) batches at month interval. The sampling stations created were; S_1; at the dumpsite, at western side, S_2; at the dumpsite, at northern side, S_3: at the dumpsite, at southern side, S_4: at the dumpsite, at eastern side, S_5: a open space 200m away from the dumpsite (to serve as control).

Soil Sample Preparation: Debris and stones were carefully removed from soil samples. The samples were air-dried by spreading on previously washed and dried polythene sheets. The dried soil samples were pulverized to fine dust using plastic mortar and pestle, sieved to pass through 2mm mesh sieve mixed by coning and quartering, and stored in a previously washed, dried and labeled polythene containers. The soil samples were divided for use in total metal determination and fractionation procedure.

Soil Digestion: Wet digestion using Nitric-perchloric acids digestion {HNO_3:$HClO_4$} in 4:1 ratio was utilized following the procedure recommended by the ADAC[14]. Two gram (2g) of each of the soil samples was placed in a 250 - digestion tube and 20ml of conc. HNO_3 (67% BDH, UK) added. The mixture was heated gently for 30-45mins to oxidize all the easily oxidizable matter. After cooling, 10ml of 70% $HClO_4$ acid (Merck, Germany) was added and the mixture boiled gently until dense white fumes appears. A 30ml of deionized water was added after cooling and the mixture boiled further to release white fumes. The solution was cooled, and filtered through whatman No 42 filter paper and <0.43mm Millipore paper and transferred quantitatively to a 50ml volumetric flask and mark made with demonized water. A blank was similarly prepared but without soil sample. The extract solutions were stored in acid-pre-washed polythene bottles ready for analysis.

Sequential Extraction Procedure: The method used in this study was according to Tessier's et al[11] and commonly used Roo et al[15]. The treatment of the soil into different fractions was to systematically leach the metals that were; exchangeable (loosely bounded), bound to carbonate, bound to Fe - Mn oxides, bound to organic matter and finally, the residual metals as elaborated below; in the extraction procedure, 2g of the soil sample was placed in a 50ml centrifugation tube, and the sample was exposed to reagents and shaken. Each fraction was separated from the supernatant by centrifugation (at 4000 rpm) for 30 min. The solution was separated, while the residue was rinsed with 12ml of deionized water and centrifuged again for 20 min.

The washing water was then added to the supernatant and the residue ready for subsequent extractions. The extracts were transferred quantitatively to a 50 ml volumetric flask and was reached to mark with demonized water, and ready for AAS analysis. The residue remaining at the end of the 4th fractionation was digested as the same method used in total heavy metal digestion. For each fraction a blank was similarly prepared. i. Fraction 1 (F_1): Exchangeable metals- The soil was extracted at room temperature for 1h with 12 mL of magnesium chloride solution (1M $MgCl_2$ pH 7.0) with continuous agitation. ii. Fraction 2 (F_2): Metals Bound to Carbonates -The residue from (i) was leached at room temperature with 12 mL of 1M sodium acetate (NaOAc) adjusted to pH 5.0 with acetic acid (HOAc). Continuous agitation was maintained for 3hrs. iii. Fraction 3 (F_3): Metals Bound to Fe-Mn Oxides - The residue from (ii) was extracted with 30mL 0.04 M hydroxyiamine hydrochloric acid NH_2OH/HCl) in 25% (v/v) acetic acids (HOAc) for 6h. This was done at 96^0C with occasional agitation for 6hrs for a complete dissolution of the free iron oxides. iv. Fraction 4 (F_4): Metals bound to Organic Matter - To the residue from (iii) were added 4.5mL of 0.02 M HNO_3 and 7.5mL of 30% H_2O_2 adjusted to pH 2 with nitric acid, and the mixture was heated at 85^0C for 3h with occasional agitation. After cooling, 7.5 ml of 3.2 M NH_4OA_C in 20% (v/v HNO_3 + 7ml water) was added and agitated for 20 min, and at room temperature. The addition of NH_4OAc is designed to prevent adsorption of extracted metals onto the oxidized soil. v. Fraction 5 (F_5): The residual metals -The residue from (iv) was digested with HNO_3 and $HClO_4$ in a ratio of 2:1 mixture according to the procedure described for total metal analysis.

Analysis of Heavy Metals: The digested samples and all the supernatants were analyzed for heavy metals (Pb, and Cd) using Atomic Absorption Spectrophotometer (Shimadzu AA7000 made in Japan) fitted with deuterium lamp for background correction.

Data analysis: The laboratory results were subjected to statistical analysis. Using Microsoft excel, the data was subjected to descriptive statistical analysis. The experimental results were expressed as mean ± standard deviation (SD) of triplicate determinations. The concentrations of elements from the sequential extraction were summed and compared with their concentration after total digestion of the original sample. All analysis was made in triplicates and their averages reported.

Mobility factor: In sequential extraction procedure, the early fractions F1 and F2 are the most mobile and bioavailable fractions. Hence, the mobility factor value provides the relative mobility and biological availability of heavy metals in soils. In this study, the mobility factor of heavy metals was evaluated using the equation[16]:

$$\text{Mobility Factor} = \frac{F1+F2}{[F1+F2+F3+f4+F5]} \times 100$$

where: F1 = exchangeable fraction; F2 = carbonates fraction; F3 = Fe-Mn oxide fraction; F4 = organic fraction; F5 = residual fraction.

High mobility factor values mean or have been interpreted as proof of relatively high reactivity, high lability and high biological availability of heavy metals in soil[17,18].

The Enrichment Factor (EF): EF, as introduced by Alina Kabata-Pendias[18], is used to assess the degree of anthropogenic influence on element load in the soil, and also to differentiate between elements originating from the natural or anthropogenic activities. The enrichment factor (EF) or accumulation factor for an element was calculated using the formula.

$$EF = \frac{\left[\frac{Xi}{Fe}\right]soil}{\left[\frac{Xi}{Fe}\right]control}$$

Where: Xi, Fe are the concentrations of element 'i' and Fe respectively.

Quality control: Glass wares were washed and rinsed properly with deionized water. Reagents of pure analytical grade were used. Deionized water was used throughout the study. Reagent blank determinations (deionized water and acids) were used to correct the instrument readings. The most sensitive wavelength for each element was selected for analysis, and calibration of AAS was done using multi-elemental solution prepared by serial dilution of 2, 4, 6, 8, and 10. Also, as quality control measures, recovery of the sequential extraction procedure was obtained as follows:

Recovery (%) = ({Fraction 1+Fraction2+ Fraction 3 +Fraction 4 + Fraction 5)/single total metal concentration} x 100

Results and Discussion

Levels of the metals: The concentrations in mean concentration ± standard deviation of the heavy metals (Pb and Cd,) in the studied soil are presented in Table-1. The results show that the metal loads of the soil samples at the abandoned dumpsite were higher than the control sample and this could be attributed to the serious anthropogenic influence which is in connection with the presence of assorted wastes ranging from domestic, commercial and industrial wastes in the dump sites. The result is similar to work by Narwal et al.[16]; Oluwakemi et al[1] and Ale et al[20], who reported that lead and cadmium are normally abundant in upper layer soils as a result of anthropogenic activities. The study by Aja[21] confirmed that the concentration of heavy metals in the dumpsite soil are higher in dry season than in rainy season due to more heavy metal loss as a result of run-off and infiltration in rainy season. It encourages the mobility of metals through leaching and water runoff during the raining seasons. The order of abundance of the six heavy metal monitored in the vicinity of the dumpsite as compared with the control was Pb > Cd.

The concentration of Pb in the various soil samples ranges from 35.72±0.917 mg/kg to 21.15±0.928mg/kg with an average of 34.294±0.681 mg/kg at the abandoned dumpsite, and 6.94± 0.972mg/kg in the control site as shown in Table-1. All the values were lower than the EU[22] upper limit of 300mg/kg and the maximum tolerable levels proposed for agricultural soil (90–400 mg/kg) set by WHO[23]. This results agree with the results obtained from related study by Umoh and Etim[24] for soil from dumpsite within Ikot- Ekpene in Akwa-Ibom State Nigeria.

Cadmium level in the soil samples ranges from 8.861±0.139 mg/kg to 7.94±0.119mg/kg, with an average concentration of 8.039±0.660mg/kg, while in the control the Cd content was found to be 6.80±0.390mg/kg. These values are far higher than the natural limits of 0.01-3.0mg/kg in agricultural soil, as recommended by EU[22] and UNEP[25] but lower than the upper limits (85mg/kg) as recommended by WHO[23]. This is in line with similar findings by Abata et al.[26] and Amos-Tautua[27]. Sources of Cd in the site could be as a result of cadmium contaminated wastes used for agriculture, the decay of abandoned electric batteries and other electronic components[28]. Cd is a non-essential element and actually displaces Zn, leading to Zn deficiency. It accumulates principally in the kidney and liver[29]. Cd and several Cd compounds are known as carcinogens and can induce many types of cancers. Saplakogelu and Iscan[30] have reported that long-term intakes of Cd caused renal, prostrate and ovarian cancers.

Soil fractionation: Table-2 and 3 show the result of soil fractionations for Pb and Cd by Tessier's sequential extraction scheme their total concentrations, their percentage recovery, and mobility factor. The mean content of Pb and Cd in the exchangeable fraction (F1), fraction bound to carbonate (F2), bound to Fe – Mn oxide (F3), bound to organic matter (F4), and residual fraction (F5), were listed. Table-9 and 10 gives the percentage content of Cd and Pb in the various fractions of the studied soil samples.

The concentrations of the metals (relative abundance %) in the mobile phases (first three fractions) are of the order; Cd (62%) > Pb (29%) while the immobile concentrations are of the order; Pb (79%)> Cd (38%). Similar result is gotten in Lai et al[31].

The element (Pb and Cd) content determined in the five fractions following the sequential extraction procedure are shown in Table-2 and 3. Exchangeable, bound to carbonates, and bound to Fe – Mn oxides species, corresponding to the first three fractions (F1–F3),are the active heavy metals and are generally called the bioavailable, as they exhibit a mobility relative to the environment and are potentially available for plants. Results show that the amounts of Cd, in bioavailable form is significantly high and evenly distributed among the various soil fractions. Similar result was reported for soil by Guire et al.[32] and Tesi et al[2]. Pb was not in the bioavailable fractions (F1-F3) of most soils, and this could be attributed to the facts that Pb is a low mobility metal in soil, it binds strongly with organic matter and chemisorbs on oxides of Fe, Mn, and Al.

Table-1: Total metal concentrations of the soil samples (mg/kg).

Sample station	S_1,	S_2	S_3	S_4	Mean	$S_{5\ (control)}$
Pb	21.15± 0.928	22.82± 0.928	35.72 ± 0. 917	33.62±0.972	34.29 ± 0.681	6.94 ± 0.972
Cd	8.15± 0.533	7.94± 0.119	8.86± 0.139	8.33±0.383	8.04 ± 0.660	6.80 ± 0.390

Table-2: Fractional and total lead (Pb) concentration (mg/kg) of the soils.

Sample I.D	F_1	F_2	F_3	F_4	F_5	Sum	Total metal	Recover %	Cf
S_1	6.545	6.245	4.100	3.345	0.600	20.38	21.15	98	13.89
S_2	6.946	4.250	3.900	4.639	2.100	21.84	22.83	96	1.892
S_3	6.280	5.609	4.945	4.000	12.5	33.33	35.72	93	1.667
S_4	6.830	5.300	6.245	6.540	8.105	33.02	33.62	98	0.3
Mean ± STD	6.650 ± 0.07	5.351 ± 0.84	4.800 ± 0.08	4.631 ± 0.25	5.831 ± 4.30				
R.A (%)	6.526	15.167	7.165	25.648	45.499	100			
S_5 (control)	ND	ND	ND	3.417	3.361	6.778	6.945	98	

Mf = mobility factor, ND = not detected; R.A = Relative abundance.

Table-3: Fractional and total cadmium concentration (mean, mg/kg) of the soils.

Sample ID	F1	F2	F3	F4	F5	Sum	Total Cd	Recovery %	Mobility factor
S_1	1.196	1.929	1.817	1.718	1.250	7.910	8.15	97	3.511
S_2	1.306	1.583	1.694	1.361	1.778	7.722	7.94	97	3.141
S_3	1.472	1.778	2.000	1.667	1.833	8.750	8.86	99	2.864
S_4	1.583	1.722	1.556	1.639	1.278	7.778	8.33	93	3.804
Mean ± STD	1.389 ± 0.22	1.753 ± 0.61	1.767 ± 0.57	1.596± 0.40	1.535 ± 0.41	8.04 ± 0.44	8.32 ± 0.72	97	3.43 ± 088
R.A (%)	14.83	26.150	21.400	17.51	19.830	100			
S_8 (control)	1.250	2.250	1.639	0.556	1.083	6.778	6.801	100	4.744

The relative abundance (%) of Cd and Pb are shown in Figure-2 and 3.

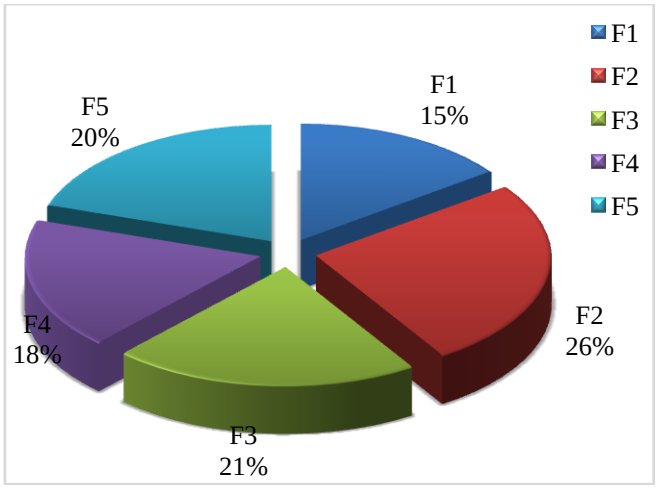

Figure-2: Relative abundance for Cd.

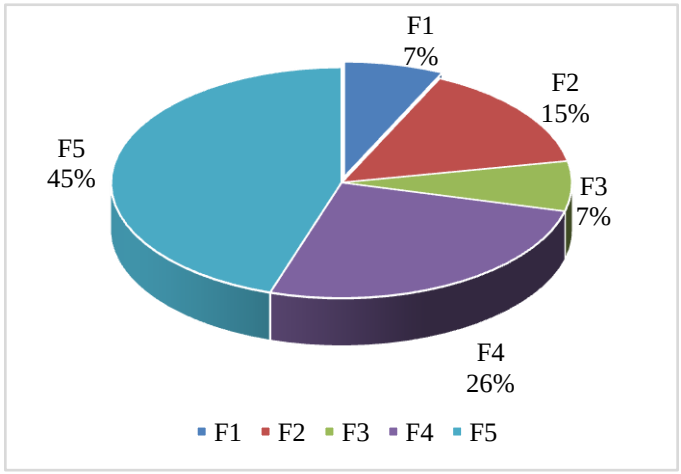

Figure-3: Relative abundance for Pb.

Mobility of heavy metals in the soils: The results of the mobility factor of heavy metals in soils of refuse dumpsites in this study are shown in Table-4. The relative abundance of Cd and Pb in the soil followed the trend; Cd; F_2>F_3> F_5>F_4>F_1., Pb; F_5> F_4 > F_2> F_3>F_1.

Table-4: Mobility factor of Pb and Cd in soils of abandoned MSW.

Sample station	S1	S2	S3	S4
Pb	63	51	36	37
Cd	40	37	37	42

The mobility factor values of the heavy metals ranged from 36 to 63 for Pb and 37 to 42 for Cd. The mobility factors of the heavy metals were generally moderate on the average indicating that the heavy metals in these soil profiles on the site of abandoned MSW dumpsites have relatively moderate reactivity, lability and biological availability[18].

Element enrichment factors (efc): Element enrichment factors **(**EFc), is used to assess the degree of anthropogenic influence on element load in the soil, and also to differentiate between elements originating from the natural and anthropogenic activities[33], result of EFc assessment is shown in Table-5.

Table-5: Enrichment Factor of Metals in abandoned Ugwuaji dumpsite.

Sample site	S_1	S_2	S_3	S_4	Mean
Pb	2.235	2.625	3.004	3.292	2.780 ± 0.71
Cd	0.799	2.095	1.073	0.727	1.170 ± 0.21

An enrichment factor (EFc) greater than unity, is an indication that the investigated metal is more abundant in the dumpsite relative to that found in the control station/site. The enrichment factor of the soil in the site of the vicinity of the dumpsite was quite high for all the metals as compared with the control, suggesting a very serious anthropogenic influence which is as a result of all kinds of assorted wastes ranging from domestic, commercial and industrial wastes which the landfill receives[2, 32]. The mean accumulation factors of trace heavy metals in the soil samples were ± 1.71 and 1.170±0.21 for Pb and Cd respectively which were higher than unity indicating that the investigated metals were more abundant in the dumpsite relative to that found in the control station/site[34]. The order of abundance in the dumpsite area due to anthropogenic influence is; Pb > Cd. However, lower values of heavy metal enrichment factors obtained suggest good retention of metals in soils of those areas.

Conclusion

The rate of increase in heavy metal pollution in the study area is alarming. Although some of the studied heavy metal (Pb, Zn and Cr) were within the maximum permissible limits, others like Cd, Fe and Al are above their recommended limits. This study recommends the need for an immediate intervention by the government to ameliorate pollution (especially Cd pollution) in the area. Any threat to the environment should be minimized or eliminated.

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