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Speciation study of heavy metal concentrations of roadside dusts of Ibara round about, Sokori road in Abeokuta north local government area of Ogun state, Nigeria

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ABSTRACT

This study was designed to determine the total concentrations of various species of heavy metals in surface soils and associated sediments obtained from some major roads in Abeokuta, Ogun State; identify the major sources of the metals in each of the areas sampled and identify measures to prevent the contamination of the environment. The study involved sampling of roadside dusts of five major locations in Abeokuta, Ogun state to assess the level of contamination. Speciation studies were carried out to determine the various fractions of seven metals namely: Zn, Pb, Cu, Mn, As, Cr and Cd. Selective sequential extraction procedure developed by Tessier et al, (1979) was used to fractionate roadside dust samples. This method involves partitioning heavy metals into five operationally defined fractions: exchangeable, carbonate, Fe-Mn oxide bound, organic and residual. The concentration of Zinc in the investigated samples was the highest (164.83µg/g). Chromium had the second highest value with a concentration of 101.3µg/g. This was followed by Manganese and Lead with concentrations of 87.03 and 80.33 respectively. The percentage mobility factors reported were in the order: Mn > Zn > Pb > Cu > Cd > Cr > As. There were indications that the levels of Zn, Pb, Mn, and Cu detected for the sampled areas were mainly associated with the carbonate bound fraction in the soil samples. The distribution of the metals in the various fractions and results of mobility factors confirmed their differences in mobility and bioavailability. There was also an indication that sources of these metals were mainly anthropogenic.

Keywords: Heavy metals, Contamination, Roadside dusts, Speciation.

1. INTRODUCTION

There is increased exposure to heavy metals and this highlights the need to identify and quantify those species in the environment that pose the greatest potential threat to human and organisms in order to take appropriate measures. In addition, metal species identification has use for researchers studying soil fertility, land-use planning, water quality, soil genesis and geomorphology, environmental quality, soil ecology and soil remediation. Quantification is typically done using chemical solutions of varying but specific strengths and reactivity to release metals from the different fractions of the examined soil (Ryan et al, 2008).

The solid soil samples are normally dissolved prior to measurement in a solution strong enough to cause total dissolution of all solid phases. Solutions of this type include microwave-HNO_3 digestion, hot-plate reflux digestion with HNO_3-HCl-H_2O_2, aqua regia-hydrofluoric acid mixtures, and digestion in hydrofluoric acid.

The resultant solutions can then be analyzed using one of several analytical techniques including atomic absorption spectrometry (AAS) and inductively coupled plasma-mass spectrometry (ICP-MS) spectrometry. Other means include X-ray fluorescence (XRF) which helps to determine total metal concentration in soils. The benefit of this technique is that we do not have to use harsh chemicals for total dissolution because the analysis is done using the whole soil, but metal concentrations normally have to be upwards of 5mg kg^{-1}. In addition, metal concentrations in solid slurries may be gleaned using ICP-MS.

An experimental approach commonly used for studying the mobility, transport and bioavailability of metals in soil is the use of selective sequential extraction procedure developed by Tessier et al (1979). This method consists of partitioning heavy metals into five operationally defined fractions: exchangeable, carbonate, Fe-Mn oxide bound, organic and residual. The purpose of selective sequential extractions is to provide detailed information on metal origin, biological and physicochemical availability, mobilization, and transport (Tessier et al, 1979). Typically, the first step in a sequential extraction process extracts the most labile metals, and each successive step increases in strength until the most non-labile fraction is left.

According to Tessier et al,

- the exchangeable fraction is likely to be affected by changes in water ionic composition as well as sorption-desorption processes;
- the carbonate fraction or specifically adsorbed fraction is susceptible to changes in P^H;
- the reducible fraction consists of iron and manganese oxides which are unstable under anoxic conditions;
- the organic fraction can be degraded leading to a release of soluble metals under oxidizing conditions;
- the residual fraction contains mainly primary and secondary minerals, which may hold metals within their structure i.e. they are bound to soil matrix. These metals are not expected to be released in solution over a reasonable time span under the conditions normally encountered in nature.

Studies on determination of heavy metal concentration in samples of roadside dusts have been carried out on some locations in Nigeria. The present study, however, is designed to determine the levels of heavy metal contamination in the surface soils of some major roads in Abeokuta, Ogun State Nigeria, and equally to assess their health impact on human. The results obtained in the study will help to identify and quantify the species of elements in the sampled areas.

This study will provide information on the levels of heavy metal contamination in the soils, arising from human activities including industrialization and urbanization of the areas under study. It is expected to reveal the potential health hazards of these metals and preventive measures that could be taken to safeguard life and the environment.

The information provided by this study will serve to educate the public on the impact of contamination of the environment and steps that could be taken to prevent pollution of the environment. The study will also add to available data on heavy metal toxin levels in the environment.

2. EXPERIMENTAL

2.1. Study Area and Sampling Procedure

This study was carried out on roadside dusts from Abeokuta. Roadside dust samples were taken from five major roads in the area 10 metres away from each other. The dust samples were made into a composite to give a representative sample and stored in a polythene bag. The samples were taken to the laboratory for analysis.

2.2. Apparatus and Reagents

All glassware, polypropylene tubes, polyethylene sample bottles and Teflon beakers used were first washed with liquid detergent , rinsed with tap water and distilled water, and then soaked in 10% HNO_3 (v/v) for 48 hours. They were then rinsed thoroughly with doubly distilled water. Thereafter, the apparatus were oven dried for 12 hours at a temperature of 80^oC.

All reagent used were of analytical grade. They included nitric acid, HNO_3 (BDH Chemical Ltd, Poole, England), hydrochloric acid, HCl (BDH Chemical Ltd, Poole, England), hydrofluoric acid, HF (Riedel-deHaën, Germany), acetic acid, CH_3COOH (BDH Chemical Ltd, Poole, England), ammonium acetate, CH_3COONH_4 (M&B Ltd, Dagenham England), hydrogen peroxide, H_2O_2 (BDH Chemical Ltd, Poole, England), magnesium chloride, $MgCl_2$ (M&B Ltd, Dagenham England). Solutions were prepared using doubly distilled water.

2.3. Sample Pre-Treatment

The collected composite dust samples were air dried for two weeks in an aerated cupboard to reduce cross-contamination. The sample was sieved to remove large gravel-sized particles before analysis.

2.4. Sequential Extraction of Trace Metals from the Roadside Dusts

The sequential extraction procedure of Tessier et al (1979) was used for the extraction and partitioning of Al, Cd, Cu, Mn, Pb and Zn into exchangeable, bound to Carbonate, bound to Fe-Mn Oxides, bound to organic matter and residual fraction. A gram of the air-dried and homogenized dust sample of ≤ 2.0 mm diameter particle size was subjected to various leaching treatments to separate the heavy metals into five operationally-defined fractions. All extractions were performed in 50 mL polypropylene centrifuge tubes. The resulting supernatant aqueous layer was carefully filtered into analytical vials and taken for AAS determination of Al, Cd, Cu, Mn, Pb and Zn. The operationally-defined fractions are as follows:

2.5. Exchangeable fraction (F1)

The weighed sample was extracted at room temperature with 20 mL 1M $MgCl_2$ (P^H 7) with continuous agitation for 1hour. It was later centrifuged, filtered and the collected solution made up to mark in a 25ml standard volumetric flask for AAS analysis.

2.6. Carbonate Fraction (F2)

The residue from F1 was treated at room temperature with 20 mL 1M NaOAc adjusted to P^H 5 with HOAc. Continuous agitation was maintained for 5 hours, followed by centrifuging and filtration. The collected solution was made up to mark in a 25ml standard flask and analyzed for the trace metals with AAS.

2.7. Fe-Mn Oxides-bound Fraction (F3)

The residue from F2 was extracted with 40 mL 0.04 M $NH_2OH.HCl$ in 25% (v/v) HOAc at 96 ± 1^oC with occasional agitation for 5 hours. The remaining solution was centrifuged while the resulting aqueous layer was filtered into a 25 ml standard flask and taken for AAS determination.

2.8. Bound to Organic Matter fraction (F4)

To the residue from F3 were added 7.5 mL of 0.02 M HNO_3 and 12.5 mL of 30% H_2O_2, adjusted to P^H 2 with HNO_3. The mixture was heated to 85^oC for 2 hours with occasional agitation. A second 7.5 mL aliquot of 30% H_2O_2 was added and heated further for 3hours at 85°C with intermittent agitation. After cooling, 12 mL of 3.2M NH_4OAc in 20% (v/v) HNO_3 was added to prevent the adsorption of extracted metals into the oxidized sediments and the mixture was diluted to 40 mL. Continuous agitation for 30 minutes followed.

2.9. Residual Fraction (F5)

The residue from F4 was digested with concentrated HNO_3 and later refluxed for 1hour, filtered and washed with 10 mL of hot 3M HNO_3.

2.10. Description of Study Area and its Suitability

Abeokuta is geographically located in southwest Nigeria with a population of 888,924 (National Population Census 2012). The sampling sites for the study were along Bara round-about, Sokori road, Abeokuta North Local Government area. The study area lies within longitudes 004^0 32′15′′ to 004^0 33′36′′E and latitudes 7^0 9′39′′N to 3^0 20′54′′N, with a land elevation of 66 m (217ft) above sea level. A map of the study area is presented in Figure 1.

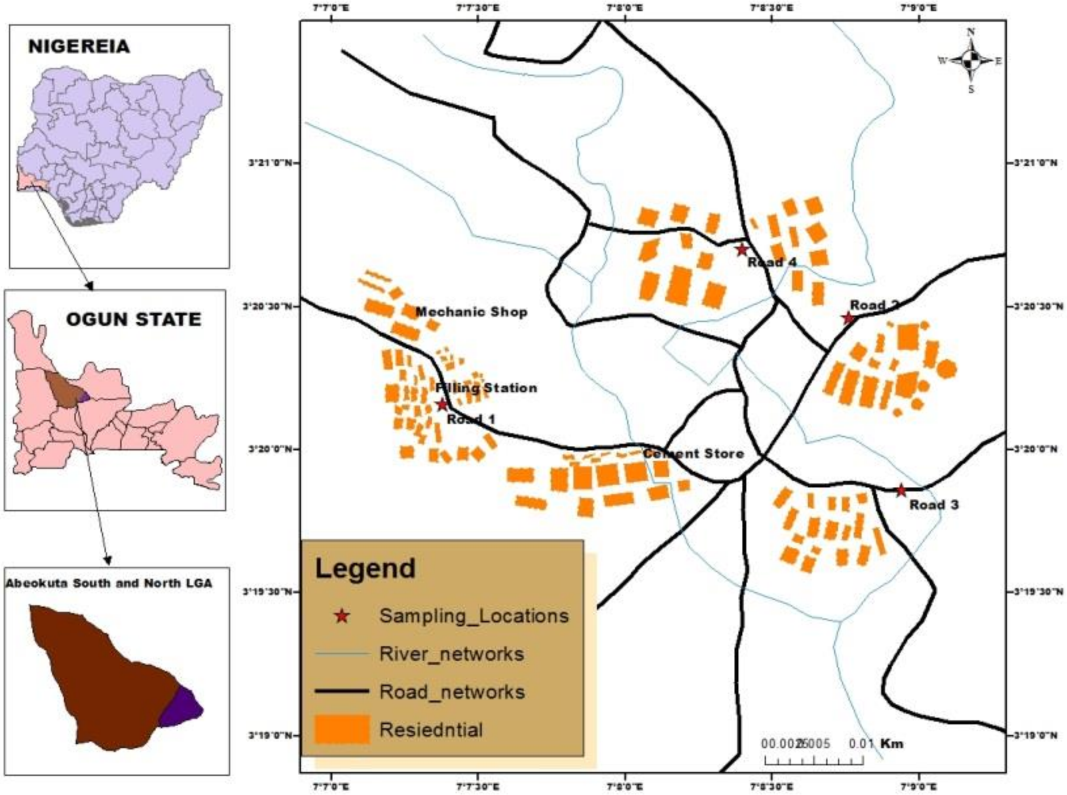

Figure 1 Map showing the sampling locations in Abeokuta Metropolis, Nigeria

2.11. Mobility Factor (MF) Determination

The relationship adopted by Ogunfowokan et al, (2009) was used to determine the mobility factor of the metals. This relationship is as follows:

$$MF = \frac{F_1 + F_2}{F_1 + F_2 + F_3 + F_4 + F_5} X100$$

Where F_1 = Exchangeable metal content fraction

F_2 = Metal content bound to carbonate fraction

F_3 = Metal content bound to Fe-Mn oxide fraction

F_4 = Metal content bound to organic matter fraction
F_5 = Residual metal content fraction.

2.12. Coefficient of Variation (CV)

The CV was calculated in order to determine temporal and spatial variations of the various metal fractions from one location to the other. The CV values were obtained from the relationship:

$$CV = \frac{SD}{x} X\ 10$$

Where SD is standard deviation and x is the mean.

3. RESULTS AND DISCUSSION

3.1. Total Metal Determination in Roadside Dusts

The total contents of the metals namely: Zn, Pb, Cu, Mn, Cr, Cd, and As in the roadside dust samples of the study areas in Abeokuta North local government area are shown in Table 1.

The concentration of Zn in the investigated samples was 164.83µg/g (Table 1). Zn has the highest concentration and this makes the metal a serious environmental concern in the area investigated. The level obtained was higher than that reported earlier in 2012 by Babatunde and Oladele in their study of heavy metal concentrations of roadside dusts of different traffic density in Abeokuta. Zinc is considered essential to plant growth and physiological function of organisms. However excessive exposure to Zinc poses a serious health concern.

Chromium had the second highest value with a concentration of 101.3µg/g. This was followed by Manganese and Lead, with concentrations of 87.03 and 80.33 respectively. Copper, Cadmium and Arsenic had relatively low concentrations with 19.90, 10.53 and 2.60 respectively.

The presence of Cd in the roadside surface soils could also be due to attrition of automobile tyres (Akhter and Madany, 1993), careless disposal of Cadmium-containing batteries and corrosion of Cadmium alloy. Table 1 also compares the maximum permissible limits of heavy metals in soils as set by standard regulatory bodies such as World Health Organization (WHO), Food and Agricultural Organization (FAO) and Ewers U, Standard Guidelines in Europe, with total metal concentrations in the study samples. With the exemption of Chromium and Cadmium, the values reported for all the other investigated metals revealed lower concentrations of the metals than the maximum permissible limits set.

Table 1 Comparison of total metal concentrations (µg/g) with permissible limits

Metal	Zn	Pb	Cu	Mn	Cr	Cd	As	Total
Concentration	164.83	80.33	19.90	87.03	101.03	10.53	2.60	466.25
Permissible Limit	300	100	100	2000	100.0	3.0	20.0	

3.2. Speciation of Metals in Soil Samples

Results of speciation studies conducted on the roadside dust samples of Abeokuta are as shown in Table 2. Metal chemical speciation carried out by sequential extraction of the metals is essential to the metal mobility (Tessier *et al.*, 1979). The order of mobility of the metals considering their abundance in the fractions is: exchangeable > bound to carbonate > bound to Fe-Mn oxide > organic matter > residual (Tessier *et al.*, 1979). The chemical forms of the metal control its bioavailability or mobility.

Zinc

The results of speciation analysis of Zn in the sample gave the amount of Zn in the following decreasing order: bound to carbonate (225.35µg/g) > bound to organic matter (33.60µg/g) > bound to Fe-Mn Oxide (15.50 µg/g) > exchangeable (2.05 µg/g) > residual (1.98 µg/g). The low content of Zn in residual fraction of the sampled area may be due to low retention of silicates in residual fraction.

Lead

The results of speciation analysis of lead in the sample showed that the levels of Pb in the various fractions were of this order: bound to carbonate (168.95 µg/g) > exchangeable (42.35 µg/g) > bound to Fe-Mn oxide (31.15 µg/g) > bound to organic matter (28.33 µg/g) > residual (12.38 µg/g). The high Pb level in carbonate fraction of the sampled area of Abeokuta indicates its potential environmental leachability (Hsu and Lo, 2000). Acid environment enhances the mobility of metal in the environment. Metals bound to carbonate and residual phases are sensitive to P^H changes and they are leached by lowering the P^H (Pere-Cid *et al.*, 1999). The low content of Pb in residual fraction of the sampled area may be due to low retention of silicates in residual fraction.

Manganese

The results of speciation analysis of Mn revealed that the levels of the metal were in the order: bound to carbonate (217.18 µg/g) > exchangeable (31.4 µg/g) > residual (29.3 µg/g) > bound to Fe-Mn oxide (18.65 µg/g) > bound to organic matter (7.13 µg/g). The high Mn level in the sampled area resided in the carbonate bound and exchangeable. This may be as a result of high association or retention ability of the mineral crystal structure such as detrital silicates and resistant sulphide. This means Mn was more mobile and available than other metals in this environment. The high level of Mn in carbonate bound in the sampled area may be attributed to the ease of complexation between the metal and carbonates.

Copper

The results of speciation analysis of Cu revealed that the levels of the metals were in the order: bound to carbonate (46.50 µg/g) > residual (19.90 µg/g) > bound to organic matter (8.08 µg/g) > exchangeable (5.25 µg/g) > bound to Fe-Mn oxide (4.53 µg/g). The high levels of the metal in the carbonate and residual fractions may be as a result of high retention of silicates in residual fraction and its potential environmental leachability in bound to carbonate fraction (Hsu and Lo, 2000). Acid environment enhances the mobility of metal in the environment. Metals bound to residual and carbonate phases are sensitive to pH changes and they are leached by lowering the pH (Pere-Cid *et al.*, 1999). Metals present in the residual fractions are a measure of the degree of environmental pollution. The higher the metals present in this fraction, the lower the degree of pollution (Banat, 2001).

Cadmium

The result of speciation analysis of Cd gave the amount of Cd in the following decreasing order: bound to organic matter (11.40 µg/g) > bound to carbonate (7.88 µg/g) > residual (3.95 µg/g) > exchangeable (1.75 µg/g) > bound to Fe-Mn oxide (0.45 µg/g). The high level of Cd in the organic matter fraction in the sampled area may be attributed to the ease of complexation of products formed between the metal and natural organic matters such as humic acids and fulvic acids.

Chromium

The result of speciation analysis of Cr gave the amount of Cr in the following decreasing order: residual (92.00 µg/g)> exchangeable (34.75 µg/g) > bound to carbonate (16.20 µg/g) > bound to organic matter (13.93 µg/g) > bound to Fe-Mn oxide (4.08 µg/g).

Arsenic

The result of speciation analysis of Cr gave the amount of Cr in the following decreasing order: bound to organic matter (1.73 µg/g) > bound to carbonate (0.60 µg/g) > bound to Fe-Mn oxide (0.50 µg/g) > residual (0.35 µg/g) > exchangeable (0.10 µg/g).

Table 2 Concentration of Metals (µg/g) in various Chemical Fractions of soil samples

Fractions/Metals	F1 (Exchangeable)	F2 (Carbonate Bound)	F3 (Fe-Mn Oxide Bound)	F4 (OM Bound)	F5 (Residual)	Total
Zn	2.05	225.35	15.50	33.60	1.98	278.48
Pb	42.35	168.95	31.15	28.33	12.38	283.16

Cu	5.25	46.10	4.53	8.08	19.90	75.86
Mn	31.4	217.18	18.65	7.13	29.93	304.29
Cr	34.75	16.20	4.08	13.93	92.00	160.96
Cd	1.75	7.88	0.45	11.40	3.95	25.43
As	0.10	0.60	0.50	1.73	0.35	3.28
Overall Mean ±S.D	16.81±18.46	97.47±102.02	10.69±11.51	14.89±11.72	22.93±32.27	162.79
CV (%)	109.82	104.67	107.67	78.71	140.73	541.60
Percentage Fraction	20.30	19.33	19.88	14.53	25.98	

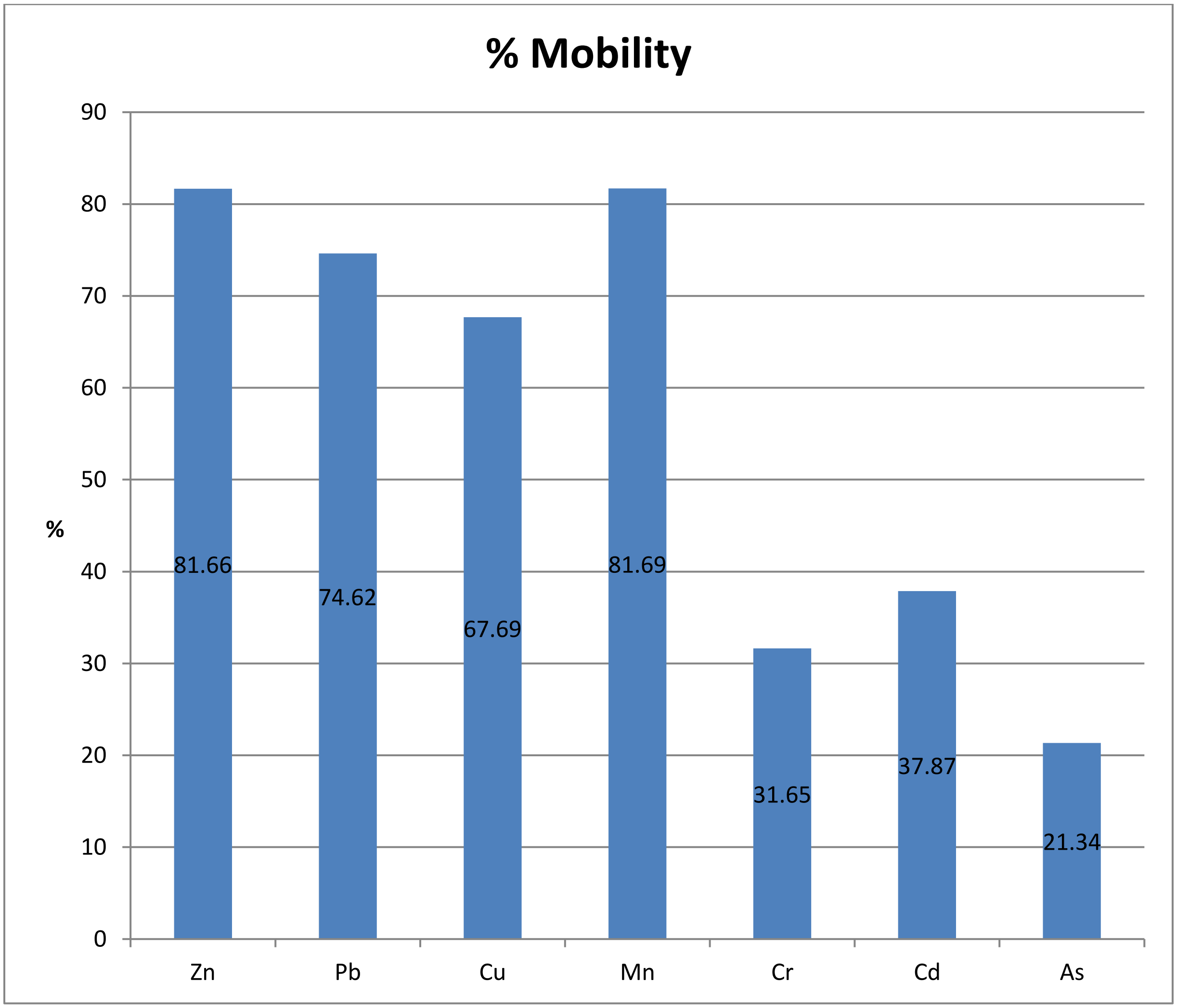

Figure 2 Chart showing percentage mobility of the metals

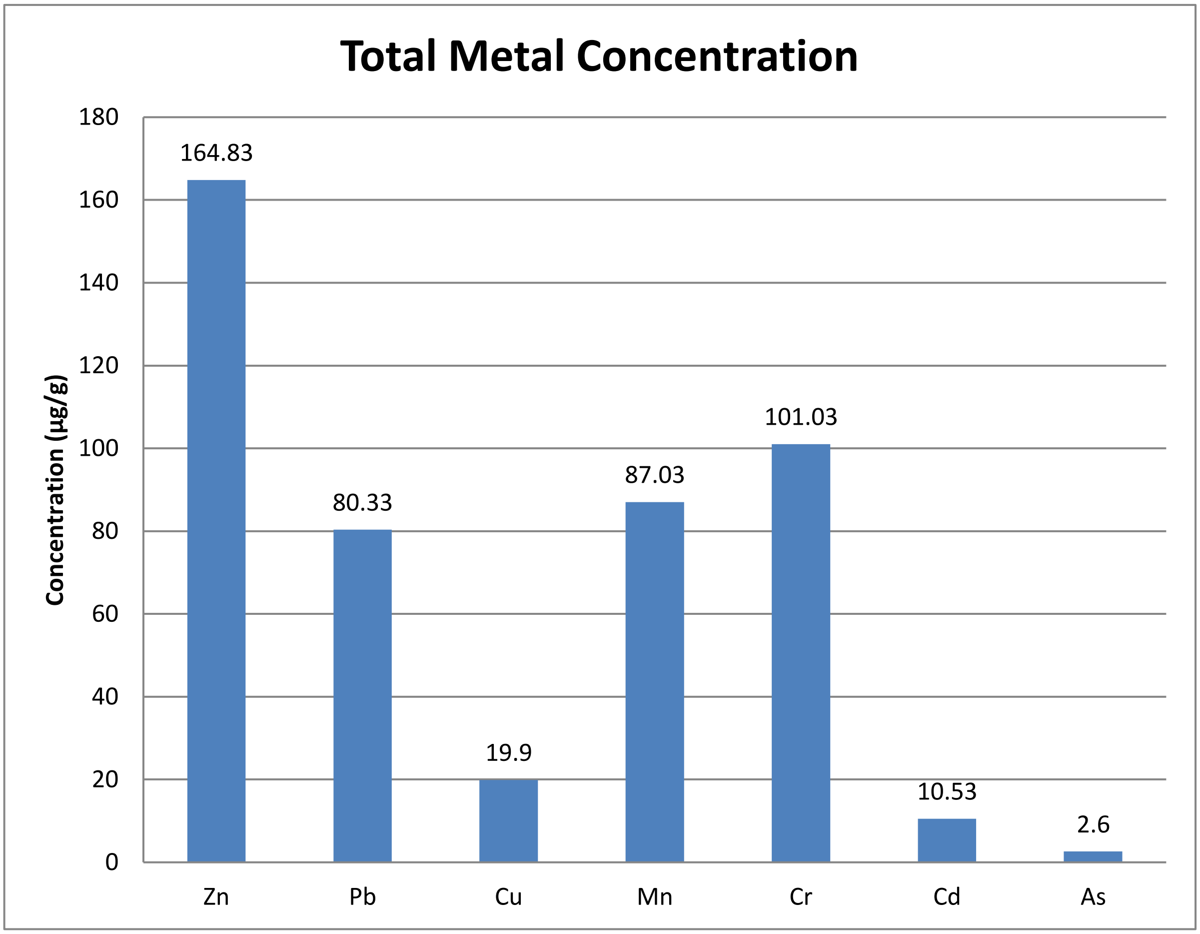

Figure 3 Chart showing total metal concentrations of soil samples

3.3. Mobility Factor (% MF) of Metals in the Soils

The relatively high level of Pb in the sampled area gives a great cause for concern given the serious effects Pb exposure tends to have on human health. The high MF value of this metal may be due to automobile workshops, filling stations and motor parks in the area. The mobility factor (MF) for Mn in this study was higher than those of the other metals investigated. Hence, Mn is bioavailable in the sampled area. However, in terms of toxicity, levels of Pb and Cd give a cause for concern even at lower concentrations compared to Mn, and their effects may be seven-fold higher than those of the Mn equivalents.

Although Mn is primarily considered an essential element, excessive exposure to it can lead to very serious health problems (Keen and Ziderberg- Cherr, 1993). High Mobility Factor values are an indication of lability and biological availability of heavy metals (Salbu *et al.*, 1998). Cd has no biological function and toxic at all levels (Badawy *et al.*, 2002).

The relatively low MF values reported for Cd, Cr and As in the sampled area indicate high stability of these metals in the sample. A high % mobility factor (MF) value for heavy metals in sediments has been interpreted as an evidence of relatively high lability and biological availability (Narwal *et al.*, 1999; Kabala and Singh, 2001).

The relatively high mobility factor (MF) observed is in agreement with the high percentage of exchangeable fraction recorded from the chemical fractionation results. High mobility factor (MF) indicates a correspondingly high vulnerability of living things generally to heavy metals (Ogunfowokan *et al.*, 2009). Generally, the mobility factors were less than 100, indicating low pollution due to the metals.

Table 3 Mobility Factor (% MF) of Metals in Soils of study sample

Metal	Zn	Pb	Cu	Mn	Cr	Cd	As
Mobility Factor (%)	81.66	74.62	67.69	81.69	31.65	37.87	21.34

4. CONCLUSION

A five-stage sequential extraction procedure by Tessier et al., (1979) was adopted to fractionate heavy metals in the roadside dusts of Abeokuta. The levels of Zn, Pb, Cu, Mn, As, Cr and Cd in the geochemical fractions evaluated for the soils of the sampled area were in the following orders:

Zinc: bound to carbonate (225.35µg/g) > bound to organic matter (33.60µg/g) > bound to Fe-Mn Oxide (15.50 µg/g) > exchangeable (2.05 µg/g) > residual (1.98 µg/g).
Lead: bound to carbonate (168.95 µg/g) > exchangeable (42.35 µg/g) > bound to Fe-Mn oxide (31.15 µg/g) > bound to organic matter (28.33 µg/g) > residual (12.38 µg/g).
Manganese: bound to carbonate (217.18 µg/g) > exchangeable (31.4 µg/g) > residual (29.3 µg/g) > bound to Fe-Mn oxide (18.65 µg/g) > bound to organic matter (7.13 µg/g).
Copper: bound to carbonate (46.50 µg/g) > residual (19.90 µg/g) > bound to organic matter (8.08 µg/g) > exchangeable (5.25 µg/g) > bound to Fe-Mn oxide (4.53 µg/g).
Cadmium: bound to organic matter (11.40 µg/g) > bound to carbonate (7.88 µg/g) > residual (3.95 µg/g) > exchangeable (1.75 µg/g) > bound to Fe-Mn oxide (0.45 µg/g).
Chromium: residual (92.00 µg/g) > exchangeable (34.75 µg/g) > bound to carbonate (16.20 µg/g) > bound to organic matter (13.93 µg/g) > bound to Fe-Mn oxide (4.08 µg/g).
Arsenic: bound to organic matter (1.73 µg/g) > bound to carbonate (0.60 µg/g) > bound to Fe-Mn oxide (0.50 µg/g) > residual (0.35 µg/g) > exchangeable (0.10 µg/g).

There were indications that the levels of Zn, Pb, Mn, and Cu detected for the sampled areas were mainly associated with the carbonate bound fraction in the soil samples. The high levels of some of the metals investigated in the study area is a cause for concern as these metals can accumulate to pollute the environment and damage human health. The distribution of the metals in the various fractions and results of mobility factors confirmed their differences in mobility and bioavailability. There was also an indication that sources of these metals were mainly anthropogenic.

RECOMMENDATIONS

In view of the findings in this study, the following recommendations are therefore made:

1. The Ministry of Environment should ensure effective waste management practices to prevent the accumulation of these metals and protect our environment.
2. Exhausts from factories and vehicle engines should be properly controlled to ensure a clean and safe environment.
3. Once metals are introduced into the environment, they are very difficult to eliminate. They do not degrade like carbon-based compounds. Hence concerted and collaborative efforts should be made by government, the industry and the academia to help prevent soil contamination.
4. Government at all levels should legislate against indiscriminate dumping of pollutants in the environment to prevent the accumulation of these metals with their attendant health effects.
5. Management practices such as high temperature treatment and leaching out contaminant can be adopted to remediate and decontaminate polluted soils.
6. Further investigations on environmental pollution should be carried out often in order to monitor the variation in levels of trace metals.

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