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Geochemical Partitioning of Metal Pollutants in Agricultural Soil as Index of Human Health Risk Assessment

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Abstract Bioavailability and toxicity of metals depend not only on their total concentrations but also on the form in which they exist. Therefore full understanding of the metals' behavior and prediction of their potential risk in agricultural soil is possible only when all the geochemical forms in which they exist under different environmental conditions are identified. The aim of this study was to assess the geochemical partitioning of Cd and Pb in agricultural soil on the bank of Delimi River in Nigeria as an index of predicting their bioavailability to crop and potential toxicity to humans. The study area comprised of three farmlands irrigated with polluted water from River Delimi while the control area was a farmland irrigated with water from Lamingo Dam (a municipal water supply reservoir) all within Jos city. Total metals content in soil collected in the rhizosphere of Carrot and Cabbage were extracted using aqua-regia. A five-step sequential extraction procedure was used to determine the partitioning of the metals into different geochemical fractions of the soil. The metals extracted were determined using Atomic Absorption Spectrophotometer (AAS). The partitioning of both Pb and Cd into different geochemical fractions of soil showed that the samples collected on farmland at the study site were mostly partitioned to exchangeable and other non-residual fractions that can easily be released for uptake by crops. At the control site however the metals were mainly occluded to residual non-mobile fractions. It may be concluded that there is a higher risk of metal toxicity among people that consume crops cultivated on farmlands along Delimi River than those at Lamingo Dam.

Keywords: *geochemical partitioning, metal pollutants, soil, risk assessment*

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1. Introduction

Soil does not only serve as a medium upon which plants grow, but also provides habitat for animals and other micro-organisms. Fertile soil is vital for the production of food, timber and fibre, which are all essential for human existence and economic prosperity. Soil therefore, constitutes part of vital ecological and agricultural resources that need to be protected.

Heavy metals from various sources may finally reach the surface soil, and their fate depends on soil physical and chemical properties especially their speciation. Heavy metal pollution of soil is believed to be a long-term threat to the environment. The accumulation of heavy metals in agricultural soil has attracted the attention of many investigators not only because metals can build up in the soil but also because they can be transferred to crops, where they constitute a significant potential risk to human health [1,2].

Plant species differs in their ability to mobilize Cd and Pb bound in soils. Cd could accumulate in plants which are not toxic to them yet could be quite toxic to humans and other animals eating such plants. Toxicity of Cd is however more in humans than other animals, because of its longevity and the accumulation in their essential organs. The toxicity of Cd could result in painful bone demineralization (osteoporosis) due to the replacement of Ca by Cd in bones. Other health effects caused by Cd include, diarrhoea, stomach pains and severe vomiting, reproductive failure, damage to the central nervous system, damage to the immune system, psychological disorders, and possibly DNA damage or cancer development [3,4].

According to [5] signs of chronic lead in humans include tiredness, sleeplessness, irritability, headaches, joint pain, and gastrointestinal disorder. Other symptoms include muscle weakness, disturbances in mood, and symptoms of peripheral neuropathy especially in occupationally exposed individuals [6]. In general, central and peripheral nervous systems are principal targets for lead toxicity [7]. Pb toxicity is especially more in children and brings in them behavioral disturbances and learning difficulties [8].

It should be noted however that the bioavailability and risk associated with metal do not necessarily depend on the total concentration in soil [9], what matters most is the partitioning of the metal to various geochemical fractions of the soil [10]. Thus, in soil, heavy metals may be found in one or more of the following geochemical fractions namely: exchangeable, carbonate bound, Fe/Mn oxide bound, organic matter bound as well as the residual fraction. The exchangeable and to some extent carbonate fractions are mobile and easily bioavailable. The Fe/Mn oxide and organic matter bound fractions are leached out only under extreme conditions while the residual fraction is almost inert towards bioavailability [11]. In other words the risk of metal transfer from soil to crop is high if it is mostly bound to exchangeable fraction. The risk is low when the metal is mostly found in either carbonate, Fe/Mn or organic fractions. No risk is however expected if the metal is bound to the residual fraction.

The aim of this study was to assess the geochemical partitioning of Cd and Pb in agricultural soil on the bank of Delimi River in Nigeria as an index of predicting their bioavailability to crop and potential risk of toxicity to consumers.

2. Materials and Methods

2.1. Study Area

The study was conducted at designated irrigation farmlands along River Delimi in Jos metropolis (Figure 1). Jos is a city in the north-central Nigeria and the capital of Plateau State. It is located on Lat. 9^0 52’59’’N and Long. $8^0$54’26’’E. The meteorological data for Jos recorded in the year 2009 and 2010 is presented in Table 1.

Table 1. Mean monthly meteorological data in Jos for 2009 and 2010

Month	2009			2010		
	Temperature (O°C)	Rainfall (mm)	Relative Humidity (%)	Temperature (O°C)	Rainfall (mm)	Relative Humidity (%)
January	29.40	0.00	19.56	28.50	0.00	21.48
February	29.20	0.00	19.22	27.40	0.00	16.81
March	32.40	10.00	24.56	30.70	28.80	26.85
April	31.60	87.00	49.40	31.70	78.50	46.08
May	28.80	137.10	70.10	28.20	225.60	75.74
June	27.20	129.50	74.80	26.60	218.10	78.96
July	25.00	167.80	78.15	24.70	257.90	80.20
August	25.00	299.40	79.20	23.60	346.80	82.70
September	26.60	190.30	76.30	25.70	230.90	79.00
October	28.00	195.80	62.43	27.60	83.20	75.50
November	28.40	30.40	45.50	27.10	0.00	37.45
December	28.00	0.00	24.02	28.00	0.00	23.80
Total	339.60	1246.80	623.24	329.80	1469.80	644.57
Mean	28.30	103.90	51.93	27.48	122.48	53.71

Source: National Center for Remote Sensing Jos [12].

2.2. Sampling and Sampling Strategy

Three farmlands located at Gangare (station I), Farin gada (station II) and the new campus of University of Jos (station III) along River Delimi were selected for the study. At each of the sampling stations, soil samples were collected in the rhizospheres of Carrot and Cabbage cultivated in the farmlands. The soil samples in the vicinity of the crops were also collected at another farmland (station IV) along Lamingo dam which serves as control station. Soil samples were obtained with soil auger at a depth not exceeding 15cm [13,14]. At each of the sampling stations, soil samples were collected randomly at three points with three replications each during dry seasons of 2009 and 2010.

2.3. Samples Preservation

The soil samples were collected in polythene bags and kept on ice. The samples were subsequently transported to the laboratory and kept at 4°C [15].

2.4. Pre-treatment of Samples

Soil samples meant for total metal analysis were placed in porcelain crucibles and oven dried at 80°C for 24 hours according to the procedures. The dried samples were ground using a previously acid washed porcelain mortar and pestle. The samples were then kept in desiccators to attain constant weights before being transferred into air-tight plastic bottles. All the samples were sieved with a 200µm sieve before metal analysis.

2.5. Soil Analysis

2.5.1. Digestion of Soil Samples for Total Metal Concentration

0.5g each of the sieved soil samples were placed into 100ml beaker and moistened with few drops of distilled water. 5ml of Aqua-regia (a combination of HNO_3 and HCl in the ratio 1:3) was then added. The beaker was covered with a watch glass and placed on a hot plate in a fume cupboard. The mixture was boiled on a hot plate and allowed to simmer for 45 minutes. The mixture was removed from the hot plate and placed on a heat-proof mat where it was allowed to cool. The watch glass was removed allowing any liquid to drain into the beaker. The content of the beaker was filtered through a Whatman 541 filter paper into 100ml volumetric flask. The filtrate was made up to the mark with distilled water. The volumetric flask was then inverted several times to ensure mixing and homogenization of the solution. The solution was then transferred into a labeled sample bottle and was analyzed for heavy metal content using Atomic Absorption Spectrophotometer (AAS) [16]. The accuracy of the methods used was guaranteed through double determinations and use of blanks for correction of background and other sources of error.

2.5.2. Geochemical Partitioning of Metals in Soil

1g each of the air dried soil samples were extracted in accordance with Tessier's extraction scheme [17]. At the end of each extraction step, the samples were centrifuged for 10 minutes and the supernatant decanted, filtered and analyzed for metal content using Atomic Absorption Spectrophotometer (AAS). Each step was followed by a wash stage in which 8ml of deionized water was added to the residue and centrifuged for 10 minutes to remove any trace of previous extracting solution and the supernatant discarded. The extractions were carried out as follows;

2.5.2.1. Exchangeable fraction

1g each of the air dried soil samples were leached with 8ml of 1M $MgCl_2$ at pH 7 (adjusted with NaOH) for 1hr with continuous shaking using a mechanical shaker. The solution was then filtered using a Whatman filter paper No. 1 into a 100ml volumetric flask. The filtrate was made up to the mark with distilled water.

2.5.2.2. Carbonate bound fraction

8ml of NaOAc at pH 5 (adjusted with acetic acid) was added to the residue obtained from exchangeable fraction and leached for 5hrs at room temperature with continuous agitation using a mechanical shaker. The leachate was filtered and transferred into a 100ml conical flask and made up to the mark with distilled water.

2.5.2.3. Iron-Manganese oxide fraction

To the residue obtained from the carbonate fraction, 20ml of 0.04M $NH_2OH.HCl$ (Hydroxylamine hydrochloride) in 25% acetic acid (v/v) was added. The sample was placed in a water bath at 90°C and leached for 6hrs with occasional shaking. The leacheate was then transferred into a 100ml volumetric flask and made up to the mark with distilled water.

2.5.2.4. Organic bound fraction

To the residue obtained from the Iron-Manganese fraction, 3ml of 0.02M nitric acid (HNO_3) and 5ml of 30% hydrogen peroxide (H_2O_2) at pH 2 (adjusted with HNO_3) were added. The mixture was heated in water bath at 85°C for 2hrs with occasional shaking. 9ml of 30% H_2O_2 at pH 2 was further added and the mixture continuously heated at 85°C for 3hrs. Another 9ml of 3.2M ammonium acetate CH_3COONH_4 in 20% (v/v) HNO_3 was then added and the mixture shaken for 0.5hr at a low temperature of about 25°C. The supernatant was filtered and transferred into a 100ml volumetric flask and made up to the mark.

2.5.2.5. Residual fraction

The concentration of metals in the residual fraction of soil was calculated by subtracting the concentration in the first four fractions from the total content previously determined by the aqua-regia digestion [18].

2.3. Statistical Analysis

The experimental design was a complete randomized design involving four sampling stations with the soil samples collected randomly at three points with three replications each. The soil samples were collected during dry seasons of 2009 and 2010. Tests for significant difference were carried out using the Analysis of Variance (ANOVA) of the Statistical Package for Social Sciences (SPSS) computer programme. Means were separated using the Tukey test.

3. Results and Discussion

3.1. Total Metals Concentrations in Soil

The mean concentrations of Pb and Cd recorded in soils collected under Carrot and Cabbage obtained from farmlands along River Delimi (study area) and Lamingo Dam (control site) were presented in Table 2.

Table 2. The mean concentrations (±SD) of Pb and Cd (mg/kg) in soil collected under Carrot and Cabbage from the four sampling stations at River Delimi and Lamingo Dam in Jos metropolis

Station	Soil collected under Carrot		Soil collected under Cabbage	
	Pb	Cd	Pb	Cd
I	$1.873(\pm0.375)^a$	$1.293(\pm0.328)^a$	$1.877(\pm1.102)^a$	$1.205(\pm0.446)^a$
II	$1.058(\pm0.743)^a$	$1.143(\pm0.671)^a$	$1.792(\pm1.089)^a$	$1.132(\pm0.440)^a$
III	$1.522(\pm1.168)^a$	$1.140(\pm0.402)^a$	$1.387(\pm1.181)^a$	$1.247(\pm0.205)^a$
IV	$0.527(\pm0.231)^b$	$0.583(\pm0.289)^b$	$1.212(\pm0.440)^a$	$0.752(\pm0.277)^a$
USEPA LIMITS	10.0	0.1	10.0	0.1

Means followed by same letter within column are not statistically different at 95% level of confidence.

The statistical analysis has revealed a significant variation ($p \leq 0.05$) in the mean concentration of Cd in soils collected under carrot obtained from the various sampling stations. The mean Cd concentrations in soil obtained from stations I (1.293mg/kg), II (1.143mg/kg), and III (1.140mg/kg) of the study area were statistically similar ($p > 0.05$), but significantly higher ($p \leq 0.05$) than 0.583mg/kg obtained from station IV of the control site. Similarly, significantly higher ($p \leq 0.05$) mean Pb concentrations were recorded in soils obtained from the study compared to the control area. Stations I, II, and III with mean Pb values of 1.873mg/kg, 1.058mg/kg, and 1.522mg/kg respectively are statistically similar ($p > 0.05$) but significantly higher ($p \leq 0.05$) than the mean value of 0.527mg/kg obtained in samples at station IV. Similarly, no significant difference ($p > 0.05$) was observed in the mean concentration of Cd in soil samples collected under cabbage in all the four sampling stations. However, more Cd were recorded in soil samples obtained from station I, II and III (study area) with mean values of 1.205mg/kg, 1.132mg/kg and 1.247mg/kg respectively compared to a value of 0.752mg/kg recorded in soil obtained from station IV of the control area. Similarly, no significant difference ($p > 0.05$) was observed in the mean Pb concentration recorded in soil samples collected under the cabbage obtained from the various sampling stations. However, a steady decrease in the mean concentrations of this metal was observed from stations I – V. Thus the mean Pb concentrations were found to be 1.877mg/kg, 1.792mg/kg, 1.387mg/kg and 1.212mg/kg for stations I, II, III, and IV respectively.

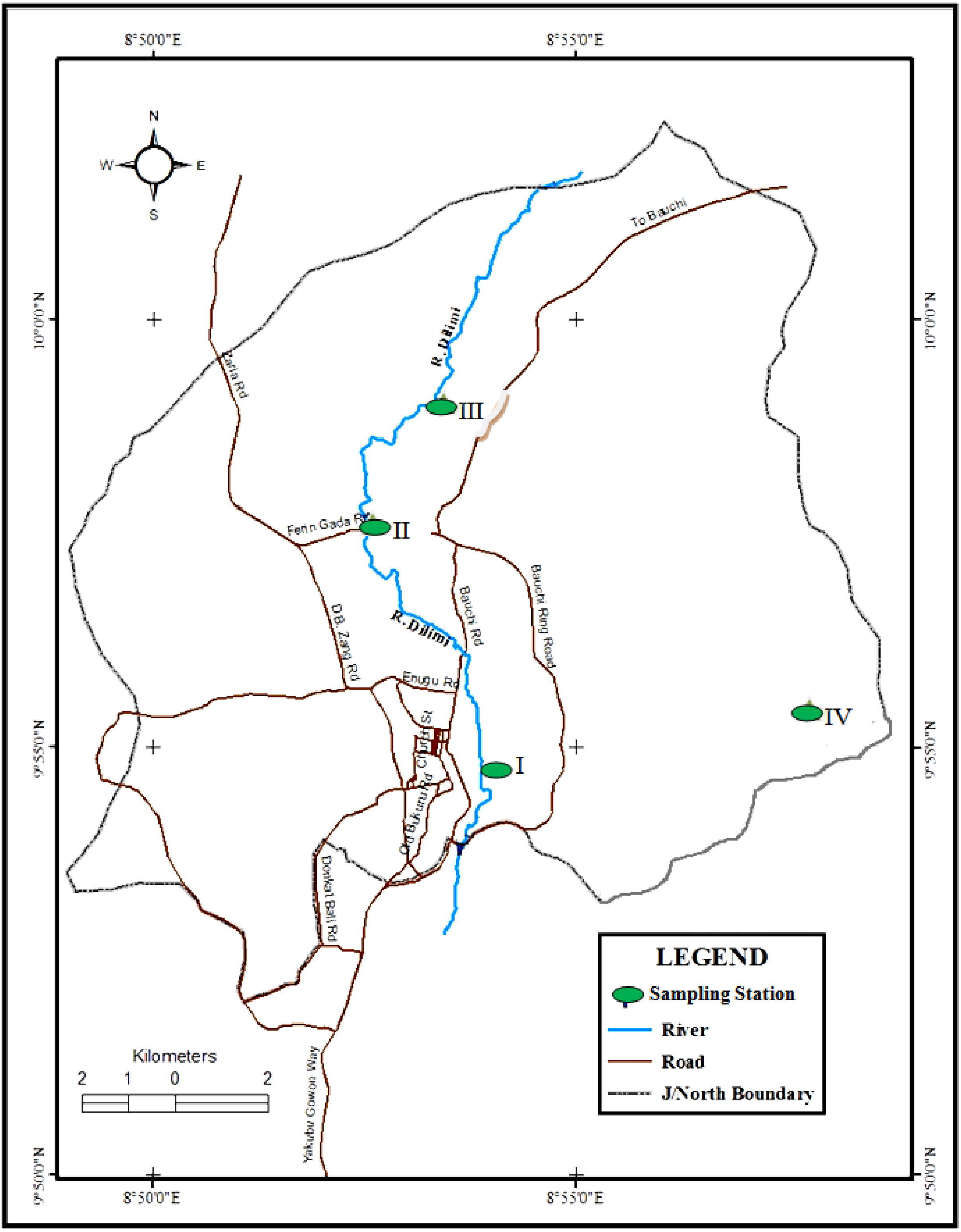

Figure 1. Map of Jos metropolis showing the sampling stations

The observed high metal concentrations in soils obtained at the study compared to the control site may not be unconnected with the use of contaminated water obtained from the Delimi River by the farmers to irrigate their farmlands. This is especially so considering the fact that the levels of all the metals in the river water used to irrigate the crops were also higher in the study compared to those in the control site [19]. Indiscriminate dumping of various kinds of domestic and industrial wastes at the study site accounts for the high level of contamination of the river water whose long term application on to the soil increases its metal contents. Contrary to what was observed at the study site, there was no intentional dumping of waste at the control site hence the lower levels of the pollutants in water and soil of the irrigated farmland. Compared to the standard for heavy metals in soil given by [20], the concentrations of Cd in soils collected under vegetable crops obtained from farmland at the study site along River Delimi have exceeded the recommended threshold limit of 0.1mg/kg. The concentration of the metal in samples from the control site however falls within the recommended limit. The mean Pb concentration from both the study and control sites did not exceed the recommended limit of 10.0mg/kg given by USEPA.

3.2. Partitioning of Metals in Soil Collected under Carrot

3.2.1. Partitioning of Lead in Soil Collected under Carrot

The partitioning of Pb into the different geochemical fractions in soil collected under carrot is as shown in Figure 2.

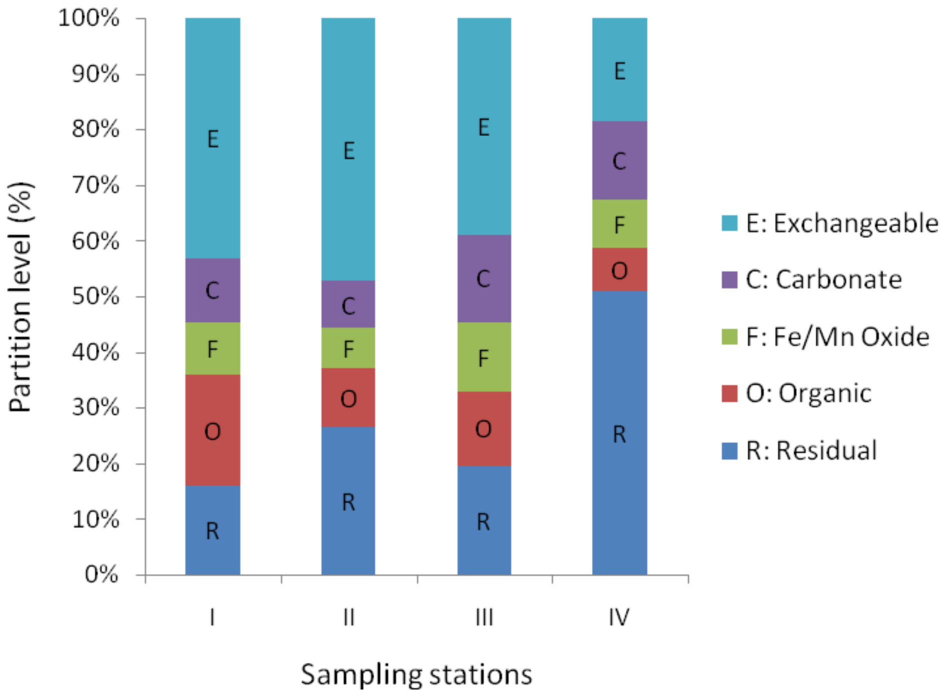

Figure 2. Partitioning of Lead into different geochemical fractions of soil collected under carrot obtained from four sampling stations along River Delimi and Lamingo Dam in Jos metropolis

At the study area (stations I, II and III) Pb was mostly bound to exchangeable fraction while at the control site it was mainly found in the residual. At station I, the exchangeable fraction, had the highest mean Pb content (43.1%) followed by organic (19.8%), residual (16.0%) and carbonate bound (11.5%). The least value of 9.6% was recorded in Fe/Mn oxide fraction. The distribution at station II was similar with the highest mean value in exchangeable (52.0%) and the least in Fe/Mn oxide (8.0%). The other three fractions carbonate, organic and residual had 9.3%, 11.6% and 19.1% mean metal recoveries respectively. At station III, the pattern of distribution was the same and the following values 38.9%, 15.7%, 12.5%, 13.3% and 19.6% were recorded for exchangeable, carbonate, Fe/Mn oxide, organic and residual fractions respectively. However, at station IV, the pattern of distribution was different and the metal was mostly bound to the residual fraction. The percentage recoveries in decreasing order were residual (50.9%), exchangeable (18.6%), carbonate (14.0), Fe/Mn oxide (8.7%) and organic (7.8%).

At the farmlands cultivated along River Delimi (study area), Pb in soil samples collected under carrot was predominantly associated with the non-residual fraction. At farmlands along Lamingo Dam (control site) however, the metal was predominantly bound to the residual non-mobile fraction. Since Pb was mostly bound to the exchangeable fraction at the study site, it may be concluded that there is a serious health risk associated with the consumption of carrot raised on farmlands along the Delimi River because the metal can easily be transferred to the crop. Little or no risk is however expected in the consumption of the crops cultivated on the bank of Lamingo Dam since a greater portion of the metal is associated with the residual fraction which is inert towards bioavailability and therefore not readily available for uptake by the crop. Furthermore, it may be concluded that the majority of Pb recovered in soil samples from control site are lithogenic and relatively chemically stable and so are not expected to be released over a reasonable period of time.

3.2.2. Partitioning of Cadmium in Soil Collected under Carrot

In both the study and control sites, Cd is mostly bound to the residual phase. Analyses of samples collected at station I revealed that residual and exchangeable were the dominant fractions in the partitioning of Cd with mean values of 44.1% and 21.1% respectively (Figure 3).

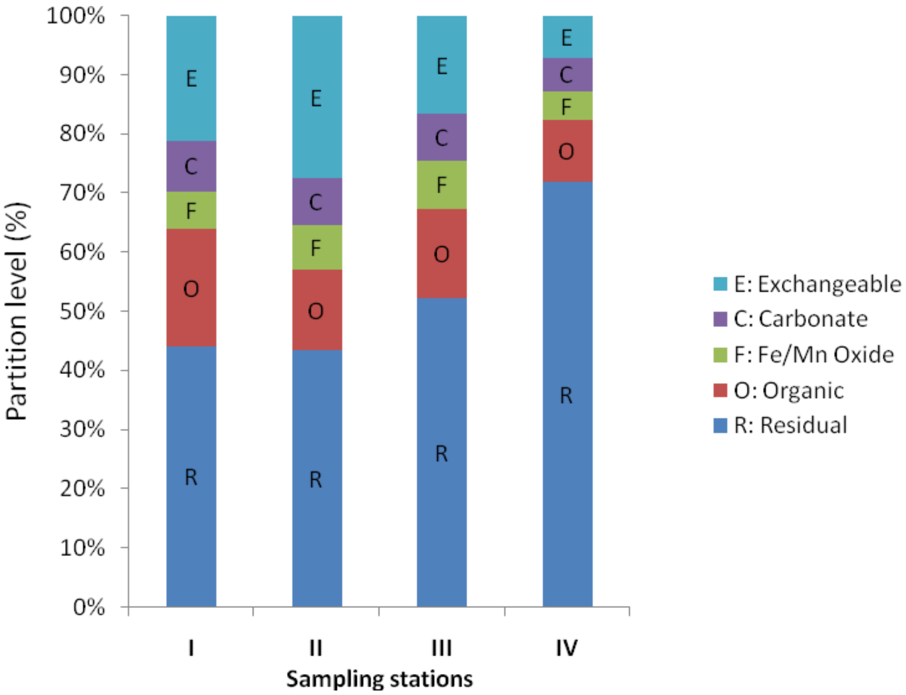

Figure 3. Partitioning of Cadmium into different geochemical fractions of soil collected under cabbage obtained from four sampling stations along River Delimi and Lamingo Dam in Jos metropolis

A mean value (19.8%) closely related to that of exchangeable was obtained in organic fraction. The other non-residual fractions - carbonate and Fe/Mn oxide had 8.6% and 6.4% respectively. At station II, 43% of Cd was found in the residual fraction. The next important fraction was the exchangeable phase with mean Cd value of 27%. The other values were distributed among carbonate (7.9 %,) Fe/Mn oxide (7.6%) and organic (13.5%). The residual fraction (with mean Cd content of 52.2%) was also the dominant phase in soil samples collected at station III. The mean values in the non- residuals fractions in decreasing order were carbonate (7.3%), Fe/Mn oxide (8.1%), organic (15.2) and exchangeable (16.6%). A remarkably high Cd value up to 71.9% was also recovered in the residual fraction at station IV. The remaining values were shared among other fractions as follows; exchangeable (7.2%), carbonate (5.5%), Fe/Mn oxide (5.0%) and organic (10.4%).

The non-residual fractions dominated the distribution of Cd in soil samples obtained from the study areas except those collected at station III where the metal is predominantly bounded to the residual phase. Among the non-residual fractions the exchangeable fraction is particularly the most important. Metals in exchangeable phase correspond to those metals that are only weakly bound and are generally considered the most mobile. The Cd content of soils collected under carrot could therefore be easily released from the soil particles into the soil water for absorption by the vegetable crop and therefore poses a serious health risk to the consumers. This is in agreement with the earlier assertion made by [21] and [22] who opined that Cd is readily desorbed from the solid phase in soil which results in its categorization as a generally mobile and bioavailable element. At station III and IV however, a relatively lower risk is expected from the consumption of the crop since greater portion of the metal was recovered from the residual non mobile fraction of the soil which do not allow the release of the metal into soil pore water.

3.3. Partitioning of Metals in Soil Collected under Cabbage

3.3.1. Partitioning of Lead in Soil Collected under Cabbage

At station I, exchangeable fraction with a mean Pb concentration of 51% was the dominant fraction. The next important fractions were residual and carbonate fractions having 15.5% and 12.5% metal recoveries respectively. The mean concentrations recovered in the other fraction as shown in Figure 4 were closely related as follows; Fe/Mn oxide (10.8%) and organic (10.3%).

At station II, 40.5% of the total Pb was bound to exchangeable fraction being the most dominant phase in the metal distribution. The next dominant fraction was the residual in which 23.0% of the metal was recovered. Relatively lower values were obtained in the other fractions as follows; carbonate (12.9%), Fe/Mn oxide (8.4%) and organic (15.2%). At station III (just like the case of station II) Pb was predominantly associated with the exchangeable fraction accounting for 39.4% of the total Pb concentration. The residual fraction had 28.0% while the other fractions - carbonate, Fe/Mn oxide and organic fractions recovered 13.0%, 8.4% and 11.2% respectively. At station IV the most important phase is the residual fraction accounting for as much as 64.4% of the total Pb content. The remaining non- residual fractions (exchangeable, carbonate, Fe/Mn oxide and organic) had lower mean concentrations of 10.5%, 6.8%, 8.1% and 10.2% respectively.

The partitioning of Pb to the various fractions of the metals collected under cabbage at the study site (stations I, II and III) has revealed a lot of variability in the distribution of the metal among the various soil geochemical fractions and higher values were generally recovered from the exchangeable fraction of the non-residual phases. The accumulation of this metal at the study site may therefore be considered human induced. Similar higher mean percentage values of Pb in the extractable fractions of up to 69.60% was earlier reported by [23] in agricultural soils obtained from Benue State of Nigeria. The higher Pb contents in non-residual fractions of the soil at the study site confirmed human interference with the soil quality in the study area and a relatively high risk of Pb toxicity among people that consume cabbage cultivated on the farmlands.

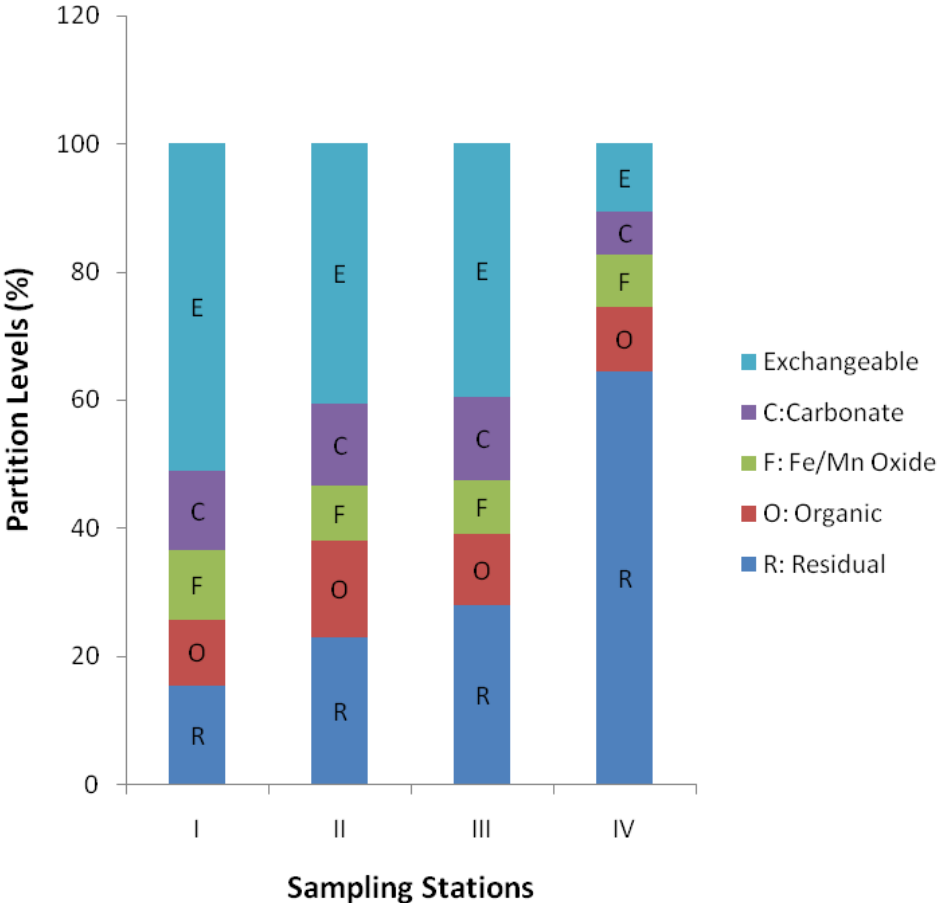

Figure 4. Partitioning of Lead into different geochemical fractions of soil collected under cabbage obtained from four sampling stations along River Delimi and Lamingo Dam in Jos metropolis

3.3.2. Partitioning of Cadmium in Soil Collected under Cabbage

In both the study and control sites, Cd was mostly bound to the residual phase of the soil collected under cabbage. Analyses of samples collected at station I revealed that residual and exchangeable were the dominant fractions in the partitioning of Cd with mean values of 44.1% and 21.1% respectively (Figure 5).

A mean value (19.8%) closely related to that of exchangeable was obtained in organic fraction. The other non-residual fractions - carbonate and Fe/Mn oxide had 8.6% and 6.4% respectively. At station II, 43% of Cd was found in the residual fraction. The next important fraction is the exchangeable phase with mean Cd value of 27%. The other values were distributed among carbonate (7.9 %,) Fe/Mn oxide (7.6%) and organic (13.5%). The residual fraction (with mean Cd content of 52.2%) was also the dominant phase in soil samples collected at station III. The mean values in the non- residuals fractions in decreasing order were carbonate (7.3%), Fe/Mn oxide (8.1%), organic (15.2) and exchangeable (16.6%). A remarkably high Cd value up to 71.9% was also recovered in the residual fraction at station IV. The remaining values were shared among other fractions as follows; exchangeable (7.2%), carbonate (5.5%), Fe/Mn oxide (5.0%) and organic (10.4%).

The distribution of Cd into the various geochemical fractions of soil obtained from the study area shows that a substantial amount of the metal was in soil residual matrix. However, the amount distributed among the other non-residual fractions were higher than what was recovered in the residual fraction alone. This corroborates the findings of [23] who reported that about 74% of the total Cd concentration was distributed among the first three extraction stages in soils obtained from farmland in Benue State, Nigeria. The high concentration of Cd associated with the non-residual phases means that it can easily be transferred into the food chain through uptake by crops growing on the soil thereby posing a serious health risk to the consumers. The high level of Cd in the non-residual fractions of soils is therefore a source of concern [23], since it constitutes potential health risk to humans [24]. At station IV however there was a dramatically higher percentage mean Cd concentration (71%) in the residual fraction. This was a clear indication that the Cd encountered in the soil samples obtained from the control site was non-anthropogenic but rather a product of weathering of the parent materials. The metal at this station is not likely to be available for uptake by vegetable crops being cultivated in the area and therefore poses little or no risk to consumers.

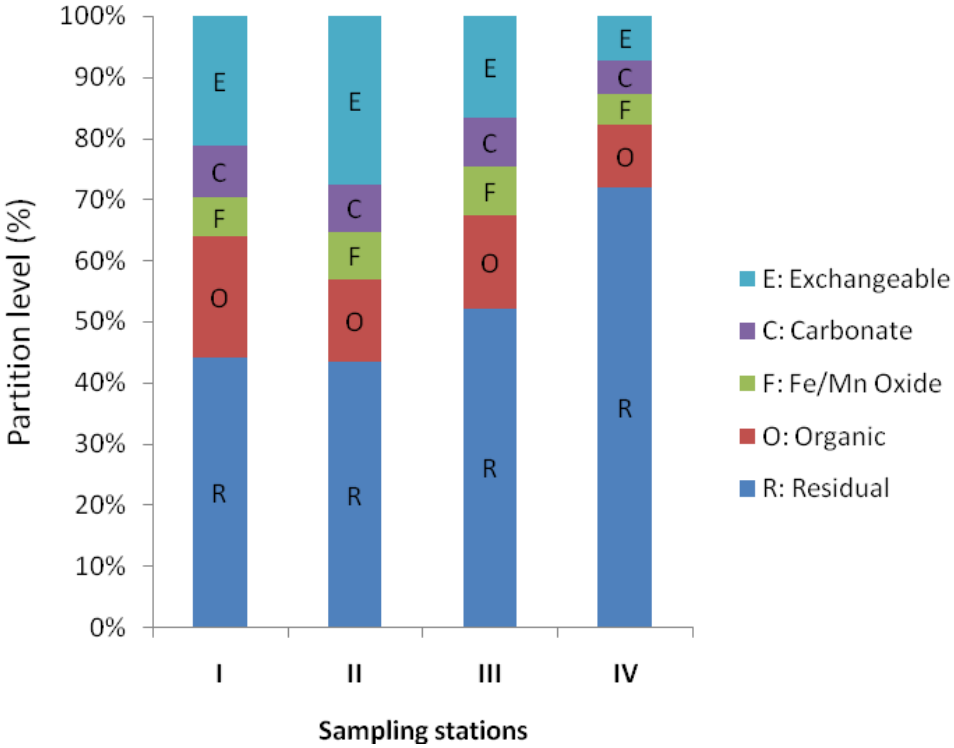

Figure 5. Partitioning of Cadmium into different geochemical fractions of soil collected under cabbage obtained from four sampling stations along River Delimi and Lamingo Dam in Jos metropolis

4. Conclusion

The result of this investigation revealed that soil samples collected from farmlands in the study area (along River Delimi) had more Pb and Cd than samples collected from farmlands at the control site (on the bank of Lamingo Dam). The partitioning of the metals in to different geochemical fractions revealed that at the study site, the metals were mostly found in the non- residual fraction especially the exchangeable fraction. Thus the metals could be released into soil pore water and readily available for uptake by the crops. At the control site however, the metals were found predominantly held in residual fraction and therefore not likely to be of serious environmental or human health concern. Little or no risk is therefore expected from the consumption of carrot and cabbage cultivated on farmlands along Lamingo Dam – the control site.

5. Recommendations

a. The farmers should be encouraged to cultivate crops with low metal transfer factors (e.g. pea nut and other industrial crops) instead of vegetable crops thus reducing the transfer of the metals into the food chain.
b. Soil organic amendment prior to sowing should be encouraged as it reduces the movement of the metals from soil to plants.
c. Liming of the soil to the pH of 7 is also recommended as it reduces the uptake of metals by the crops.

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