



## Mobility and Geochemical Partitioning of Heavy Metals (Pb, Cd, Cu, and Ni) in Sediments of an Urban River, Enugu Nigeria

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### To cite this article:

Okorie, E. and Ugwuene D.C. Environmental Mobility And Geochemical Partitioning Of Heavy Metals (Pb, Cd, Cu, And Ni) In Sediments Of An Urban River, Enugu Nigeria *International Journal of Research and Technopreneurial Innovations*, 2025; 2(1): 135-142

### Keywords:

Heavy metals,  
Sediments,  
Mobility,  
Pollution,  
Partitioning.

### ABSTRACT

The mobility of some selected heavy metals (Pb, Cd, Cu and Ni) in sediment of an urban river, Enugu Nigeria was carried out using Flame Atomic Absorption Spectrophotometric (FAAS) detection. The result of the speciation studies indicates that Pb was not detected in the Exchangeable fraction for Abakpa and Awkunanaw streams sediments. Cd was not detected in the Exchangeable, bound to carbonate and organic matter fractions in the three samples under investigation. Ni was not detected in the Exchangeable, bound to carbonate and organic matter from Abakpa and Ngwo stream. Pb indicated a high concentration at the carbonate bound fraction from Abakpa ( $6.329 \pm 0.001 \mu\text{g/g}$ ) and Awkunanaw ( $10.386 \pm 0.002 \mu\text{g/g}$ ) streams. Cu was detected in all the fractions from the three samples which serve as indication of an anthropogenic activity taken place at the stream sediments. The total Pb concentrations from the three samples are  $6.956 \mu\text{g/g}$  (Abakpa),  $12.419 \mu\text{g/g}$  (Awkunanaw) and  $1.746 \mu\text{g/g}$  (Ngwo). All the elements are below the EPA value for sediment quality guideline and therefore are fit for agricultural use. However, bioaccumulation of Pb and Cd may occur with time. The contamination factor analysis indicates that Pb, Cu and Ni have low contamination while Cd at Awkunanaw and at Ngwo are moderately contaminated to the environment. The geo-accumulation index showed that Pb, Cu and Ni occur as unpolluted to moderately polluted in conjunction with Cd at Abakpa and Awkunanaw sediments. Cd at Ngwo sediment exhibited a moderately polluted to strongly polluted accumulation index.

## 1. INTRODUCTION

The growth in urbanization worldwide as a result of technological advancement leads to socio-economic growth of any country. This advancement is a precursor to the continuous modification of the physical, chemical and biological composition of

our living environment. The consequences of these results in large numbers of people resident in and around urban centres to be exposed to an unnatural and unhealthy environment [1]. Sediments are the carriers of heavy metals in the environment and thus is an indication of the quality of the system. Like in

natural environments, sediments from urban area have a very high potential for the accumulation of different kinds of metals. Unlike natural sites, a considerable large amount of the metal found in urban sediments is not associated with the natural geologic parent material, but with the steady supply of those elements, both dissolved and in particulate form [1].

Thus, the proximity of a site close to an urban settlement center has the tendency to be polluted by both anthropogenic and naturally occurring metals. Obviously, natural sources of metals come from physical and chemical weathering of parent materials (rocks and minerals). Sources of these metals from the urban area and from the anthropogenic activities include industrialization, wastes and sewage effluents from urban centres, underground deposition of industrial wastes etc [2]. In sediments, metals occur in a number of several chemical forms, and generally exhibit different physical and chemical behaviour in terms of chemical interaction, mobility, biological availability and potential toxicity [3].

It is therefore important to identify and quantify the various forms in which a metal exist in sediment so as to acquire an in depth understanding of the potential and actual effects of elevated concentrations of metals in sediments and to evaluate processes of downstream transport, deposition and release under changing environmental conditions. The general behaviour of metals in an aquatic environment is influenced by the associations of metals with various geochemical phases in sediments [4]. Large amount of industrial metal (mainly Cr) contained waste was deposited into the environment due to industrialization about 30 years ago [5]. Metals are mostly distributed in the sediments components and are also associated with them in various geochemical forms which include ion exchange, adsorption, precipitation, and complexation. As a result, environmental changes such as acidification, appearance of strong current, dredging, redox potential or organic ligand

concentrations, oxidation state can lead to mobilization of metals from solid to liquid phase [6]. The metals accumulated in sediments can cause contamination of the surrounding and represent a potential risk for an aquatic environment [7].

Rivers are predominant pathway for metals mobility in the ecosystem [8-9], and these heavy metals significantly pollute many of our rivers [10]. The behaviour of metals in natural waters depends on the sediment composition and the water chemistry. In the course of their mobility, the heavy metals undergo various transformations in their chemical occurrence (speciation) due to dissolution, precipitation, sorption and complexation phenomena which affect their behaviour and bioavailability [11-13]. Hence, heavy metals are sensitive indicators for monitoring changes in the aquatic environment. However, to assess the environmental impact of contaminated sediments, information on total metal concentrations is not sufficient and particular interest is the fraction of the total heavy metal content that may take part in further biological processes [14-15].

The aim of this study is to determine the mobility and geochemical partitioning of heavy metals Lead (Pb), Copper (Cu), Cadmium (Cd) and Nickel (Ni) in sediments of an urban riverine sites in Enugu State, Nigeria.

## **2. MATERIALS AND METHOD**

### **2.1 *Materials and reagents***

Volumetric flask, 250 ml of Measuring Cylinder, 50 ml of Beaker, Whatman filter paper, Funnels, Conical Flask, pH meter, Heating mantle, Weighing balance, Spatula, Glass rod, Flame Atomic Absorption Spectrophotometer (FAAS) (Buck Scientific Model AVG 210 UK). Acetic acid, Hydroxylamine hydrochloride, Nitric acid, Hydrogen peroxide, Sulphuric acid and distilled water.

### **2.2 *Sample collection/preparation***

Sediment samples were collected from

Ngwo, Abakpa and Awkunanaw sites, Enugu State, for the determination of heavy metals (Pb, Cu, Ni and Cd). 1 gram of sediment sample was measured for each fraction and 20 ml of 0.1 mol<sup>-1</sup> of HOAc, 20 ml of 0.5 mol<sup>-1</sup> NH<sub>2</sub>OH.HCl, 5 ml of 8.8 mol<sup>-1</sup> H<sub>2</sub>O<sub>2</sub> was adjusted to pH 1.5 with HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub> for extraction of aqua regia. Each step was poured before agitation under a temperature of 22±5°C. The filtrate was filtered using filter paper and poured into a sample bottle for analysis using Flame Atomic Absorption Spectrophotometer.

### 2.3. Methods

A modified sequential extraction technique was used to extract content of chemical speciation of heavy metal in soil. The extraction was carried out in Teflon containers provided with screw stoppers as described by [16]. The modified Bureau Community of Reference (BCR) Sequential Extraction Procedure (1g dry weight mass sample) is described in Table 1.

**Table 1: Bureau Community of Reference (BCR) Sequential Extraction Protocol [16].**

Fraction	Components Extracted	Extraction Reagent	Extraction Method
Fraction 1, Acid solution	Exchangeable and bound to carbonates	20 ml 0.11 mol/L HOAc	Shaking at 22 ± 5 °C for 16h
Fraction 2, Reducible	Bound to Fe/Mn oxides	20 ml 0.5 mol/L NH <sub>2</sub> OH.HCl (adjusted to pH 1.5 with HNO <sub>3</sub> )	Shaking at 22±5°C for 16h
Fraction 3, Oxidizable	Bound to organic matter and sulphides	5 ml 8.8 mol/L H <sub>2</sub> O <sub>2</sub> (adjusted to pH 2.0 with HNO <sub>3</sub> )/ another 25 ml 1 mol/L NH <sub>4</sub> OAC (adjusted to pH 2.0 with HNO <sub>3</sub> )	Digesting at room temperature for 1hr with occasional manual shaking, then cover the tube and heat at 85 ± 5 °C for 1hr and then remove the cover and reduce the volume to a few milliliters/cover the tube and heat at 85 ± 2 °C for 1 hr and then remove the cover and reduce the volume to near dryness/shaking at 22 ± 5 °C for 16 hr
Fraction 4, Residual	Metals within lithogenic minerals	Aqua regia	

The residue of each step was rinsed with 20 ml distilled water and filtered through filter paper into pre-cleaned 100 ml volumetric flask. The

supernatant liquid of each fractions after filtration were stored for metal determination using FAAS.

## 3. RESULTS AND DISCUSSION

### 3.1 RESULT

**Table 2: Results of speciation of soil sample at Abakpa Stream Enugu State (µg/g)**

Fraction	Pb	Cd	Cu	Ni
Exchangeable	ND	ND	2.859±0.001	ND
Bound to Carbonate	6.329± 0.001	ND	0.445± 0.000	ND
Organic Matter	ND	ND	1.588± 0.000	ND

Residual	0.627± 0.001	0.081± 0.000	0.146± 0.000	0.592± 0.002
Total	6.956	0.081	5.038	0.592
E. P. A. Value	16	0.38	36	30

**Table 3: Results of speciation of soil sample at Awkunanaw Stream Enugu State ( $\mu\text{g/g}$ )**

Fraction	Pb	Cd	Cu	Ni
Exchangeable	ND	ND	0.119± 0.001	ND
Bound to Carbonate	10.386± 0.002	ND	0.190± 0.0	0.121± 0.000
Organic Matter	2.033± 0.001	ND	0.081± 0.001	ND
Residual	ND	0.118± 0.000	0.173± 0.0	0.153± 0.001
Total	12.419	0.118	0.563	0.274
E. P. A. Value	16	0.38	36	30

**Table 4: Results of speciation of soil sample at Ngwo Stream Enugu state ( $\mu\text{g/g}$ )**

Fraction	Pb	Cd	Cu	Ni
Exchangeable	0.123± 0.0	ND	0.213± 0.001	ND
Bound to Carbonate	0.807± 0.001	ND	0.321± 0.0	ND
Organic Matter	0.070± 0.0	ND	1.722± 0.001	ND
Residual	0.746± 0.0	0.104± 0.002	0.151± 0.0	0.583± 0.002
Total	1.746	0.104	2.407	0.583
E. P. A. Value	16	0.38	36	30

The results of the environmental mobility and geochemical partitioning of Pb, Cd, Cu and Ni in sediments of an urban river, Enugu Nigeria are presented in Tables 2 to 4. Table 2 shows the result from soil sample at Abakpa stream in Enugu State while Tables 3 and 4 are the results of the soil samples at Awkunanaw Stream and Ngwo Stream respectively. From Table 2, Pb was not detected at Exchangeable and organic matter phases but was observed at bound to carbonate and residual fraction with concentrations of  $6.329\pm 0.001 \mu\text{g/g}$  and  $0.627\pm 0.001$ . The total Pb concentration is  $6.956 \mu\text{g/g}$ . Cd at Abakpa site was not detected in fractions 1 to 4, but a low concentration of Cd ( $0.081\pm 0.0$ ) was detected at the residual phase. The observed concentration of Cu in Table 2 ranged from  $0.146$ - $2.859 \mu\text{g/g}$  with a total Cu concentration of  $5.038 \mu\text{g/g}$ . Ni was only detected at the residual phase with a concentration of  $0.592\pm 0.002 \mu\text{g/g}$ .

The soil samples at Awkunanaw Stream as

indicated in Table 3 showed that Pb was only detected at the bound to carbonate fraction and organic matter fractions with concentrations of  $10.386\pm 0.002 \mu\text{g/g}$  and  $2.033\pm 0.001 \mu\text{g/g}$  respectively. The total Pb concentration is  $12.419 \mu\text{g/g}$ . Cd was only detected at the residual fraction with a concentration of  $0.118 \mu\text{g/g}$ . Cu concentration ranged from  $0.081$ - $0.19 \mu\text{g/g}$ . The total Cu concentration is  $0.563 \mu\text{g/g}$ . Ni was not detected in exchangeable and organic matter phases while a concentration of  $0.121\pm 0.0$  and  $0.153\pm 0.001 \mu\text{g/g}$  was observed in the bound to carbonate and residual fractions respectively.

For the soil sample at Ngwo Stream (Table 4), the Pb observed ranged from  $0.07$ - $0.807 \mu\text{g/g}$  with a total Pb concentration of  $1.746 \mu\text{g/g}$ . Cd and Ni were only detected in the residual phase with a concentration of  $0.104\pm 0.002 \mu\text{g/g}$  and  $0.583\pm 0.002 \mu\text{g/g}$  respectively.

### 3.2 DISCUSSION

From the results, the Pb concentrations appear more in the bound to carbonate fraction than the other fractions. Carbonate is often an important component of marine sediments and has been found to be an important indicator of provenance and dispersal of terrigenous materials [17]. However, all the Pb concentrations and the other metals in the three samples are all below the EPA permissible limit [18] (Table 8). Cd and Ni occurrence in all the samples may be attributed to natural occurrence and weathering. There is no anthropogenic contribution to the occurrence of Cd and Ni in these samples. Cu ability to form complexes may have been responsible for its presence in the organic fractions of the three samples.

The association between trace metals and the residual fraction of uncontaminated soil is so strong that metals association with non-residual fractions has been used as an indicator of anthropogenic enrichment [19]. The high value of oxidizable-organic fraction of Cu identified in this research is an indication that the organically-bound materials such as humic and fulvic acids can easily

be available for biological uptake [20].

### 3.3 Geo-Accumulation Index

An approach to estimate the enrichment of metal concentrations above background or baseline concentrations is to calculate the geo-accumulation index (I<sub>geo</sub>) as proposed by Muller [21]. Sediment I<sub>geo</sub> is the quantitative measure of heavy metal in aquatic sediments. This index is basically a single metal approach to quantify metal pollution in sediments when the concentration of toxic heavy metal is 1.5 or more times greater than their lithogenic background values [22].

$$I_{geo} = \log_2 [C_n/1.5B_n]$$

Where C<sub>n</sub> is the measured concentration of the element n and B<sub>n</sub>, the geochemical background value element n in average crust. The factor 1.5 is used to compensate possible variations in the sediment. Geochemical background values of Fe and Mn were taken from Wedepohl [23]. The classification for I<sub>geo</sub> is shown on Table 5.

**Table 5: Classification For Geo-Accumulation Index [21].**

I <sub>geo</sub> value	Class	Sediment quality
<0	0	Unpolluted
0-1	1	From unpolluted to moderately polluted
1-2	2	Moderately polluted
2-3	3	From moderately polluted to strongly polluted
3-4	4	Strongly polluted
4-5	5	From strongly polluted to extremely polluted

**Table 6: Results of the Geo-accumulation Index**

Fraction	Pb	Cd	Cu	Ni
Abakpa	0.09	0.16	0.04	0.0
Awkunanaw	0.17	0.24	0.01	0.0
Ngwo	0.02	2.22	0.02	0.0

From the result of the geo-accumulation index (Table 6), Cd found in Ngwo stream is higher than 2 and therefore moderately polluted to strongly polluted. It is essential to note that geo-accumulation of Cd may occur in this case. Other

heavy metals (Pb, Cu and Ni) are all polluted to moderately polluted.

### 3.4 Contamination Factor

A contamination was described as the

contaminating of a given toxic substance in a basin by Hakanson [24]. Contamination factor (Cf) is the ratio of the concentration of the element in the sample to pre-industrial reference value of the element.

$$C_f^i = \frac{C_e}{C_{pi}}$$

C<sub>e</sub> is the concentration of element, C<sub>pi</sub> is the concentration of the metal in pre-industrial reference (Table 10).

According to Hakanson [24], Cf has been classified into four groups as shown on Table 7.

**Table 7: Classification For Contamination Factor**

Factor	Remark
Cf<1	Low contamination
1 ≤ Cf < 3	Moderate contamination

**TABLE 9: EPA SEDIMENT QUALITY GUIDELINES (S. Q. G.)**

Metal	Non-Polluted	Moderately Polluted	Heavily Polluted	Present study		
				Abakpa	Awkunanaw	Ngwo
Pb	< 40	40 – 60	> 60	0-6.329	0-10.386	0.070-0.807
Cd	×	×	> 6	0-0.081	0-0.118	0-0.104
Cu	< 25	25 – 50	> 50	0.146-2.859	0.081-0.190	0.151-1.722
Ni	< 20	20 – 50	> 50	0-0.592	0-0.153	0-0.583

From the EPA sediment quality guideline [18] as presented on Table 9, all the heavy metals studied are < 40 and therefore non-polluted.

**TABLE 10: Geochemical background values**

Pb	Cd	Cu	Ni
14.8	0.1	25	56

#### 4 CONCLUSION

All the soil samples studied do not have any of the heavy metals in objectionable quantity and therefore can be used for agricultural purposes. The Cu and Pb may be attributed to refuse dumps along the banks of the river. The total concentration of Pb in Abakpa stream (6.956 µg/g) and Awkunanaw

3 ≤ Cf < 6	Considerable contamination
Cf > 6	Very high contamination

**Table 8: Results Of The Contamination Factors**

Fractions	Pb	Cd	Cu	Ni
Abakpa	0.47	0.81	0.20	0.01
Awkunanaw	0.84	1.18	0.02	0.0
Ngwo	0.12	1.04	0.01	0.01

The contamination factor analysis of the study areas (Table 8) showed that Cd found in Awkunanaw and Ngwo stream are moderately contaminated. The other heavy metals (Pb, Cu, and Ni) are all of low contamination.

stream (12.419 µg/g) may however bio-accumulate with time. Thus, the streams are not good sources of drinking water for the inhabitants of the area.

#### COMPETING INTERESTS

The authors have declared that no competing interests exist.

#### AUTHORS CONTRIBUTIONS

The authors received no specific funding for this work.

#### ACKNOWLEDGEMENT

The authors wish to acknowledge the the department of science Laboratory technology, The Federal Polytechnic Idah Kogi State Nigeria for

giving us access to the AAS machine for the heavy metals analysis.

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