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# **Spatial Distribution and Speciation of Heavy Metals in Sediment of River Ilaje, Nigeria**

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## *Authors' contributions*

*This work was carried out in collaboration between all authors. Author AS designed the study and wrote the protocol. Author AS preformed the statistical analysis, managed the literature search and wrote the first draft of the manuscript with assistance from authors KR and EO. All authors read and approved the final manuscript.*

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# **ABSTRACT**

Speciation of heavy metals (Zn, Fe, Cr, Cd, Cu and Pb) was carried out on the top and bottom sediment samples of two sampling sites in river Ilaje, Nigeria during the dry and rainy seasons. The samples were subjected to sequential extraction method and the extracts were analyzed for heavy metals using atomic absorption spectrophotometer. The result shows that Zn and Fe are abundant in reducible fraction, oxidizable fraction and highest in residual fractions in both seasons. The concentrations of Cr and Cd varied greatly with the seasons, while the highest concentration of Pb occurs in residual fraction for both seasons. The concentration of Cu occurs abundantly in oxidizable fraction and residual fraction, Cu and Pb shows a low concentration in exchangeable fraction and carbonate fraction. This study reveals that the mobility and bioavailability of the heavy metals in the sediment for the two seasons are in this order: Fe > Cr > Zn > Pb > Cu > Cd. It was observed that the origin of metals in the environment is likely to be lithogenic.

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#### **1. INTRODUCTION**

Heavy metals enter into the aquatic environment either through the naturally occurring<br>biogeochemical processes or through biogeochemical processes or anthropogenic sources. These influence the nature of the sediments in the coastal water areas such as the Ilaje coastal area. The Ilaje coastal area of Ondo State, Nigeria lies on Latitude.  $5^{\circ}$  50'N –  $6^{\circ}$  09'N and Longitude.  $4^{\circ}$ 45'E – 5° 05'E, river Ilaje is a deltaic region which takes its source from river Oluwa. Ilaje communities depend on the river Ilaje for their economic and domestic activities.

Heavy metal contamination is a major problem because of their toxicity and threat to living organisms and the environment [1]. Heavy metals are being accumulated in urban rivers, estuaries and adjacent coastal zones as a result of urbanization, industrialization, water runoff, landfill leachate e.t.c [2,3]. Sediments may therefore act as either sources or sinks for metals and this is strongly dependent on their chemical forms [4]. Determination of the total metal concentration may provide information concerning possible pollution levels but generally there are insufficient details for estimating their biological effects, risk of bioavailability, toxicity, capacity for remobilization of heavy metals in the environment. It is the chemical form of metals in the soil that determine its toxicity, mobilization capacity and behavior in the environment [5,6,7,8]. The bioavailability, toxicity and distribution of trace metals are best described when the speciation (chemical fractionation) in the soil/sediment is known [9]. Heavy metal pollution produced in industrial wastewater is a major issue throughout the world. In particular, Cu (II) ions, whose sources are metal plating, mining, tanneries, painting, car radiator manufacturing, as well as agricultural sources where fertilizers and fungicidal spray are used intensively. The effect on human health and aquatic life are regarded as harmful, and of course are not biodegradable [10]. Heavy metals are stable and persistence environmental contaminant of marine sediments. The vast increase in population growth, urbanization and industrialization leads to increase in marine discharges, which results in total loads of pollutants being delivered to the sea. Heavy metal pollution in aquatic environment and subsequent uptake in food chain by aquatic organisms and humans put public health at risks

[11]. The aim of water preservation within each country seeks to be achieved through the increased concern about the water, which often means controlling of the water quality. The most important anthropogenic sources of metals in aquatic ecosystem are certainly wastewaters, that are being discharged untreated or with different levels of purity, so they can cause many changes in the water bodies. Heavy metals pass through biogeochemical process with different retention time in different parts of atmosphere. They do not decompose and have the ability of bioaccumulation, because they are being retained in plants, animals and nature in general. Chemical analysis and speciation of heavy metals such as lead, cadmium, zinc, copper, etc. is of great importance [12,13,14]. The main anthropogenic sources of cadmium appear to be mining, metals smelting, industries involved in the manufacture of alloys and batteries, transport of cadmium to the sediments occurs mainly through sorption to organic matter and through co-precipitation with iron, aluminum, and manganese oxides. The main sources of chromium to the environment are emissions from the ferrochromium and metal plating industries, with coal and oil burning, refractory production, cement manufacturing, and the production of chromium steels. Under anaerobic conditions, Cr (VI) may be reduced to Cr (III). In contrast to Cr (VI), Cr (III) readily sorbs onto organic particulates and co-precipitates with hydrous iron and manganese oxides. Chromium (VI) is more readily accumulated than Cr (III) and is considered to be the more toxic form. In sediments, lead is primarily found in association with iron and manganese hydroxides. However, it may also form associations with clays and organic matter. Lead tends to remain tightly bound to sediments under oxidizing conditions. However, it may be released into the water column under reducing conditions. Aquatic organisms exhibit a wide range of sensitivities to lead. Lead may be accumulated to relatively high levels by aquatic biota. Bioconcentration factors (BCFs) in algae may be as high as 20,000 however, BCFs for fish and invertebrates tend to be much lower 500 to 1700. The principal sources of zinc to aquatic systems include municipal wastewater effluents, zinc mining, smelting, and refining activities, wood combustion, waste incineration, iron and steel production, and other atmospheric emissions, zinc may be deposited in sediments by sorption to hydrous iron and manganese oxides, clay minerals, and organic matter. However, sorption to organic matter appears to be the most important environmental fate process in fine grained sediments. Under reducing conditions, organically-bound zinc generally forms insoluble sulfides [15,16,17]. Due to the toxicity and bioaccumulation of heavy metals in sediment, it is essential to carry out a chemical speciation study of heavy metals in the sediment of river Ilaje.

## **2. MATERIALS AND METHODS**

### **2.1 Sampling and Pre-treatment of Sample**

A total of 24 sediment samples were collected from two sampling sites (Abereke and Ogogoro) in river Ilaje, Nigeria during the dry (December 2014) and rainy (May 2015) seasons. Sampling points were geo-located using Geographical Position System (GPS) to ensure consistency, top (0-15 cm) and bottom (15-30 cm) sediment samples were collected using hand hugger, and stored in a polythene bag prior to analysis. The samples were air dried in the laboratory for two weeks, and later pulverized using laboratory mortar and pestle and sieved with 2 mm mesh size sieve. Fig. 2 shows a descriptive diagram of the study area showing the two sampling sites.

#### **2.2 Multi-step Sequential Extraction**

The sediment samples were subjected to sequential extractions method and heavy metals were associated in five fractions [18]. As stated below:

#### **2.2.1 Fraction 1 (exchangeable fraction)**

1 g of sediment samples were extracted at room temperature for 1 hour with 8ml of  $MgCl<sub>2</sub>$  solution  $(1M \text{ MgCl}_2)$  at pH 7. Sediment and extraction solution were thoroughly agitated throughout the extraction using a centrifuge at 4,000 rpm. The extracts were decanted from the residue and analyzed for heavy metals using Atomic Absorption Spectrophotometer.

## **2.2.2 Fraction 2 (bound to carbonates)**

The residue of Fraction 1 was extracted with 8 ml of 1 M sodium acetate/acetic acid buffer at pH 5, the extraction process was carried out for 5 hours at room temperature with thorough agitation using a centrifuge at 4,000 rpm. The extracts were decanted from the residue and analyzed for heavy metals using Atomic Absorption Spectrophotometer.

#### **2.2.3 Fraction 3 (bound to iron and manganese oxides)**

The residue from fraction 2 was extracted under mild reducing conditions using 20ml of 0.04M NH2OH.HCl in 25% (v/v) acetic acid with agitation at 96°C in a water bath for six hours. The extracts were decanted from the residue and analyzed for heavy metals using Atomic Absorption Spectrophotometer.

#### **2.2.4 Fraction 4 (bound to organic matter and sulphide)**

The residue from fraction 3 was extracted under a mild oxidizing condition as follows: 3 ml of 0.02M HNO<sub>3</sub> and 5 ml of 30% (v/v)  $H_2O_2$ , which has been adjusted to pH 2 with  $HNO<sub>3</sub>$ , were added to the residue from fraction 3. The mixture was heated to 85°C in a water bath for 2 hours with occasional agitation and allowed to cool down. Another 3ml of 30%  $H<sub>2</sub>O<sub>2</sub>$ , adjusted to pH 2 with  $HNO<sub>3</sub>$ , was then added. The mixture was heated again at 85°C for 3 hours with occasional agitation and allowed to cool down. Then 5ml of 3.2M ammonium acetate in 20% ( $v/v$ ) HNO<sub>3</sub> was added, followed by dilution to a final volume of 20 ml with de-ionized water and agitated continuously for 30 minutes. The extracts were decanted from the residue and analyzed for heavy metals using Atomic Absorption Spectrophotometer.

#### **2.2.5 Fraction 5 (residual or inert fraction)**

The residue from fraction 4 was oven dried at 105°C. Digestion was carried out with a mixture of 5ml conc. Nitric acid (HNO $_3$ , 70% w/w), 10 ml of hydrofluoric acid (HF, 40% w/w) and 10 ml of perchloric acid (HClO<sub>4</sub>, 60%w/w) in Teflon beakers. The digestate was decanted and analyzed for heavy metals using Atomic Absorption Spectrophotometer.

Fig. 1 shows a schematic diagram for the sequential extraction procedure of heavy metals in sediment from the two sampling sites. After each extraction step, the sample was subjected to centrifugation at 4,000 rmp and the supernatants were separated from the residue in order to ensure selective dissolution and prevent possible interface mixing between the extractants. Analysis of each extract was carried out using atomic absorption spectrophotometer (AAS). All chemicals used for the sequential extraction of heavy metals in the sediment of river Ilaje were of analytical grade. Blanks were also used for correction of background and other sources of error.



**Fig. 1. Showing a schematic diagram for the sequential extraction procedure for heavy metals in sediment**



**Fig. 2. Map of the study area (Ilaje community, Ondo State. Nigeria)**

#### **2.3 Data Analysis**

Data generated on concentrations of each metal were analyzed for spatial variation using one way analysis of variance (ANOVA) with SPSS package version 20.

## **3. RESULTS AND DISCUSSION**

Tables 1-4 and Figs. 3-6 shows the results of the sequential extraction of top and bottom sediment samples for both dry and rainy seasons in the 2 sampling sites (Abereke and Ogogoro) in the coastal area of river Ilaje in Ilaje local government area of Ondo State, Nigeria.

Analysis of the data obtained in the two sampling site shows that in the dry season, all the heavy metals in the top sediment were mostly abundant in the residual fraction in the following order Fe  $(9.80-10.75$  ppm) > Cr  $(1.91-1.98$  ppm) > Zn  $(1.60-1.83$  ppm) > Pb  $(0.40-1.19$  ppm) > Cu  $(0.24 - 0.35$  ppm $) > Cd$  (0.13-0.14 ppm), while for the bottom sediment it follows the same order Fe  $(9.85-10.60$  ppm) > Cr  $(1.67-1.97$  ppm) > Zn  $(1.46-1.90$  ppm $)$  > Pb  $(0.33-0.34$  ppm $)$  > Cu

 $(0.22 - 0.29$  ppm $)$  > Cd  $(0.06 - 0.10$  ppm $)$ . However, for both top and bottom sediment samples, Fe was also abundant in the bound to organic matter and sulphide fraction (top {6.60-9.00 ppm}, bottom {9.20-9.35 ppm}) and bound to iron and manganese oxide fraction (top {8.45-9.80 ppm}, bottom {5.65-8.50 ppm}), while Pb occurs at a very low concentration in the bound to organic matter and sulphide fraction (top {0.40- 0.16 ppm}, bottom {0.14-0.17 ppm}) as well as the bound to iron and manganese oxide fraction (top {0.11-0.19 ppm}, bottom {0.08-0.13 ppm}. All the heavy metals in the top sediments occurs at a low concentration in the Exchangeable fraction in the following order Fe  $(0.75-0.95$  ppm)  $> Cr$  $(0.59-1.04$  ppm)  $>Zn$  (0.19 ppm)  $> Cu$  (0.06-0.11  $ppm$ ) > Pb (0.03-0.04) = Cd (0.03-0.04), while for the bottom sediment was as follows Fe (0.55-  $(0.70) >$  Cr  $(0.40-0.55) >$  Zn  $(0.17-0.21) >$  Cu  $(0.08)$  >Pb  $(0.05)$  > Cd  $(0.03)$ . Also the heavy metals in the top sediment associated with the bound to carbonate fraction occurs at a low concentration as follows Cr  $(0.62-0.77$  ppm) > Fe  $(0.35-0.40$  ppm $) > Zn$   $(0.23-0.30$  ppm $) > Pb$  $(0.07 - 0.20 \text{ ppm})$  > Cu  $(0.08 - 0.10 \text{ ppm})$  > Cd (0.03 ppm).



**Fig. 3. A graph of concentration metals in ppm against metals in Abereke top sediment samples**





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**Fig. 4. A graph of concentration metals in ppm against metals in Abereke bottom sediment samples**



<b>Fraction/</b>	Zn (ppm)		Fe (ppm)		$Cr$ (ppm)		Cd (ppm)		Cu (ppm)		Pb (ppm)	
heavy metals	Drv	Rain	Dry	Rain	Drv	Rain	Drv	Rain	Drv	Rain	Drv	Rain
F,	0.17	0.33	0.70	0.50	0.55	3.70	0.03	0.15	0.08	0.12	0.05	0.30
F <sub>2</sub>	0.34	1.01	0.55	4.41	1.00	4.35	0.04	0.11	0.03	0.13	0.06	0.45
$F_3$	2.04	1.52	5.65	7.17	0.64	2.03	0.04	0.31	0.07	0.12	0.13	0.40
F <sub>4</sub>	1.06	0.83	9.35	6.55	1.42	5.45	0.05	0.30	0.23	0.23	0.14	0.55
$F_5$	1.46	2.82	9.85	7.41	1.97	12.51	0.06	0.12	0.29	0.62	0.34	0.95

**Table 3. Concentration of heavy metals in sediment samples at Ogogoro site (F= fractions, Top= 0-15 cm)**

<b>Fraction/</b>	$Zn$ (ppm)		Fe (ppm)		$Cr$ (ppm)		Cd (ppm)		$Cu$ (ppm)		Pb (ppm)	
heavy metals	<b>Drv</b>	Rain	Drv	Rain	Drv	Rain	Drv	Rain	Drv	Rain	Drv	Rain
F,	0.19	0.55	0.75	0.18	0.59	2.57	0.03	0.09	0.06	0.19	0.04	0.15
F <sub>2</sub>	0.30	0.71	0.35	1.54	0.77	4.52	0.03	0.17	0.08	0.16	0.20	0.40
$F_3$	0.67	1.48	9.80	7.24	0.85	8.76	0.05	0.11	0.14	0.13	0.11	0.35
F <sub>4</sub>	1.30	0.81	9.00	6.01	1.66	2.22	0.05	0.28	0.47	0.22	0.16	0.40
$F_5$	1.83	3.09	10.75	7.35	1.91	8.03	0.13	0.26	0.24	0.38	1.19	3.55

**Table 4. Concentration of heavy metals in sediment samples at Ogogoro site (F= fractions, Bottom= 15-30 cm)**



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**Fig. 5. A graph of concentration metals in ppm against metals in Ogogoro Top sediment samples**



**Fig. 6. A graph of concentration of metals in ppm against metals in Ogogoro bottom sediment samples**

In the rainy season, most of the heavy metals in the top sediments have their highest concentration in the residual fraction as follows Cr (8.03-12.40 ppm) > Fe (7.35-7.37 ppm) > Zn  $(3.01-3.09$  ppm $)$  > Pb  $(0.75-3.55$  ppm $)$  > Cu  $(0.38 - 0.71$  ppm $) > Cd$  (0.22-0.26 ppm), the heavy metals in the bottom sediments follows a similar order Cr (10.21-12.51 ppm) ˃ Fe (7.41-7.50 ppm) ˃ Zn (2.82-3.20 ppm) ˃ Pb (0.70-0.95 ppm)  $>$  Cu (0.61-0.62 ppm)  $>$  Cd (0.12-0.27 ppm). However, Fe (top {6.01 ppm}, bottom {6.10-6.55 ppm}) ˃ Cr (top {2.22-3.61 ppm}, bottom{4.83- 5.45 ppm}) ˃ Zn (top{0.81-0.93 ppm}, bottom{0.83-0.95 ppm}) occurs abundantly in the bound to organic matter and sulphide fraction, and also in the bound to iron and manganese oxide fraction as follows Cr (top {7.16-8.76 ppm}, bottom {2.03-8.30 ppm}) ˃ Fe (top {7.01-7.24 ppm}, bottom {7.17-7.20 ppm}) ˃ Zn (top {1.33- 1.48 ppm}, bottom {1.52-1.64 ppm}). While Pb (top {0.40 ppm}, bottom {0.40-0.55 ppm}, Cd (top {0.25-0.28 ppm}, bottom {0.28-0.30 ppm) and Cu (top{0.22-0.24 ppm}, bottom {0.23-0.26 ppm}) occurs at a low concentration in the bound to organic matter and sulphide fraction, they also occurs at a low concentration in the bound to iron and manganese fraction as follows Pb (top {0.35 ppm}, bottom {0.35-0.40 ppm}), Cd (top {0.11- 0.26 ppm}, bottom {0.17-0.31 ppm}) and Cu (top {0.11-0.13 ppm}, bottom 0.12-0.13 ppm}). All the heavy metals occurs at a very low concentration in the exchangeable fraction Zn (top{0.31-0.55 ppm}, bottom {0.33-0.57ppm), Fe (top{0.18-0.45 ppm 0.20-0.50 ppm}) Cr (top {0.77-2.57 ppm}, bottom {1.30-3.70 ppm}), Pb (top {0.15-0.20 ppm}, bottom {0.30 ppm}) Cd (top {0.09-0.18 ppm}, bottom {0.15-0.16 ppm}) and Cu (top {0.13-0.19 ppm}, bottom {0.12-0.13 ppm}). They also occurs at a low concentration in the bound to carbonate fraction as follows Fe (top {1.54- 3.99 ppm}, bottom {1.99-4.41 ppm}), Cr (top {2.82-4.52 ppm}, bottom {4.35-7.41 ppm}), Zn (top {0.71-0.90 ppm}, bottom {0.88-1.01 ppm}), Pb (top {0.40-0.45 ppm, bottom {0.35-0.45 ppm}), Cd (top {0.14-0.17 ppm}, bottom {0.10- 0.11 ppm}) and Cu (top {0.14-0.16 ppm}, bottom {0.13-0.36 ppm}). This indicates that, there is a visible variation in the concentration of heavy metals in each fraction in relation with the dry and rainy seasons.

## **4. CONCLUSION**

The heavy metals (Zn, Fe, Cr, Cd, Cu and Pb) contents of river Ilaje sediment have been fractionated in order to determine their environmental fate; the following conclusion can be summarized:

Regarding the group of heavy metals and their potential toxicity, there is a wide range of spatial variation of heavy metals contents occurring in the top and bottom sediment samples, this indicate that there is a wide variation in the concentration of heavy metals in the top and bottom sediment deposit and this has an influence on the toxicity of heavy metals from the two sampling sites during the dry and rainy seasons;

The increase in the concentration of heavy metals within some fractions during the rainy season may have resulted from leaching which could be attributed to possible anthropogenic activities;

Most of the heavy metals were mainly associated with the bound to iron and manganese, oxidizable and residual fraction, the heavy metals bound to iron and manganese fraction are easily released into the environment under a reducing chemical condition which determines the rate of mobility of the heavy metals bound to iron and manganese fraction, while the heavy

metals bound to oxidizable fraction are easily release into the environment under an oxidation condition which determines the rate of mobility of the heavy metals bound to oxidizable fraction, but the heavy metals associated with the residual fractions are not easily release into the environment because the metals are firmly bounded within the crystal structure of the minerals comprising the sediment, hence they have a low mobility. This allows us to predict their mobility and bioavailability of heavy metals in the sediment samples because the higher the degree of mobility the higher the degree of bioavailability. The residual fraction has the highest concentration, therefore the heavy metals in the sediment are firmly bounded within their crystal structure and are not easily released into the environment hence they have a low mobility and a low bioavailability. While the exchangeable and bound to carbonate fraction has the lowest concentration, therefore the concentration of heavy metals released into the environment is very low because the exchangeable and bound to carbonate fraction which naturally has a high degree of mobility and bioavailability is occurring at a low concentration in this study. This is an indication that the heavy metals in the sediment are of a low mobility and a low bioavailability. This study reveals that the mobility and bioavailability of heavy metals in the top and bottom sediment deposit of river Ilaje for the dry and rainy seasons are in this order: Fe > Cr > Zn > Pb > Cu > Cd. Also the origin of heavy metals in the environment of the study areas is likely to be lithogenic.

The present study of the mobility and bioavailability of heavy metals in sediment of river Ilaje by a sequential extraction procedure has been able to provide vital information in addressing serious environmental problems of bioaccumulation and biomagnifications of heavy metals which could possible occur in aquatic organisms of Abereke and Ogogoro fishing communities as a result of anthropogenic activities. It is recommended that further work on heavy metals analysis should be carried out on both flora and fauna of the river.

## **COMPETING INTERESTS**

Authors have declared that no competing interests exist.

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