

## TRACE METALS IN WATER, SEDIMENT AND BIOTA FROM IKPUKULU-AMA CREEK PORT HARCOURT, RIVERS STATE, NIGERIA.

Vincent-Akpu Ijeoma Favour and Okoseimiema Ibifubara Joshua

Department of Animal and Environmental Biology,  
University of Port Harcourt, Nigeria.

P.O. Box 150, Uniport, Choba.

+ +234(0)8163807135, +234(0)7032519186

**Ijeoma.vincent-akpu@uniport.edu.ng**

### **ABSTRACT**

The concentrations of trace metals; Lead (Pb), Iron (Fe), Copper (Cu), Zinc (Zn) and Cadmium (Cd) were determined in water, sediment and biota (Fish and plankton) from Ikpukulu-ama creek in Port Harcourt, Nigeria. The metals were analyzed using Atomic Absorption Spectrophotometer (AAS 500). Concentrations of metals in water were in the order of Cd<Cu<Pb<Zn<Fe, in sediments Cd<Cu<Pb<Zn<Fe, in fish Cd<Cu<Pb<Zn<Fe while in plankton the order of concentration were Cd=Pb<Cu<Zn<Fe. The mean concentrations of metals in surface water (mg/L) were 5.2, 0.83, 0.03, 0.46, and 0.01 for Fe, Zn, Cu, Pb and Cd respectively, in sediments (mg/g) the values were 16.0, 0.65, 0.09, 0.32, and 0.01 for the same metals. The mean concentrations of metals in fish (mg/g) were Fe (2.96), Zn (1.87), Cu (0.03), Pb (0.08) and Cd (<0.001), while for plankton (mg/L) were Fe (13.2), Zn (2.09), Cu (0.06), Pb (<0.001) and Cd (<0.001). The pollution index in the sediment was low. Trace metal concentrations in water, sediment and biota were below the maximum permissible levels recommended by National standard, but aquatic environment should be monitored regularly to avoid excess intake of metal into the water body.

### **INTRODUCTION**

Pollution causes unfavourable changes in the environment which could affect the components of the ecosystem. Some of the sources of pollution include industrial effluents, sewage, agricultural discharge and other household residue. Most aquatic environments have been influenced by a number of stressors which significantly destroys the biodiversity. These stressors include population growth pressure, urbanization, pollution from industries, mining, oil and gas activities, erosion, sea rising, sand storm, drought, desertification etc. Environmental protection is a major global concern. In recent times, according to Palaniappan *et al.*, (2010) it has become more challenging to maintain the quality of the aquatic ecosystem. Water pollution is the introduction of contaminant by physical, chemical, biological or radioactive agents in the natural water body (Hogan, 2013). According to Hossain *et al.*, (2012) there are pollutants that naturally enter the aquatic system e.g. from natural fires, volcanoes and oil seeps etc. Some non-natural sources of water pollution include underground storage leakages, industrial waste, marine dumping, (Gambhir *et al.*, 2012). The coastal and brackish water environment is usually identified with large industrial settlements and urbanization with resultant effluent discharge which results in the accumulation of heavy metal (Ridgway and Shimmiel, 2002). The brackish water environment is endangered by discharges of untreated waste and industrial effluent. This eventually causes harm to the sustainability of the living resources and public health. The waste transports high level of

toxicants, especially the heavy metals which have the ability to accumulate in the basic food chain and also move up to the higher trophic level. Heavy metals are important environmental pollutants and their toxicity is a challenge because of their ecological, nutritional, environmental and evolutionary effects (Nagajyoti *et al.*, 2010; Jaishankar *et al.*, 2013). Activities of human and industries have caused various discharge of pollutants into the marine environment endangering the health of the population and destroying the quality of the environment by making the water bodies unfit (Abowei and Sikoki, 2005). The main entry routes of toxic substances into the surface water are normally via point source and non-point source.

Ikpukulu-ama creek is a small marine tidal creek that flows through in the Niger Delta region. It is bordered by Okrika Island with Bie-ama community inhabiting the area. The area surrounding the creek has been urbanized and industrialized due to quest for crude oil and other natural resources. Large industries such as NLNG Dock, Almarine Base, Federal Fishing Terminal, Dredging Company and John Holt Plc are within the creek. The deliberate and accidental discharges from these industries, in addition to human waste, and illegal refineries and bunkering loading activities may be detrimental to the quality of the creek.

### **Study Area**

The Ikpukulu-ama creek is a small tidal creek in the Niger Delta region (Figure 3.1) that flow through a small fishing settlement behind Government Comprehensive Secondary School, Borikiri in Port Harcourt City Local Government Area of Rivers State. The creek is a tributary of the Bonny River that flows into the Atlantic Ocean. Some industries NLNG Dock, Almarine base, Federal Fishing Terminal, Dredging Company and John Holt Plc located within this area are involved in oil and gas servicing, marine technology, fishing, transportation and other anthropogenic activities.

### **Materials and methods**

#### **Study sites**

Three sampling stations were located along the creek. The vegetation of Ikpukulu-ama creek are sparse, full of dead roots of mangrove trees (*Rhizophora racemosa*, *Aveicinnia africana*) with few pockets of Nipa palms (*Nypa fruticans*) trees found in some stations.

Station 1 is located upstream with latitude 04°44'01.4" N and longitude 007°01'40.4" E (Figure 3.2), it has a distance of 3m from station 3. It has no vegetation at all. The major activities of this station includes; bathing, washing and fecal discharge.

Station 2 is located midstream with latitude of 04°44'02.2"N and longitude of 007°1'33.4"E (Figure 3.3), it has a Distance of 2m from station 1. The station lacked vegetation. Fecal discharge, illegal bunkering discharge and waste discharge, also effluents from bile creek are found there. The surface of the water is filled with oil film.

Station 3 is located downstream with latitude of 04°43'57.4"N and longitude of 007°01'43.6"E (Figure 3.4), it has a distance of 3m from station 1. It has good vegetation Nipa palms (*Nypa fruticans*), some dead roots of mangrove trees and fresh *Rhizophora racemosa*, *Avecinnia africana*. Fecal discharge is the major activity of this station.

### **Sample collections**

Composite sampling method was used in collection of sample in each sampling station during high tide. Sample collections were carried out monthly covering a period of March to August, 2017. Triplicates samples were collected from each location. Water samples were collected from under the surface of the water while sediment samples were collected using the Ekman grab sampler at the same stations for water collection. Sediment was put in sampling bottles (Schott glass bottles) and transported with the water on ice to the laboratory. In the laboratory samples were stored at 4°C until analysis. Fishes were collected from the local fishermen and stored in a cooler packed with ice block in order to maintain the freshness and later transported to the laboratory. Plankton (zoo- and phytoplankton) samples were collected with the help of a plankton net (mesh size: 20 µm) through vertical hauls from the upper layer of 10 cm. Filtered plankton samples were kept in sampling bottles and preserved in ice packs.

### **Analysis of samples**

All water samples were analyzed directly while sediment samples were oven dried, homogenized and sieved to remove large particulates. One gram (3 g) of each powdered sample was weighed into conical flasks with 2ml of Sulphuric acid, 2ml of Nitric acid and 1ml of Perchloric acid allowed to digest for 5 minutes. The solution was filtered using Whatmann No. 1 cellulose filter paper and the filtrate was made up to 100 ml using deionised water.

The fish samples were wrapped with Aluminum foil and oven dried at 105 °C. After drying to constant weight, the dried fish samples were crushed in a small porcelain mortar and pestle to a homogenized powder. One gram (3 g) of the dried fish powder was heated in a muffle furnace temperature of 630 °C for 3hours. The ash was dissolved in 2ml of Sulphuric acid, 2ml of Nitric acid and 1ml of Perchloric acid was heated on an electro-thermal heater hotplate. The solution was filtered using Whatmann No. 1 cellulose filter paper and solution was diluted to 100ml with distilled water and the trace metal contents were determined using an atomic absorption spectrophotometer (AAS) Agilent technologies 4210 MP-AES model as described in Standard Methods (APHA, 1995). Plankton samples were acidified with 1ml of concentrated HCL analyzed according to the method described by Kalay *et al.*, (1999). The concentration of the individual metal ions of the samples were extrapolated from the standard graph of the calibrated curves of the individual metal ions and was presented in parts per million.

### **Quality control**

Each sample was analyzed in triplicate, and a blank determination was carried out with every batch of samples. All reagents used were of AnalaR grade and all glassware and polyethylene were properly cleaned with acid – cleansing reagents and rinsed thoroughly with distilled deionized water. The blank values were generally low and below the detection limits of the instrument for the metals.

### **Statistical analysis**

Statistical package for social sciences (SPSS version 22) IBM 2013 were used. Two way-ANOVA and Duncan Multiple Range Test were used to determine the significant difference at  $p < 0.05$ .

**Pollution load index (PLI):** Pollution load index for each site was evaluated as indicated by Tomilson *et al.*, (1980). Pollution load index=  $(CF_1 * CF_2 * \dots * CF_n)^{1/n}$  Where, n is the number of metals (five in the present study) and CF is the contamination factor. The contamination factor can be calculated from; Contamination factor (CF) = metal concentration in sediments/Background values of the metal. The PLI value > 1 is polluted while PLI value < 1 indicate no pollution (Chakravarty and Patgiri, 2009; Seshan *et al.*, 2010). The background value used is that of average shale (Turekian and Wedepohl, 1961).

## **RESULTS**

The mean trace metal concentrations in mg/L with standard error of the water samples from the study area is recorded in Table 1. These values were compared with the Nigerian Industrial Standard (NIS) and world Health Organization (WHO). The concentration of Fe varied significantly across the stations ranging from 4.1986 – 6.1955. Fe was highest in station 2 and least in station 1. The concentration of Zn varied significantly across the stations ranging from 0.4371–1.4051. Zn was highest in station 2 and least in station 3. The concentration of Cu varied significantly across the stations ranging from 0.0264 – 0.0414. Cu was highest in station 2 and least in station 1. The concentration of Pb varied significantly across the stations. Pb was highest in station 1 and least in station 2. Concentration of Cd had no significant difference across the stations. In station 1, the order of concentration of metal for the water samples was Cd<Cu<Pb<Zn<Fe and station 2 and station 3 Cd<Cu<Zn<Pb<Fe.

**TABLE 1. MEAN CONCENTRATIONS OF TRACE METAL IN WATER (mg/L) OF IKPUKULU-AMA CREEK DURING THE STUDY COMPARED WITH STANDARDS**

METALS	STATION 1	STATION 2	STATION 3	WHO(2011)	NIS(2007)
Fe	4.1986±0.02 <sup>a</sup>	6.1955±0.02 <sup>c</sup>	5.0838±0.02 <sup>b</sup>	-	-
Zn	0.6332±0.00 <sup>b</sup>	1.4051±0.00 <sup>c</sup>	0.4371±0.00 <sup>a</sup>	15.0	3.0
Cu	0.0291±0.00 <sup>b</sup>	0.0414±0.00 <sup>c</sup>	0.0264±0.00 <sup>a</sup>	2.0	1.0
Pb	0.5063±0.01 <sup>c</sup>	0.4288±0.01 <sup>a</sup>	0.4523±0.00 <sup>b</sup>	0.01	0.01
Cd	0.0149±0.00 <sup>b</sup>	0.0108±0.00 <sup>b</sup>	0.0108±0.00 <sup>b</sup>	0.003	0.003

With same superscript there is no significant difference (p<0.05).

NIS- Nigerian Industrial Standard, WHO- World Health Organization

The mean trace metal concentration in mg/g with standard error in the sediment samples from the study area are recorded in Table 2. These values were compared with Department of Petroleum Resources (DPR) Nigeria. The concentration of Fe varied significantly across the stations ranging from 13.801–19.023. Fe was highest to be station 2 and least in station 3. The concentration of Zn varied significantly across the stations ranging from 0.409–0.993. Zn was highest in station 2 and least in station 3. The concentration of Cu varied significantly across the stations ranging from 0.019 – 0.202. Cu was highest in station 3 and least in station 1. The concentration of Pb varied significantly across the stations ranging from 0.270 – 0.389. Pb was highest in station 3 and least in station 1. The concentration of Cd showed little variation across the stations, ranging from 0.012– 0.018. Cd was highest in station 2 and least in station 1 and 3. The result revealed trace metal distribution in sediment to be the same in all stations Cd<Cu<Pb<Zn<Fe. The PLI was below 1 which indicates that the sediment is not contaminated.

**TABLE 2. MEAN CONCENTRATIONS OF TRACE METALS IN SEDIMENTS (mg/g) OF IKPUKULU-AMA CREEK DURING THE STUDY COMPARED WITH STANDARDS**

METALS	STATION 1	STATION 2	STATION 3	DPR(2002)
Fe	15.2098±0.04 <sup>b</sup>	19.0233± 0.04 <sup>c</sup>	13.8014±0.04 <sup>a</sup>	20
Zn	0.5319±0.00 <sup>b</sup>	0.9932±0.00 <sup>c</sup>	0.4085±0.00 <sup>a</sup>	50-300
Cu	0.0189±0.00 <sup>a</sup>	0.0418±0.00 <sup>c</sup>	0.2022±0.00 <sup>b</sup>	35/20
Pb	0.2701±0.08 <sup>a</sup>	0.3145±0.08 <sup>b</sup>	0.3891±0.08 <sup>c</sup>	2-20
Cd	0.0121±0.00 <sup>a</sup>	0.0183±0.00 <sup>b</sup>	0.0121±0.00 <sup>a</sup>	0.03-0.3
PLI	0.0033	0.0052	0.0052	
Pollution condition	(<1)	(<1)	(<1)	

PLI= Pollution Load Index; with same superscript there is no significant difference ( $p<0.05$ ).

DPR- Department of Petroleum Resources

The mean trace metal concentration in mg/g with standard error in the fish samples from the study area is recorded in Table 3. These values were compared with Federal Environmental Protection Agency (FEPA) Nigeria. The concentration of Fe varied significantly across the stations ranging from 2.404 – 3.471. Fe was highest in station 2 and least in station 3. The concentration of Zn varied significantly across the stations ranging from 1.538 – 2.385. Zn was highest in station 2 and least in station 3. The concentration of Cu varied significantly across the stations ranging from 0.026 – 0.041. Cu was highest in station 2 and least in 0.026. The concentration of Pb varied significantly across the stations ranging from 0.061 – 0.094. Pb was highest in station 1 and least in station 2. The concentration of Cd across the stations from 0.000-0.001, this is below detectable limit ( $<0.001$ ), hence was not detected. The result revealed trace metal distribution in fish to be the same in all stations  $Cd<Cu<Pb<Zn<Fe$ .

**TABLE 3. MEAN CONCENTRATIONS OF TRACE METALS IN FISH (mg/g) OF IKPUKULU-AMA CREEK DURING THE STUDY COMPARED WITH STANDARDS**

METALS	STATION 1	STATION 2	STATION 3	WHO/FEPA(2003)
Fe	3.0068±0.00 <sup>b</sup>	3.4709±0.00 <sup>c</sup>	2.4037±0.00 <sup>b</sup>	0.5
Zn	1.6883±0.01 <sup>b</sup>	2.3849±0.01 <sup>c</sup>	1.5381±0.01 <sup>a</sup>	30.0(FAO 1983)
Cu	0.0291±0.00 <sup>b</sup>	0.0414±0.00 <sup>c</sup>	0.0264±0.00 <sup>a</sup>	3.0
Pb	0.0938±0.00 <sup>c</sup>	0.0609±0.00 <sup>a</sup>	0.0767±0.00 <sup>b</sup>	2.0
Cd	ND	ND	ND	0.5

ND: Not detected; with same superscript there is no significant difference ( $p>0.05$ ). FEPA- Federal Environmental Protection Agency

The mean trace metal concentrations in mg/L with standard error in the plankton samples from the study area are recorded in Table 4. The concentration of Fe varied significantly across the stations ranging from 11.798 – 15.821. Fe was highest in station 2 and least in station 3. The concentration of Zn varied significantly across the stations ranging from 1.465 – 2.705. Zn was highest in station 2 and least in station 1. The concentration of Cu varied significantly across the stations ranging from 0.0156 – 0.0158. Cu was highest in station 1 and least in station 3. The concentration of Pb and Cd ranges across the stations from 0.000-0.001, this is below detectable limit ( $<0.001$ ), hence

was not detected. The result revealed trace metal distribution in plankton to be the same in all stations  $Cd=Pb<Cu<Zn<Fe$ .

**TABLE 4. MEAN CONCENTRATIONS OF TRACE METALS IN PLANKTON (mg/L) OF IKPUKULU-AMA CREEK DURING THE STUDY PERIOD**

METALS	STATION 1	STATION 2	STATION 3
Fe	12.0309±0.11 <sup>b</sup>	15.8214±0.11 <sup>c</sup>	11.7982±0.11 <sup>a</sup>
Zn	1.4654±0.04 <sup>a</sup>	2.7046±0.04 <sup>c</sup>	2.1261±0.04 <sup>b</sup>
Cu	0.1583±0.00 <sup>a</sup>	0.0163±0.00 <sup>b</sup>	0.0156±0.00 <sup>a</sup>
Pb	ND	ND	ND
Cd	ND	ND	ND

ND: Not detected; With same superscript there is no significant difference ( $p>0.05$ )

## **DISCUSSION**

Metals occur naturally in low concentrations. According to Bakarat *et al.*, (2012), metals enter into the water via oxidation-reduction reactions, adsorption-desorption reaction, sedimentation re-suspension and degrading organisms. This study reveals that metals when compared with WHO (2011) and NIS (2007) are in a satisfactory level for drinking and surface water. Fe (Iron) is the highest because it is one of the most abundant metals in nature. This is in line with the work of Vincent-Akpu *et al.*, (2015) that Iron concentration was higher than all metals in her work in Bodo creek Niger Delta. This study reveals low values of trace metals when compared with other water bodies in Port Harcourt, this according to Chindah *et al.*, (2004) could be caused by high energy of current, volume of water that dilutes and adequate flushing of the system. However high concentrations of trace metals were seen in Woji River which receives effluent from domestic, industrial and agricultural site (Leton and Akpila, 2008). According to Bader *et al.*, (2015), the purpose why the metal concentrations found in water do not indicate the relative contributions of pollution from activities in the creek. They said it might be caused by some physico-chemical processes such as tapping, precipitation, settling and storage of pollutants in the sediments and bioaccumulation in the aquatic organisms. This study revealed that sediment contained higher metal concentration when compared with their concentrations in water. According to Gupta *et al.*, (2009) sediments are sinks for metals and other pollutants of the marine ecosystem. Fernandes *et al.*, (2007) reveals that metal concentration in sediment affects water quality and bioaccumulates in aquatic organisms which causes long-term effect on the human health and the environment. The results indicated that low concentrations when compared with DPR (2002) where Fe as 20ppm, Zn as 50-300ppm, Cu as 35/20ppm, Pb as 2-20ppm and Cd as 0.03-0.3ppm. Obire *et al.*, (2003) and (Asaolu and Olaofe, 2004) also confirms high metal concentration in sediments than in water. Belize *et al.*, (2003) states that when there is high pH, absorption of metals is promoted while low pH stops metal retention by sediment. Low levels of cadmium is in line with previous works done in the Niger Delta by (Otitaju and Otitaju, 2013; Vincent-Akpu and Babatunde, 2014; Ideriah *et al.*, 2012). Low levels of Pb is in line with previous works in the Niger Delta (Vincent-Akpu and Yanadi 2014; Adeleye *et al.*, 2011). Metals that usually goes into solution first would be the metals within the top of the surface sediment, this will give an indication of current heavy metal concentration in the sediment. The present study indicates heavy metals have mean values lower than WHO (2003) and FEPA (2003) where Fe is 0.5ppm, Cu is 3.0ppm, Pb is 2.0ppm and Cd -

0.5 while Zn -30ppm FAO (1983), and recommended limits in fish except that of Fe which was higher than the permissible standard. This could be as a result of the differences in fish sizes, ages and sampling periods. Low levels of cadmium in fish is in line with Davies *et al.*, (2006). According to Adeyeye (1996) difference in metal concentration in fish could be dependent on the fish species, while Idodo-Umeh (2002) said bigger fishes accumulate high metal concentrations than smaller ones. Fe is necessary for red blood formation and also plays a vital role in some metabolic processes. Iron is an essential trace metal, but can be harmful to living organisms even at low exposure.

According to Elamci *et al.*, (2007), heavy metal concentration in plankton is dependent on the following; absorption properties of the plankton species, metal content in water and partially from the sediment. Heavy metals concentrations in plankton is dependent on several factors, which includes productivity of the body of water, the physicochemical properties of the water, quantitative and qualitative species composition of zoo- and phytoplankton, the capacity of heavy metal absorbance, and the seasons (Kerrison *et al.*, 1998; Radwan *et al.*, 1990; Elmaci *et al.*, 2007). The present study revealed low concentrations in plankton when compared with the metal concentrations in sediment and water.

## **CONCLUSION**

Pollution index in Ikpukulu-ama creek was low, because the creek is tidal with continuous ebb and flows which guarantees adequate flushing of the creek. The heavy metals results obtained indicated that these metals from this creek are within the permissible limit but the risk of deleterious effects after long period cannot be exempted. This is due to the fact that this water body acts as a receptor for domestic and industrial wastes including agricultural run-offs. It is very important to control the level of discharge of the pollutants from non-point and point sources. Ensuring healthy status of the creek will improve the quality of the water and protect the biodiversity of the water body without putting-on health risk to humans who eat aquatic products from the water body. It is important for public enlightenment on the significance of not depositing waste in the water bodies and drainage ways. This will help in reducing occurrence of water pollution and permit adequate use of water resources.

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