



## Assessment of heavy metals concentration in sediments of Utibete River, Eastern Obolo, Southeastern Nigeria using particle induced X-ray emission (PIXE) technique

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### Abstract

The contamination of heavy metals constituent of sediments in Utibete River in Eastern Obolo, Akwa Ibom, Nigeria was assessed. In application of PIXE techniques, ten heavy metals, Ti, Cr, Mn, Fe, Cu, Zn, Rb, Sr, Zr and Pb, were determined from each of the sediment samples obtained at 6 sampling sites. The concentration of Fe was the highest in all the sampling sites ranged 11371.6 – 45095.9 ppm.

Pollution indices such as enrichment factor (EF), geo-accumulation index (I<sub>geo</sub>) and contamination factor (CF) were employed to assess the toxicity threat posed on that part of the ecosystem by the investigated heavy metals. The results of the three pollutions indices were similar, indicating that that part of that River was highly polluted with Cr, Rb, Zr, Zn, and Mn, Pb at some point. Pollution lead index (PLI) employed for contamination assessment showed 5 of the sampling sites of Utibete River were contaminated with heavy metals determined with the exception of site 2 where there is low contamination of those heavy metals. These high inputs of the heavy metals might be due to pronounced anthropogenic influences around the studied areas. Analysis of multivariate statistics such as Pearson's Correlation coefficient and cluster analysis were also applied to ascertain the relationship and sources of investigated heavy

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metals. Both positive and negative correlations were observed among some heavy metals while the cluster analysis revealed four main clusters.

**Keywords** Heavy metals; sediments; Particle induced emission technique (PIXE); enrichments factor (EF); geo-accumulation index (Igeo); Contamination factor (CF); pollution load index (PLI).

## 1.0 Introduction

The particle induced X-ray emission (PIXE) technique developed by Johansson and colleagues in Lung, Sweden, in the 70's has been used since then in a high number of studies requiring detection and quantification of trace elements at high sensitivity (better than 1 mg/kg) (Solis *et al.*, 2006). Most of the other analytical techniques being used are destructive, while PIXE is a highly sensitive but relatively nondestructive technique (Rao *et al.*, 2010). PIXE has been proven to be outstanding in the determination of the concentrations of various elements at parts per million (ppm) levels (Preteasa *et al.*, 2004). PIXE is a multi-elemental analytical technique that can provide concentrations of many elements with atomic numbers between 11 and 92 (sodium and uranium) (Nejedly *et al.*, 2003). In PIXE technique, the samples are bombarded with particles (Maxwell *et al.*, 1995; Miranda *et al.*, 2000; Guereca and Ruvalcaba-Sil, 2005; Solis *et al.*, 2006; MeV protons generally) accelerated in an accelerator. The PIXE technique is based on spectroscopy of characteristic X-rays induced from a sample by accelerated ions (Johansson and Johansson, 1976; Johansson *et al.*, 1995). Sediments which are environmental samples play fundamental role in toxic compounds distribution in aquatic systems and in the evaluation of current state and course of changes taking place in the environment. It is a naturally

occurring material that is broken down by processes of weathering and erosion which can further be transported by factors such as action of wind, water etc. It contains particles that are either environmental friendly or toxic to the biosphere. Golaś *et al.*, (2004) observed that physical, chemical, biochemical and geochemical processes that influence toxic compounds and elements fate in sediments are numerous and complex (For example; adsorption-desorption, oxidation-reduction, ion-exchange, biological activity). Potentially harmful heavy metals are effectively accumulated in the sediment and in certain conditions they can be readily released into water in an unpredictable way. On the other hand, concentrations of elements in sediments can serve as an indicator of water quality (Golaś *et al.*, 2004). Sediments aggregates contaminants over time, and are in constant flux with the overlying water column (Usero *et al.*, 2005; Bai *et al.*, 2010; Ayejuyo *et al.*, 2010; Deng *et al.*, 2010). According to Ra *et al.*, (2013) and Burton, (2002), sediments show less variation in time and space, they are preferable monitoring tools to allow consistent assessment of temporal and spatial contamination. Ra *et al.*, (2013) and Caccia *et al.*, (2003) reported that monitoring of metal levels in sediments are useful indicators of anthropogenic inputs to evaluate metal contamination and to predict influence on marine ecosystem.

Heavy metals are naturally occurring elements that have a high atomic weight and a density at least 5 times greater than that of water. The levels of the contamination of heavy metals depend on the anthropogenic influences on ecosystem. According to Demirbas *et al.*, (2005), heavy metals are regarded as serious pollutants of aquatic ecosystems because of their environmental persistence, toxicity and ability to

be incorporated into food chains. Heavy metals may enter into aquatic ecosystems from anthropogenic sources, such as industrial wastewater discharges, sewage wastewater, fossil fuel combustion and atmospheric deposition (Linnik and Zubenko, 2000; Campbell, 2001; Lwanga et al., 2003; El Diwani and El Rafie, 2008; Idrees, 2009; Sekabira et al., 2010).

The rivers and other water bodies are the ultimate repository of man's wastes (Yusuf and Osibanjo, 2004; Awofolu et al., 2005; Usero et al., 2005; Adebowale et al., 2008; Adeniyi et al., 2008; Xianfei et al., 2009; Nassif and Saade, 2010; Kucuksezgin et al., 2010; Adeniyi et al., 2011) through which some toxic heavy metals got sunk into the coastal sediments. The highly dynamic nature of the marine environment allows for a rapid assimilation of waste materials by processes such as dilution, dispersal, oxidation and degradation into sediment (Adeniyi et al., 2011). The occurrence of elevated levels of metals especially in the sediments can be attributed to anthropogenic influences rather than natural enrichment of the sediment by geological weathering (Adebowale et al., 2008; Wang et al., 2010; Bai et al., 2010; Adeniyi et al., 2011). The distribution of metals in water and sediments provides a record of the spatial and temporal history of pollution in a particular region or ecosystem (Awofolu et al., 2005; Xianfei et al., 2009; Adeniyi et al., 2011).

The anthropogenic activities in this area among which are uncontrollable dispose of wastes to the water body and the tilling of the river base in search of aquatic food have been our concern. Such acts have made the sediments vulnerable in storing pollutants and other materials essential to the ecosystem. The porosity of the sediments determines the extent to which the sediments will act as a sink or reservoir to the pollutants.

Thus sediments with high porosity will sink heavy metals easily than those with low porosity. Pollutants and other heavy metals can easily be transported from one point to another from the sediments of high porosity when they are tilled either by anthropogenic or natural factors than those within sediments of low porosity.

Utibete creek has been the area where crude oil pipes were laid and crude oil had had been transported through these pipes to the storage tank by Shell Plc for some decades although operations have been stopped for some years now. During and after the laying of the pipes, some heavy metals could have been introduced into the ecosystem. The pipes that have stayed for some years could have been corroded thereby posing negative disturbances on health of aquatic functions and other part of ecosystem. Presently, the area is stressed by anthropogenic influences thus there is need of assessing the level of pollution from heavy metals present in the area which is the basis of this research. The aims of this study were; to determine the spatial distribution of heavy metals, to evaluate the contamination status of heavy metals using different metal assessment indices such as enrichment factors (EFs), geo-accumulation index (*I*<sub>geo</sub>), contamination factor and pollution load index (PLI), to assess ecological risk of these heavy metals by comparison with sediment quality guidelines (SQGs), and to determine the sources and relationships of those heavy metals using analysis of multivariate statistics such as Pearson's correlation coefficient (PCC) and cluster analysis (CA).

## 2.0 Materials and Methods

### 2.1 Study Area and Sediment Samples Analysis

Utibete River is one of the creeks in Eastern Obolo that tiling of riverbed in search of aquatic

food is a common practice among the dwellers. It is linked with Okoro River to separate Okoroile (Okoroete) from Okorombokho communities. Six (6) sites (stations) were sampled within the coordinates of Longitude 007°45'04" and 007°45'19" degree east, and Latitude 04°32'18" and 04°32'33" degree north respectively using Van Veen sampler. The sampling procedure employed was the procedure recommended by Sandler and Herut, (2006) and IAEA, (2003) of which the triangular grid point was considered to obtain the composite sediment samples for every sampling point. The sediment samples for each of the three grid points were mixed to form the composite sediment samples with the aids of disposable hand glove and were stored in a polyethelene bag correctly labeled for every sampling site. Unwanted particles were removed from the composite sediment samples. The composite sediment samples were transferred to drug envelope correctly labeled. The samples were conveyed to the Centre for Energy and Research Development (CERD) in Obafemi Awolowo University (OAU), Ile-Ife, where they were exposed to dryness for some weeks. They were further subjected to pro-analytical treatment such as homogenization and pulverization of the composite sediment samples before the Particle Induced X-ray Emission (PIXE) analysis. The analysis result which showed the concentrations of the heavy metals is shown in Table 1. Three pollution indices namely, Enrichment factor (EF), geo-accumulation index (Igeo) and contamination factor (CF) were employed to assess the contamination status of those heavy metals.

As proposed by Simex and Helz (1981), the enrichment factor was employed to assess the degree of contamination and to understand the distribution of the elements of anthropogenic origin from sites by individual elements in

sediments. In this study, Fe is chosen as the normalizing element while determining EF-values, since in wetlands it is mainly supplied from sediments and is one of the widely used reference element (Loska *et al.*, 2003; Chakravarty and Patgiri, 2009; Kothai *et al.*, 2009; Addo *et al.*, 2012; Etc). Ekaete *et al.*, (2015) have indicated that several researchers have also employed Al and Si as reference elements in the calculation of Enrichment factor (EF). According to Loska *et al.*, (2003), an element also qualifies as a reference one if it is of low occurrence variability and is present in the environment in trace amount.

In this study, iron is also used as a conservative tracer to differentiate natural from anthropogenic components. According to Ho *et al.*, (2010), the metal (M) EF is defined as:

$$EF = \frac{[C_{metal}/C_{norm(Fe)}]_{sediment}}{[C_{metal}/C_{norm(Fe)}]_{backgroundsource}} \quad (1)$$

where  $[C_{metal}/C_{norm(Fe)}]_{sediment}$  and  $[C_{metal}/C_{norm(Fe)}]_{backgroundsource}$  are the concentrations of metal and normaliser in the sediment samples and the background source respectively. In this approach, the normaliser is used to compensate for the differences in grain size and mineralogical composition between the sediment and the reference material (Ho *et al.*, 2010).

EF values were interpreted as suggested by Ho *et al.*, (2010), and Birch (2003), where  $EF \leq 1$  indicates no enrichment;  $1 < EF \leq 3$ : minor enrichment;  $3 < EF \leq 5$ : moderate enrichment;  $5 < EF \leq 10$ : moderately severe enrichment;  $10 < EF \leq 25$ : severe enrichment;  $25 < EF \leq 50$ : very severe enrichment;  $EF > 50$  is extremely severe enrichment. The background value (reference) is that of average shale (Ho *et al.*, 2010). Elements which are naturally derived

have an EF value of nearly unity, while elements of anthropogenic origin have EF values of several orders of magnitudes. Index of geo-accumulation was used to assess heavy metal accumulation in sediments as introduced by Muller (1969) to measure the degree of metal pollution in aquatic sediments studies (Praveena *et al.*, 2007; Praveena *et al.*, 2008; Chakravarty and Patgiri, 2009) and is given as:

$$I_{geo} = \text{Log} 2 \left( \frac{C_m}{1.5 C_o} \right) \quad (2)$$

where  $c_m$  is the measured concentration of the heavy metal in the sediment samples; and  $C_o$  is the concentration of the heavy metal in the background source; the factor 1.5 is used to compensate for possible variations of the

reference data due to lithogenic effects (Aksu *et al.*, 1997). Based on the  $I_{geo}$  value, the degree of heavy metal contamination is classified as follows: 1)  $I_{geo} < 0$ : unpolluted; 2)  $0 \leq I_{geo} < 1$ : unpolluted to moderately polluted; 3)  $1 \leq I_{geo} < 2$ : moderately polluted; 4)  $2 \leq I_{geo} < 3$ : moderately to strong polluted; 5)  $3 \leq I_{geo} < 4$ : strongly polluted; 6)  $4 \leq I_{geo} < 5$ : strongly to very strongly polluted; 7)  $I_{geo} \geq 5$ : very strongly polluted. Contamination factor parameter is expressed as:

$$CF = C_{\text{metal}} / C_{\text{background}} \quad (3)$$

where CF is the contamination factor,  $C_{\text{metal}}$  is the concentration of pollutant,  $C_{\text{background}}$  is the background source value for the metal.

**Table 1:** Concentrations (ppm) of heavy metals in sediment sample of Utibete River in Eastern Obolo, Akwa Ibom State, Nigeria

Heavy Metals	UTR1	UTR2	UTR3	UTR4	UTR5	UTR6
Ti	4773.60 ±39.14	2366.00 ±2839	4232.60 ±35.13	7626.40 ±46.52	4820.30 ±38.56	5627.40 ±39.95
Cr	271.80 ±10.49	249.70 ±8.79	255.10 ±9.18	175.90 ±9.62	166.30 ±9.71	178.10 ±8.78
Mn	166.60 ±22.44	78.60 ±19.46	254.60 ±21.39	1147.50 ±31.10	193.50 ±19.83	201.00 ±19.62
Fe	11371.60 ±59.13	16617.30 ±69.79	22268.10 ±80.17	45095.90 ±117.25	18299.80 ±73.20	18022.10 ±73.89
Cu	9.2±1.65	6.2±1.75	5.0±1.51	7.1±2.17	3.80±1.31	5.8±1.41
Zn	28.40 ±12.46	47.20 ±11.07	54.90 ±9.93	83.20 ±16.03	54.30±9.34	46.50 ±9.96
Rb	45.80 ±26.39	ND	79.50 ±21.69	54.10 ±22.21	85.60 ±22.68	29.80 ±18.17
Sr	44.30 ±24.64	ND	20.00 ±18.40	61.70 ±19.00	100.20 ±20.77	87.60 ±19.86
Zr	674.50 ±71.63	171.60 ±38.32	739.00 ±68.21	338.10 ±55.48	742.40 ±76.17	911.50 ±75.20
Pb	20.90 ±20.12	26.60 ±9.55	ND	15.00 ±12.06	9.9±9.79	15.5±8.88

The CF was employed to reflect the metal enrichment in the sediment of Utibete River, Eastern Obolo, Akwa Ibom State, Nigeria (Table 2). The CF was classified into four groups

according to Nasr *et al.* (2006), and Mmolawa *et al.*, (2011); where the contamination factor  $CF < 1$  refers to low contamination;  $1 \leq CF < 3$  means moderate contamination;  $3 \leq CF \leq 6$  indicates

high contamination and  $CF > 6$  indicates very high contamination.

**Table 2:** Contamination Factor (CF) and Pollution Lead Index (PLI) of Heavy Metals in Utibete Sediments

Station	Ti	Cr	Mn	Fe	Cu	Zn	Rb	Sr	Zr	Pb	PLI
1	1.05	3.92	0.71	0.57	0.92	0.58	1.21	0.65	1.78	1.78	1.08
2	0.52	3.60	0.34	0.83	0.62	0.97	ND	ND	0.45	2.27	0.87
3	0.93	3.68	1.09	1.11	0.50	1.12	2.09	0.29	1.95	ND	1.12
4	1.67	2.54	4.89	2.25	0.71	1.70	1.42	0.91	0.91	1.28	1.56
5	1.06	2.40	0.83	0.91	0.38	1.11	2.25	1.47	1.96	0.85	1.16
6	1.23	2.57	0.86	0.90	0.58	0.95	0.78	1.29	2.41	1.32	1.16

Multivariate analysis such as Pearson's correlation coefficients and Cluster analysis were also employed to determine the sources and relationships of those heavy metals. Pearson's correlation coefficient is a statistical measure of the strength of a linear relationship between paired data. In a sample, it is denoted by  $r$  and is by design constrained as follows:

$$-1 \leq r \leq 1 \quad (4)$$

Such that

- Positive values denote positive linear correlation;
- Negative values denote negative linear correlation;
- A value of 0 denotes no linear correlation;
- The closer the value is to 1 or -1, the stronger the linear correlation.

In order to establish relations among heavy metals and determine the common sources of these metals in the study area, a Pearson's correlation matrix (Table 3) was calculated for heavy metals in sediment samples. The most used of the cluster analysis is classification. That is, subjects are separated into groups such that each subject is more similar to other subjects in its group than to subjects outside the group. That is, cluster analysis (CA) classifies the studies of heavy metals in terms of their similarity and difference. Cluster analysis was performed on

the data set using average linkage between groups (rescaled distance cluster) to visualize elemental groupings in the studied sediments. The results are illustrated in the dendrogram on Hierarchical cluster analysis. Elements belonging to the same cluster are likely to have originated from a common source (Denutsui *et al.*, 2011). Pollution load Index (PLI) parameter was also employed to assess if in general a sampling site is polluted or not. The Pollution Load Index has been determined as the  $n$ th root of the product of  $nCF$  for the entire sampling sites and can be expressed as:

$$PLI = (CF_1 \times CF_2 \times CF_3 \times CF_4)^{1/n} \quad (5)$$

where  $CF_1$  is the contamination factor for the first metal,  $CF_2$  is the contamination factor for the second metals respectively and  $n$  is the total number of the heavy metals determined at the respective sampling site. This provides the means of assessing the pollution of heavy metals at the respective sampling site. The overall pollution threat of all the heavy metals at each of the sediment sampling sites of Utibete River is considered according to Naglaa and Samir, (2015), and Tomlinson *et al.*, (1980) interpretation of PLI (Table 2) which shows that PLI values greater than 1 are indicative of the existence of pollution while PLI values less than 1 imply no pollution.

**Table 3:** Pearson's Correlation Coefficient matrix of heavy metals in sediment samples of Utibete River, Eastern Obolo, Akwa Ibom, Nigeria.

	Ti	Cr	Mn	Fe	Cu	Zn	Rb	Sr	Zr	Pb
Ti	1									
Cr	-.614	1								
Mn	.829*	-.439	1							
Fe	.735	-.480	.968**	1						
Cu	.174	.497	.193	-.009	1					
Zn	.611	-.594	.860*	.954**	-.299	1				
Rb	.369	-.255	.191	.193	-.412	.263	1			
Sr	.608	-.824*	.175	.112	-.296	.161	.443	1		
Zr	.200	-.198	-.336	-.376	-.248	-.353	.512	.602	1	
Pb	-.245	.169	-.114	-.207	.562	-.308	-.850*	-.224	-.559	1

\*. Correlation is significant at the 0.05 level (2-tailed).

\*\* . Correlation is significant at the 0.01 level (2-tailed).

### 3.0 Results and Discussion

Knowing the concentrations of heavy metals in sediments aids in determining their toxicity threat pose by these heavy metals of high concentrations in ecosystem through which their influx can be managed by relevant authorities. Multi-elemental analysis of sediments may reveal the presence of heavy metals which are contaminants and may have toxic influence on ground and surface and also on plants and humans (Suciu *et al.*, 2008).

The heavy metals determined in this study include titanium (Ti), chromium (Cr), manganese (Mn), iron (Fe), copper (Cu), zinc (Zn), rubidium (Rb), strontium (Sr), zirconium (Zr), and lead (Pb). Sediment Quality Guidelines include a threshold effect concentration (TEC) obtained from the analysis of the standard (control) material and a probable effect concentration (PEC) obtained from the reference material and their respective concentrations of elements in samples sediments investigated. The

precision and accuracy of the analytical method employed in this study was accomplished by analyzing the standard (control) material also known as threshold effect concentration (TEC) and its results were compared with the IAEA - 433 certified material results also considered as probable effect concentration (PEC) (Table 4). Their values for the respective heavy metals followed the same trend on graph (Fig. 1) which ascertains the precision and accuracy of the method employed in this study. The spatial variations in the mean values of these heavy metals are presented in Table 4. The values of Fe in this study ranged between 11,371.6 to 45,095.9 ppm. The mean concentration of Fe were lower when compared with the reported values of 26,298 to 56966 mg/kg for sediment of Asejire Reservoir (Godwin *et al.*, 2015) but higher than the reported values of 475.2 to 704.2 mg/kg for sediments in Ala River (Abate *et al.*, 2013), 31.19 to 58.34 mg/kg for sediment of River Ngala (Akan *et al.*, 2011), 28.1 to 33.33 mg/kg for sediments of Orogodo River (Puyate

*et al.*, 2007). These changes may be attributed to differences in the nature of the bed rock materials (Godwin *et al.*, 2015). Fe has been reported to occur at high concentrations in Nigeria soil/sediments (Adefemi *et al.*, 2010; Godwin *et al.*, 2015). The ranges of other heavy

metals are from 2366.0 – 7626.4 ppm for Ti, 166.3 to 271.8 ppm for Cr, 78.6 to 1147.5 ppm for Mn, 3.8 to 9.2 ppm for Cu, 28.4 to 83.2 ppm for Zn, 0 to 85.6 ppm for Rb, 0 to 100.2 ppm for Sr, 171.6 to 911.5 ppm and 0 to 20.9 ppm for Pb.

Table 4: Comparison of measured concentrations of some heavy metals in sediments sample with the certified data in the standard reference materials (IAEA-433 sediment sample)

Heavy Metals mean (ppm)	Measured Standard (Control) Value (TEC) (ppm)	Certified Value (PEC) (ppm)
Ti	4907.70	4560.00 ± 34.66
Cr	216.20	69.30 ± 12.90
Mn	340.30	234.50 ± 14.63
Fe	21945.80	20080.20 ± 74.30
Cu	6.20	10.00 ± 2.50
Zn	52.40	48.90 ± 8.23
Rb	49.10	38.00 ± 10.47
Sr	52.30	68.00 ± 23.88
Zr	596.20	378.60 ± 62.39
Pb	14.70	11.70 ± 6.81

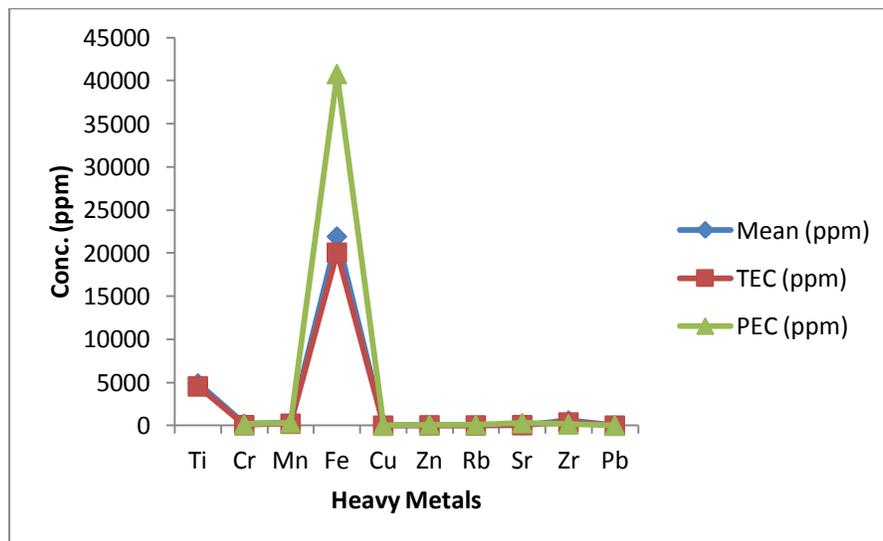


Fig.1: Comparison of PEC and TEC of heavy metals in Sediment Samples of Utibete River in Eastern Obolo, Southeastern Nigeria

Sediment just like water is usually employed as a pollution indicator by contaminants (Ogbeibu *et al.*, 2014). Using sediments heavy metals, a deeper insight into the long-term pollution state of the aquatic environment can be enumerated

(Yau and Gray, 2005). Since industrial revolution, tremendous amounts of the toxic pollutants have been discarded into coastal environment and sediment bays and estuaries have huge sinks of heavy metals (Fan *et al.*,

2002; Billon et al., 2002; Wang et al., 2010). The enrichment factor (EF) values (Table 3) of heavy metals in sediments of Utibete River in Eastern Obolo, Nigeria at sampling site 1 (UTR1) (Fig. 2) follows the sequential trend as  $Cr > (Zr = Pb) > Rb > Ti > Cu > Mn > Sr > Zn > Fe$ . Applying the interpretation above this part of the Utibete River is moderate severely enriched with Cr, moderately enriched with Zr and Pb and minorly enriched with other heavy metals. Thus, Cr poses serious pollution threat while Zr and Pb impose moderate contamination threat in part of the sediment of that River. The EF values at site 2 (UTR2) (Fig. 3) follow the trend in the descending orders as  $Cr > Pb > Zn > Fe > Cu > Zr > Ti > Mn$ . This part of the sediment is enriched moderately with Cr and minor enriched with Pb and Fe. There is no enrichment of Cu, Zr, Ti and Mn. Rb and Sr not detected. Thus, Cr might pose toxicity threat on this part of the river sediment. This might be due to those wastes with high content of Cr that got through to this part of the River sediments. The EF values of heavy metals at site 3 (UTR3) (Fig. 4) follow the descending orders as  $Cr > Rb > Zr > Zn > Fe > Mn > Ti > Cu > Sr$ . This part of the river sediment is moderately enriched with Cr and with minor enrichment of Rb, Zr, Zn, and, Fe. Pb is not detected while Cr is posing toxicity threat on that part of the ecosystem. For site 4 (UTR4) (Fig. 5), the EF values of the heavy metals follow the order as  $Mn > Cr > Fe > Zn > Ti > Rb > Pb > (Sr = Zr) > Cu$ . This part of the ecosystem has no pollution threat from any of the heavy metals present in the sediments here. The value of EF at sites 5 (UTR5) (Fig. 6) follow the order  $Cr > Rb > Zr > Sr > Zn > Ti > Fe > Pb > Mn > Cu$ . At this part of the sediment, there is no contamination threat from the detected heavy metals. At station 6 (UTR6) (Fig. 7), the Ef values of detected

heavy metals follow the sequential trends in the descending order as  $Cr > Zr > Pb > Sr > Ti > Zn > Fe > Mn > Rb > Cu$ . From the EF values, this part of the sediments is with minor enrichment of Cr, Zr, Pb, Ti and Zn. There is little pollution threat of Cr, Zr, Pb, Ti, and Zn on the ecosystem.

The values of the geo-accumulation index of heavy metals in Utibete River (Table 6) is as follow; at site 1 (UTR1) (Fig. 2), the trend is  $Cr > (Zr = Pb) > Rb > Ti > Cu > Mn > Sr > Zn > Fe$  with which their values ranges from -0.12 to 0.72. This part of the River is unpolluted to moderately pollute with the detected heavy metals. At site 2 (UTR2) (Fig. 3), the sequential trend is  $Cr > Pb > Zn > Fe > Cu > Zr > Ti > Mn > (-Rb = Sr)$ . Their values ranged from -0.35 to 0.68. This part of the sediment of the River is unpolluted to moderately pollute with heavy metals such as Mn to Cr. Rb and Sr are not detected at this part of this River sediment. At Station 3 (UTR3) (Fig. 4), the trend is  $Cr > Rb > Zr > Zn > Fe > Mn > Ti > Cu > Sr > Pb$  and their values ranged from -0.41 to 0.69. Thus, this part of the River sediment is unpolluted to moderately polluted with heavy metals such as Sr to Cr. Pb is not detected. At site 4 (UTR4) (Fig. 5), the sequential trend is  $Mn > Cr > Fe > Zn > Ti > Rb > Pb > (Sr = Zr) > Cu$  which their values ranges from -0.02 to 0.81. This part of the sediments shows unpolluted to moderately pollution of those heavy metals. For site 5 (UTR5) (Fig. 6), the heavy metals trend is in the descending order  $Cr > Rb > Zr > Sr > Zn > Ti > Fe > Pb > Mn > Cu$  with their values which ranged from 0.08 to 0.48 which implies that this part of the River sediments is unpolluted to moderately polluted with the detected heavy metals. For site 6 (UTR6), the trend of the heavy metals follow the descending order as;  $Cr > Zr > Pb > Sr > Ti > Zn > Fe > Mn > Rb > Cu$ . Their values ranged from

0.12 to 0.52. This shows unpolluted to moderately pollution of the detected heavy metals in Utibete River sediment.

On the basis of the mean values of CF, the sediments of Utibete at sampling site 1 (UTR1) (Fig. 2) is enriched with metals in the order Cr>(Zr=Pb)>Rb>Ti>Cu>Mn>Sr>Zn>Fe which implies that part of the sediment is highly

contaminated with Cr (3.92), moderately contaminated with Zr (1.78), Pb (1.78), and (1.21), while others metals are present at low contamination. Thus, Cr poses toxicity threat on that part of Utibete sediment. The values of the contaminations factor (CF) for heavy metals in sediments of Utibete at station 2 (UTR2) (Fig. 3) follow the decreasing trend as Cr> Pb> Zn> Fe> Cu> Zr> Ti> Mn> (Rb = Sr).

Table 5: Enrichment Factor (EF) of Heavy Metals in Utibete Sediments

Stations	Ti	Cr	Mn	Fe	Cu	Zn	Rb	Sr	Zr	Pb
1	1.85	6.93	1.25	1.00	1.62	1.03	2.13	1.15	3.15	3.15
2	0.52	3.60	0.41	1.00	0.75	1.17	ND	ND	0.55	2.75
3	0.84	3.32	0.98	1.00	0.45	1.01	1.89	0.27	1.76	ND
4	0.74	1.13	2.18	1.00	0.32	0.76	0.63	0.40	0.40	0.57
5	1.16	2.63	0.91	1.00	0.42	1.22	2.47	1.62	2.15	0.93
6	1.38	2.86	0.96	1.00	0.65	1.06	0.87	1.44	2.68	1.48

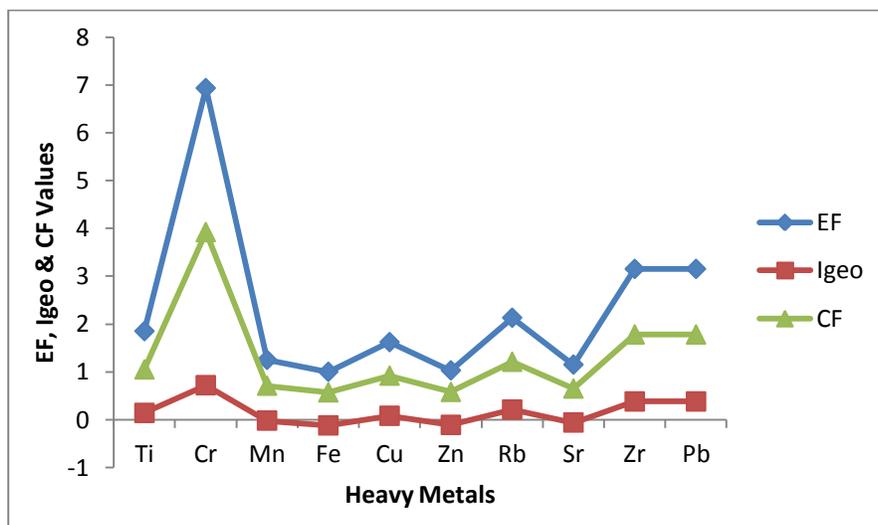


Fig. 2: Spatial variation of EF, Igeo and CF of UTR1

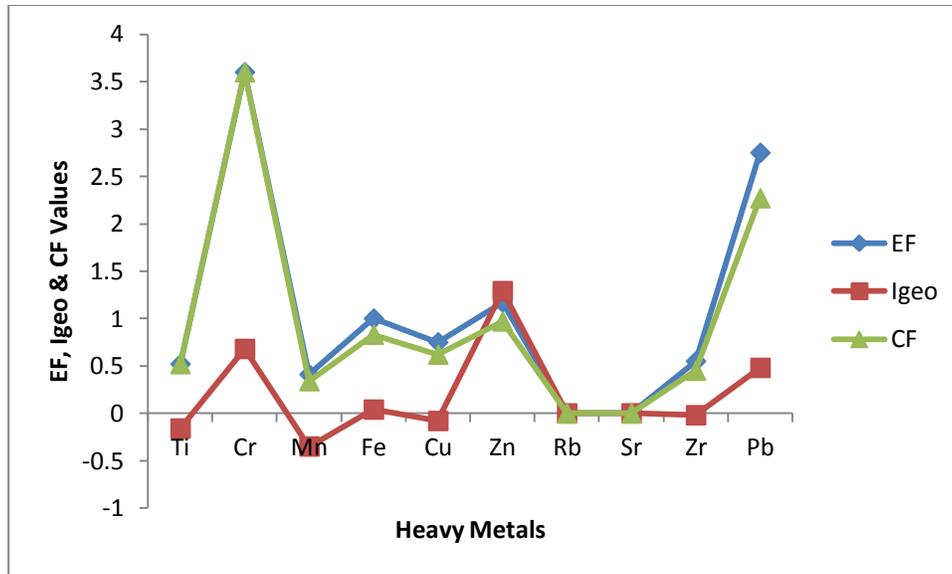


Fig. 3: Spatial variation of EF, Igeo and CF of UTR2

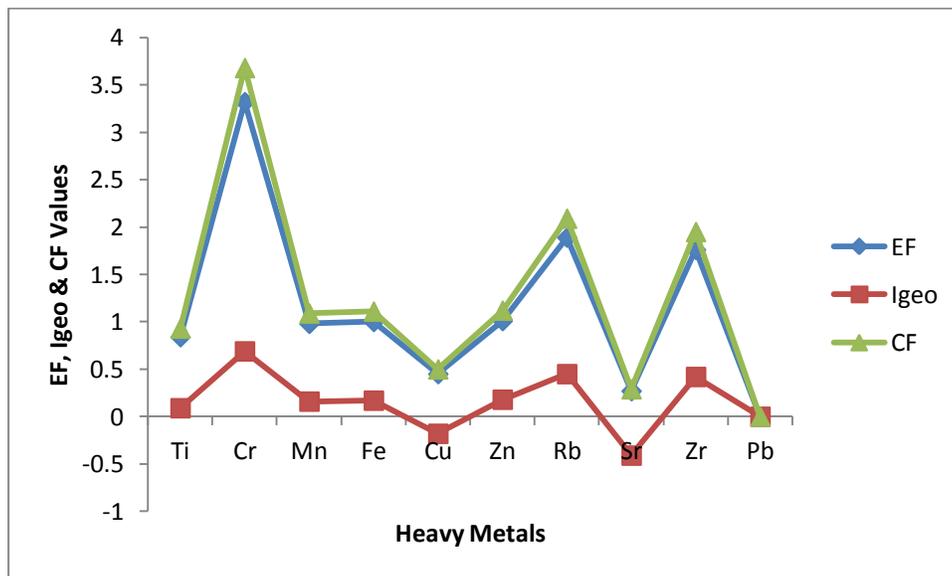


Fig. 4: Spatial variation of EF, Igeo and CF of UTR3

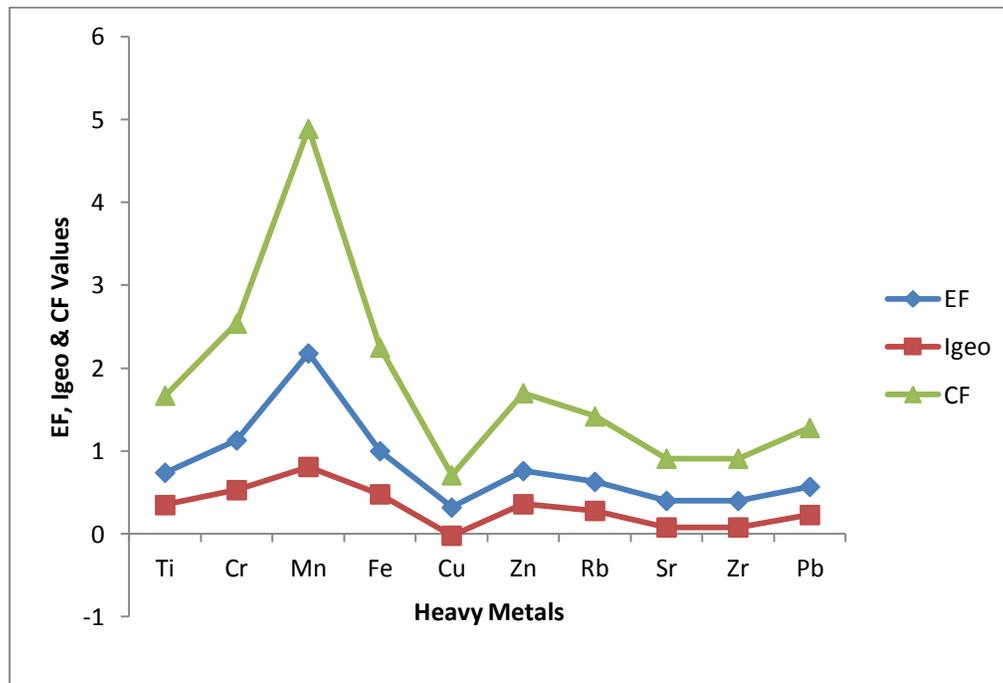


Fig.5: Spatial variation of EF, Igeo and CF of UTR4

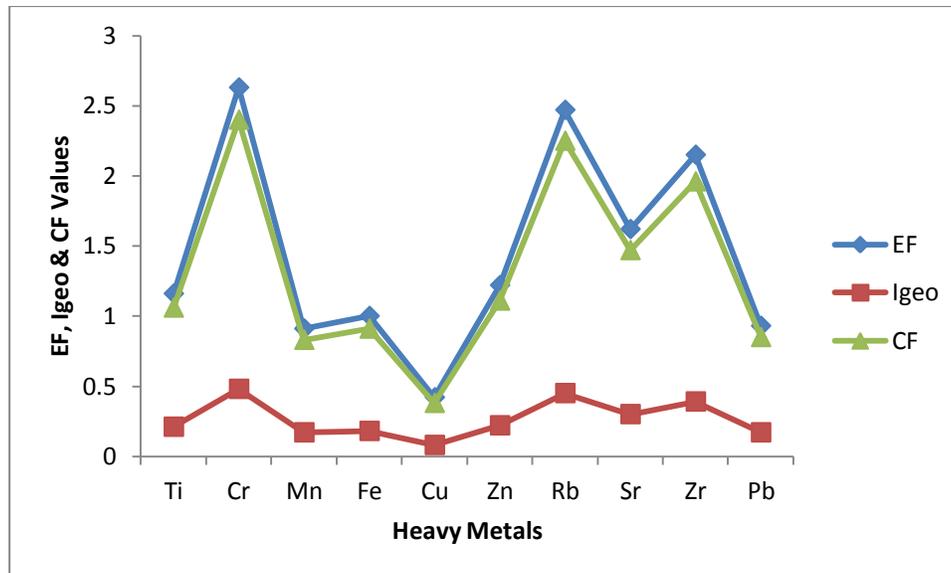


Fig.6: Spatial variation of EF, Igeo and CF of UTR5

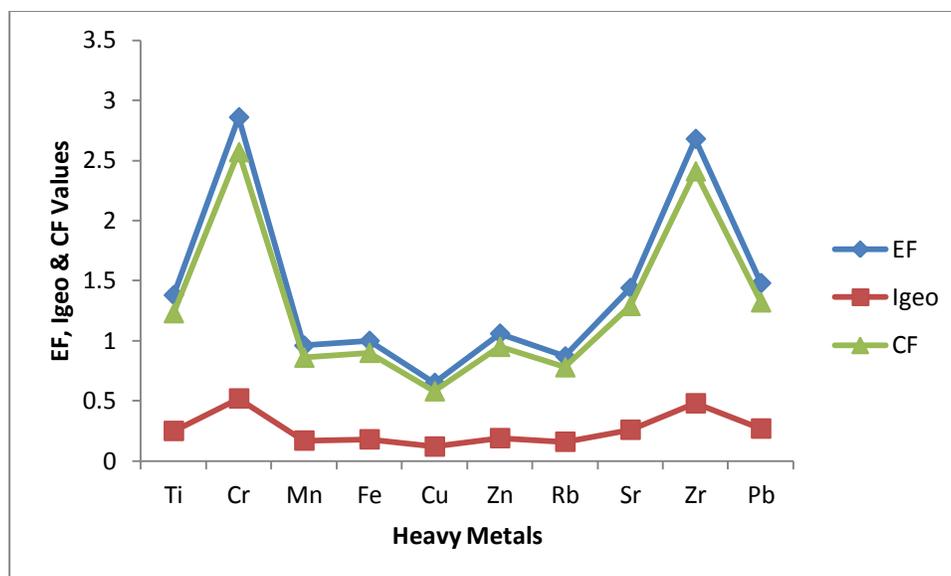


Fig. 7: Spatial variation of EF, Igeo and CF of UTR6

Table 6: Geo-accumulation Index (Igeo) of Heavy Metals in Utibete Sediments

Station	Ti	Cr	Mn	Fe	Cu	Zn	Rb	Sr	Zr	Pb
1	0.14	0.72	-0.02	-0.12	0.09	-0.11	0.21	-0.06	0.38	0.38
2	-0.16	0.68	-0.35	0.04	-0.08	1.29	ND	ND	-0.22	0.48
3	0.09	0.69	0.16	0.17	-0.18	0.18	0.45	-0.41	0.42	ND
4	0.35	0.53	0.81	0.48	-0.02	0.36	0.28	0.08	0.08	0.23
5	0.21	0.48	0.17	0.18	0.08	0.22	0.45	0.30	0.39	0.17
6	0.25	0.52	0.17	0.18	0.12	0.19	0.16	0.26	0.48	0.27

Thus, the sediment is highly contaminated with Cr (3.60), moderately contaminated with Pb (2.27) and of low contamination with other detected metals while Rb and Sr are not detected. This implies that Cr poses pollution threat on that part of the ecosystem. At site 3 (UTR3) (Fig. 4), the values of contamination factor (CF) of the heavy metals is of the sequential order Cr>Rb>Zr>Zn>Fe>Mn>Ti>Cu >Sr>Pb with which that part of the sediments is highly contaminated with Cr (3.68), moderately contaminated with Rb (2.09), Zr (1.95), Zn (1.12), Fe (1.11), Mn (1.09), lowly contaminated with Ti (0.93), Cu (0.50), Sr (0.29) while Pb is not detected. At station 4 (UTR4) (Fig. 5), the contamination factor (CF) values of heavy metals in this part of the River is of the trend

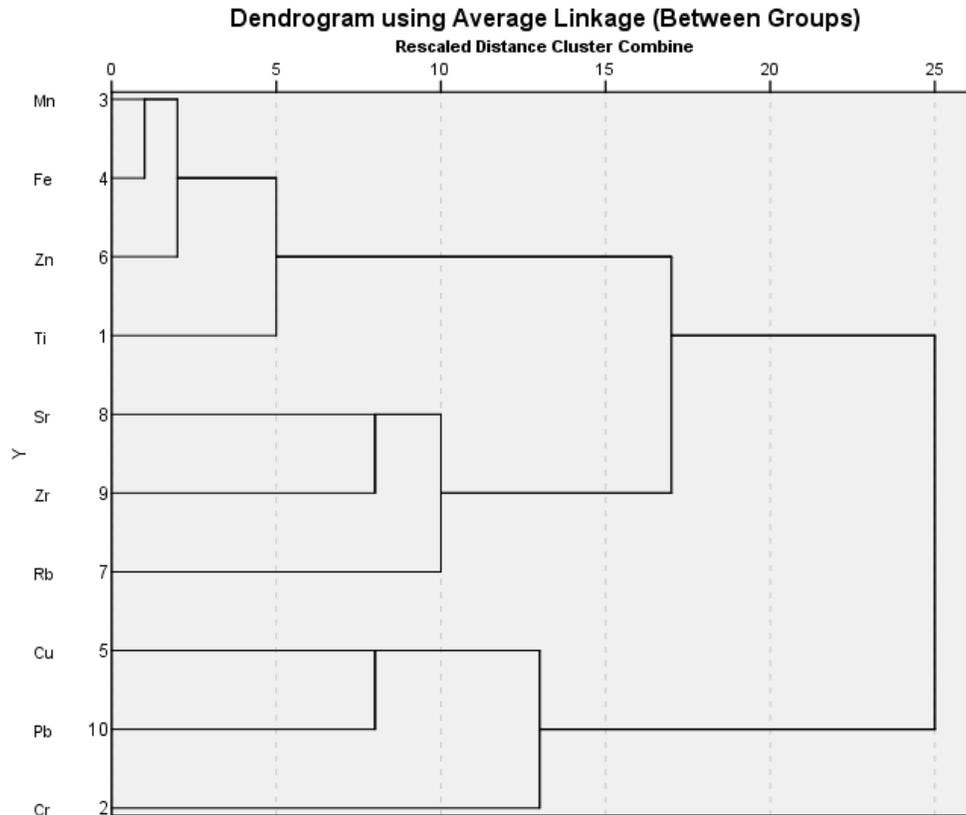
Mn>Cr>Fe>Zn>Ti>Rb>Pb> (Sr=Zr)>Cu with Mn (4.89) causing pollution on that part of the ecosystem. This part of the sediment is moderately contaminated with Cr (2.54), Fe (2.25), Zn (1.70), Ti (1.67), Rb (1.42) and Pb (1.28). These are due to influx of these heavy metals from anthropogenic activities. At station 5 (UTR5) (Fig. 6), the values of contamination factors (CF) of heavy metals in this part of that River is of sequential order Cr>Rb>Zr>Sr>Zn>Ti>Fe>Pb>Mn>Cu with which this part of the River is moderately contaminated with Cr (2.40), Rb (2.25), Zr (1.96), Sr (1.47), Zn (1.11), and Ti (1.06) which could be due to input from anthropogenic systems containing these heavy metals. At station 6 (UTR6) (Fig. 7), the values of the

contamination factor (CF) of heavy metals in this part of the River is sequentially arranged as Cr>Zr>Pb>Sr>Ti>Zn>Fe>Mn>Rb>Cu which shows that this part of the ecosystem is moderately contaminated with Cr (2.57), Zr (2.41), Pb (1.32), Sr (1.29), and Ti (1.23). The other heavy metals add no toxicity threat to that part of the ecosystem. Chromium enters into various environmental matrices (air, water, and soil) from a wide variety of natural and anthropogenic source with the largest release coming from industrial establishment (Tchounwou et al., 2012) which could results in high concentrations of Cr in almost all the sampling sites of Utibete River.

For station 1 (UTR1), the values of PLI is 1.08 thus that part of the ecosystem is contaminated with the respective heavy metals. At site 2 (UTR2), PLI values is 0.87 which implies that there is no contamination threat from the respective heavy metals in this part of the Utibete River. At station 3 (UTR3), PLI values is 1.12 thus this part of the River is contaminated with respective heavy metals. At site 4 (UTR4), the PLI value is 1.56 indicating toxicity threat of the respective heavy metals on this part of the ecosystem. At site 5 (UTR5), PLI values is 1.16. This implies that that part of the River is encountering pollution threat of the respective heavy metals. At station 6 (UTR6), PLI values is 1.16 which shows contamination threat of respective heavy metals.

The relationships and sources through which the heavy metals present in the investigated sediments of Utibete River were considered using Pearson's Correlation Coefficients (PCC) and Cluster Analysis (CA). The positive significant correlations between heavy metals in sediments of Utibete River are as follows; Ti and Mn ( $r = 0.829$ ), Mn and Fe ( $r = 0.968$ ), Mn and Zn ( $r = 0.860$ ), and Fe and Zn ( $r = 0.954$ ). These indicate the same sources of inputs. The negative significant correlations between elements in sediments of Utibete River are as follows; Cr and Sr ( $r = -0.824$ ), and Rb and Pb ( $r = -0.850$ ). These indicate different source inputs of the respective heavy metals. Other heavy metals not in the matrix indicate separate different sources for those heavy metals.

Hierarchical clustering results from heavy metals concentrations in sediment samples of Utibete River in Eastern Obolo, Akwa Ibom, Nigeria (Fig. 8) revealed 3 clusters: (1) Mn-Fe-Zn-Ti (2) Sr-Zr-Rb (3) Cu-Pb-Cr in terms of their similarities. This indicates that Mn, Fe, Zn, and Ti are from similar sources. Cluster 2 further shows that Sr, Zr and Rb originated from the same sources. Cluster 3 also shows that Cu, Pb and Cr are from similar sources. This agrees with the results obtained from Pearson's correlation coefficient.



**Fig. 8:** Hierarchical Clustering results (dendrogram) for heavy metals concentrations in sediment samples of Utibete River in Eastern Obolo, Akwa Ibom State, Nigeria

### Conclusion

In Nigeria, most domestic sewage and industrial effluents from both rural and urban areas are released into the environment without proper treatment. These wastewaters eventually find it way to lakes and reservoirs (Godwin et al., 2015). Thus, information in sediment quality condition is essential for evaluating the overall status of aquatic ecosystems. The mean concentrations of Fe in all the sampling sites are the highest indicating feldspar riverbed of Utibete River. This is also affirmed from the dendrogram results in which Fe is in one cluster along.

Three pollution indices (enrichment factor, geo-accumulation index, and contamination factor) applied to assess the pollution status of heavy

metals in sediments sample examined revealed similar sequential trends of heavy metals with respect to the sediments investigated. Thus, the three pollution indices agreed with one another on the contamination status of those heavy metals in all sediments samples studied indicating that the study area is highly contaminated with heavy metals such as Cr, and Mn, Rb, Zr, Pb and Ti at some parts of the River studied. From the pollution load index also applied in this study, every sampling sites of Utibete River is contaminated with the determined heavy metals with exception of Site 2 (UTR2) which poses toxicity threat to that part of the ecosystem. Such contamination threat might be from anthropogenic influences such as disposition of domestic effluents in that River,

runoff agricultural wastes and even patches of crude oil from oil spillage in the study area.

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