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Distribution of Heavy metals in sediments and surface waters from Iko River Marine Ecosystems, Akwa Ibom State, Niger Delta, Nigeria

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Abstract: *There have been intense crude oil and gas exploration and production activities within the Atlantic coastline of Iko River marine ecosystems, Akwa Ibom State, Nigeria, which has been on for decades, with new investments in the Oil Mining License Field 13 (OML 13) with consequent load of heavy metals. This study aims to determine the seasonal and spatial distribution of the levels of heavy metals in the sediments and surface waters of the Iko River marine ecosystem. The present study presents an opportunity to generate relevant data heavy metals distribution, pollution status, and their associated risks in these ecosystems. The heavy metals considered are cadmium (Cd), lead (Pb), zinc (Zn), vanadium (V), chromium (Cr), manganese (Mn), iron (Fe), cobalt (Co), nickel (Ni), and copper (Cu), will be determined by inductively coupled plasma (Agilent 720 ICP-OES) spectrometer, in sediments and surface waters from five (5) sampling locations (L1 – L5). The modified degree of contamination index, mC_d, in sediment for both dry and rainy seasons, indicated that L1 has the lowest contamination status (0.59 - 0.75), while, L5 has a high degree of contamination (3.76 – 4.07). Also, L5 indicated a very high contamination status for concerning Zn (7.84 – 9.01) in both seasons.. All the heavy metals of concern in this study have low potential ecological risk (maximum Ri = 20.44), in the sediments compared to the reference values ($E^i_f < 40$, and $Ri < 150$) at all the sampling sites in both seasons.*

Keywords *: Ecosystems; pollution load index; contamination factor; enrichment factor; accumulation index*

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1.0 Introduction

The Iko River marine ecosystems in Akwa Ibom State, Niger Delta, Nigeria, have long been subjected to extensive crude oil and gas exploration and production activities along the Atlantic coastline. Since the inception of oil and gas operations in the mid-20th century—oil exploration beginning in 1957 and production in 1973—the region has experienced continuous industrial expansion and rapid urbanization. More recently, renewed investments, particularly within the Oil Mining License Field 13 (OML 13), have escalated these activities through the drilling of new oil wells, construction of petrochemical facilities, and extensive infrastructural developments such as dredging, pipeline installations, and coastal vegetation clearing. Such developments have inevitably led to a heightened risk of accidental discharges, oil spills, gas flaring, and toxic waste dumping, all of which pose

significant threats to the fragile coastal marine environment.

Over the decades, a substantial body of literature has highlighted the environmental consequences of such anthropogenic activities in marine ecosystems worldwide. Numerous studies have documented the prevalence of organic and inorganic pollutants—most notably heavy metals and persistent organic pollutants (POPs)—in areas affected by oil and gas exploration (Enemugwem, 2009; Etesin et al., 2013; Akpan et al., 2019; Udoidiong, 2010; Nwaichi and Ntorgbo, 2016; Ubong et al., 2020; Yawo and Akpan, 2001). Specifically, the Iko River sediments have been characterized as predominantly medium to fine-grained sand with acidic properties, conditions which limit the sediments' capacity to act as a sink for these contaminants (Etesin et al., 2013). This characteristic implies that pollutants, once introduced, tend to remain in the water column for extended periods, thereby increasing their bioavailability and the risk of uptake by aquatic organisms.

Globally, heavy metal contamination is recognized as a severe environmental and public health issue. Heavy metals such as cadmium (Cd), lead (Pb), zinc (Zn), vanadium (V), chromium (Cr), manganese (Mn), iron (Fe), cobalt (Co), nickel (Ni), and copper (Cu) are of particular concern due to their non-degradable, persistent, and chemically reactive nature. While naturally occurring at low concentrations, these metals can become highly toxic when their levels exceed established thresholds. Their ability to accumulate in sediments, surface and pore waters, and biota tissues has raised alarms about the long-term impacts on biodiversity, human health, and ecosystem sustainability (Ansari et al., 2004; Barrera and Ariza, 2017; Naggar et al., 2018; Doležalová Weissmannová et al., 2019; Etesin et al., 2021). In fact, studies have linked heavy metal exposure to significant health risks, including cardiovascular diseases, neurotoxicity, and reproductive problems, with lead (Pb) exposure being implicated in numerous public health crises worldwide (WHO, 2010; Nnaji and Uzoekwe, 2016).

In the context of Nigeria, and particularly the Niger Delta region, the environmental impact of heavy metals has garnered significant research attention. Previous investigations have reported elevated levels of metals such as Cd, Pb, and Zn in various aquatic compartments, often exceeding World Health Organization (WHO) standards and posing considerable risks to both marine life and human populations who depend on these ecosystems for food and livelihood (Juhl, 2018; Igbemi et al., 2019; Tuo et al., 2019; Ubong et al., 2020). Despite these findings, most studies have focused on limited areas, frequently assessing sediment and water quality near the shoreline. There is a notable gap in comprehensive assessments that encompass the entire Iko River system, including its downstream channels and discharge points, which may offer a more representative understanding of the heavy metals distribution in the region.

The present study aims to address this knowledge gap by providing an in-depth analysis of the distribution of heavy metals in both sediments and surface waters from the Iko River marine ecosystems. By evaluating seasonal variations and spatial distribution patterns, this work seeks to generate critical data that can enhance public awareness and inform policymakers about the current pollution status and potential ecological risks associated with heavy metal contamination. The heavy metals of focus include Cd, Pb, Zn, V, Cr, Mn, Fe, Co, Ni, and Cu, all of which have been selected based on their environmental significance and previous reports of elevated concentrations in similar ecosystems.

In summary, this study builds on the existing literature by extending the geographical scope of analysis to provide a more holistic understanding of heavy metal dynamics in the

Iko River marine ecosystems. The findings are expected to contribute to the broader discourse on environmental pollution in petroleum-rich regions and support the development of targeted remediation strategies and sustainable management practices for the Niger Delta's coastal environments.

2.0 Materials and Methods

2. 1 The Study Area

The Iko River ecosystem is situated in the Eastern Obolo Local Government Area of Akwa Ibom State , which lies between latitudes $4^0$26' and $4^0$50' North and longitudes $7^0$30' and $7^0$55' East.

(Fig 1), within the equatorial region as well as the tropical mangrove belt (Etesin et al, 2013; Igbemi *et al*., 2019). As a Local Government Area in Akwa Ibom State, Nigeria, Eastern Obolo lies on the Atlantic coastline within the Gulf of Guinea and is bound to the North by Mkpat Enin, the South-East by Onna L.G.A, the West by Ikot Abasi LGA . and the south by the Atlantic Ocean (Enemugwem, 2009; Bassey *et al*, 2019; Igbemi *et al.,* 2019).

Eastern Obolo area has two main seasons; the wet or rainy (April - October), and the dry (November - March) seasons each year. It has an average temperature of 26 ^{0}C to 28 ^{0}C (Igbemi *et al*, 2019). Some slight variations in the climatic conditions, duration of the seasons, temperature and rainfall have been reported for the area due to the global climate change phenomenon (Bassey *et al*., 2019). The annual rainfall is

Iko River estuary empties into the Atlantic Ocean coastline at the Bight of Bonny (Enemugwem, 2009; Udoidiong, 2010; Etesin *et al.,* 2013; Harry *et al*, 2017). The estuary, tributaries and creeks feed and empty in a semi-diurnal tidal flow pattern into the Atlantic Ocean (Enemugwem, 2009; Etesin *et al*, 2013).

2.2. Sampling Locations

Five sampling sites were selected and marked out for the present study. (Table 1). The geographic coordinates of each of the sites were obtained with a GPS (Garmin GPS Map 785).

Table 1 : he sampling locations and their geographic coordinates.

		Coordinates	
Location No	**Name of Location and Sediment type**	**Latitude**	**Longitude**
L 1	Atlantic Ocean beach** (white sand)	$04^0$30.405'N	$07^0$43.663'E
L 2	Iko River (at Mmonta Beach) (mud)	$04^0$30.734'N	$07^0$45.199'E
L 3	Obolo River (mud) empty into Iko river	$04^0$32.227'N	$07^0$42.159'E
L 4	Amadaka river* (at Amadaka beach) (mud) empty into Iko river	$04^0$32.227'N	$07^0$42.159'E
L 5	Emeremen river (mud)	$04^0$31.154'N	$07^0$39.808'E

Amadaka and Emenenen rivers are located farther downstream of Iko River which feeds and empties them from, and into the Atlantic Ocean. **The sediment along L1 was ocean beach sand, but mangrove mud at all the other locations.

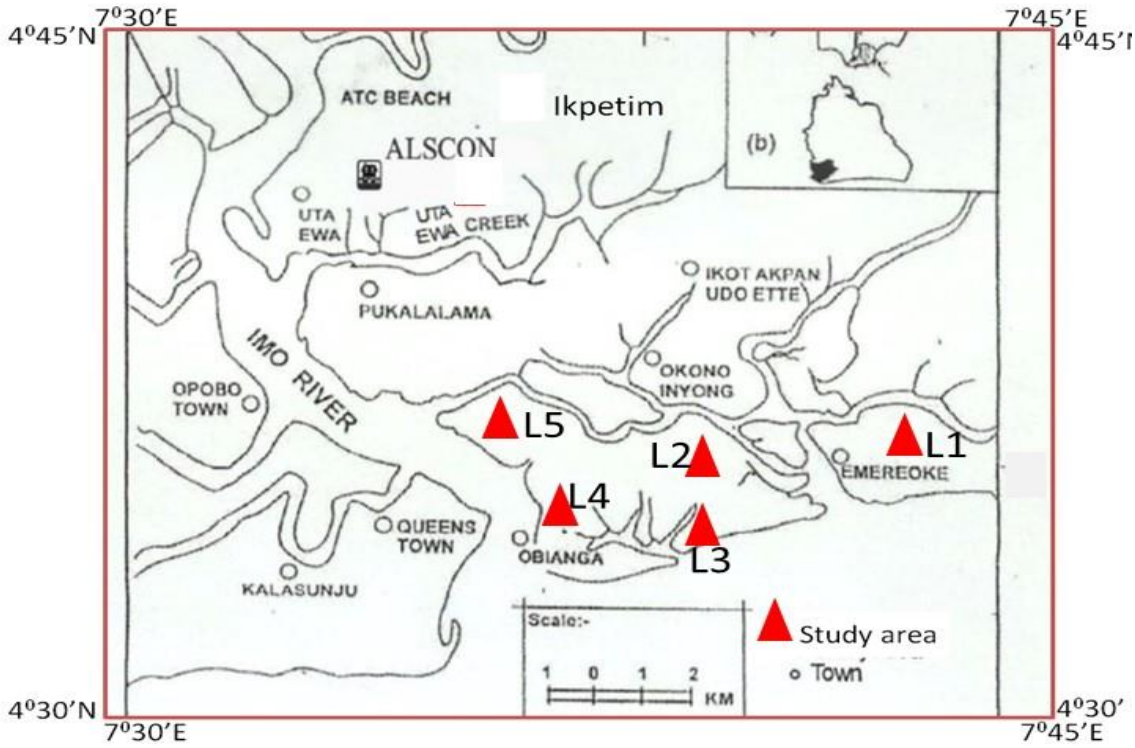

Fig .1 : Map of Study Area showing Sampling Locations

2.3 Sample Collection

Seasonal sampling was conducted at each study site in July (rainy season) and February (dry season) 2021, following the protocols outlined by Onjefu et al. (2016, 2020) and Tuo et al. (2019). At each geographic coordinate, three replicate samples of both sediments and overlying river waters were collected within a 10-meter radius (Bassey et al., 2017; Tuo et al., 2018). All sample collections were carried out during low tide, in accordance with US EPA (2002) recommendations.

2.4 Sediment Collection and Preparation

Triplicate sediment samples were obtained at each sampling site from the top 0–10 cm of the sediment profile using a Van Veen grab sampler. This depth range was selected based on studies indicating that heavy metal enrichment predominantly occurs within the surface 0–10 cm layer (Batley and Simpson, 2016; Benson et al., 2016; Simpson, 2016). Samples were collected from the intertidal, backwash, or littoral zones (Tuo et al., 2019). Immediately after collection, the sediment samples were transferred into black polyethylene bags that had been pre-washed and rinsed with 20% nitric acid to prevent metal adsorption on the bag walls. The samples were then stored in a sealed, ice-cooled container and transported to the laboratory for further analysis. In the laboratory, each sample was homogenized while still moist, and a portion was air-dried for over 48 hours. The dried sediments were ground using a porcelain pestle and mortar and sieved to remove particles larger than 2.0 mm. A 2.0 g aliquot of the sieved sediment was then digested to extract the metals: 25.0 cm^3 of freshly prepared aqua regia (a 3:1 mixture of concentrated hydrochloric acid to nitric acid, both of analytical grade from Merck) was added to each 2.0 g sample in a beaker. Anti-bumping beads were included in the digestion flask before heating the mixture nearly to dryness. After cooling, the digest was diluted with approximately 50.0 cm^3 of deionized water, filtered through Whatman No. 1 filter paper (0.45 µm pore size) into a pre-cleaned 100 cm^3 volumetric flask, and made up to volume with deionized water.

2.5 Water Sample Collection

[Text for water sample collection should be inserted here once available, ensuring that the methodology aligns with the procedures described above for consistency and clarity.]

The overlying river or ocean water was collected at each sampling site, along with the sediments. The water samples were collected with pre-rinsed and washed (with dilute nitric acid) plastic bottles below the surface at about 10-25 cm depth, labelled, and placed in an ice-cooled box, after adding some drops of analytical grade nitric acid to pH < 2. The acid was added to minimize loss of the heavy metals content through adsorption, sorption and/or precipitation on the walls of the bottles (APHA, 2005; US EPA, 2007, 2023; Benson *et al*, 2016; Simpson *et al*, 2016;).

2.6 ICP-OES Analyses of heavy metals

Inductively coupled plasma-optical emission spectrometer (Model: Agilent 720 ICP-OES) was used to analyze the total metal contents in each 100 cm^3 digest. The instrumental procedure used for the ICP-OES analysis is presented in detail in Appendix A.

2.7 The Geo accumulation Index, Contamination Factor and Enrichment Factor

Based on the index approach, the Geo-accumulation index (lgeo), Contamination factor (CF) and Enrichment factor (EF) were used to evaluate the sediment for their possible origin, health status and degree of enrichment of each metal at the respective sites.. The geo-accumulation index (lgeo), contamination factor and enrichment factor (EF) do not only predict the degree of contamination and health status of sediment and soil, but are also tools for identifying the natural or anthropogenic input of metals or a combination of both, and also give a better understanding of the distribution and enrichment of metals in the ecosystem (Pandey *et al.*, 2015; Abdullah *et al*., 2020).

The accumulation index (lgeo) determination for each metal was based on the equation developed by Muller which is widely used by researchers (Benson *et al*., 2016; Abdullah *et al*., 2021):

$$I_{geo} = \log 2 \frac{Cn}{1.5Bn} \quad (4)$$

where log2 = 0.3010, C_n is the measured concentration of the metal/element in the sediment or soil sample, and B_n represents the standard reference background level for the metal (generally considered as the pristine, pre-industrial or uncontaminated level or concentration of the metal).

The geo-accumulation index classifies sediments into seven (7) grades as originally proposed by Muller for the assessment of sediment or soil quality. These are:

i. Class 0 – lgeo value < 0 – implying that the sample is practically unpolluted;
ii. Class 1 – lgeo value > 0 - 1 rated as unpolluted to moderately polluted;
iii. Class 2 – lgeo > 1 – 2, moderately contaminated/polluted;
iv. Class 3 – lgeo > 2 – 3, moderately to slightly polluted;
v. Class 4 – lgeo > 3 – 4, moderately to strongly polluted;
vi. Class 5 – lgeo > 4 – 5, strongly to extremely polluted; and
vii. Class 6 – lgeo > 5, very strongly to extremely polluted (Abrahim and Parker, 2018)

2.8 The Contamination Factor (CF)

The contamination factor (CF) is an index method for determining the degree of contamination by contaminant like heavy metals relative to the average continental or crustal background composition of the contaminants concerned.

The equation for contamination factor (CF) is:

$$C_{fi=\frac{Ci}{Cni}} \quad (5)$$

Where Ci is the measured concentration of a given heavy metal in a sediment sample, and Cni is the standard pre-industrial

(uncontaminated) reference level of the metal in mg/kg.

The contamination factor categorizes sediment quality into four classes namely:

$C_f < 1$ – indicates low contamination status:
$1 < C_f < 3$ – moderate contamination status;
$3 < C_f < 5$ – considerable contamination status; and
$6 > C_f$ - very high contamination status, (Benson *et al*., 2016; Onjefu *et al.,* 2016).

The world average background values for shale reported by Turekian and Wedepohl (Saha *et al.,* 2022) were used (Appendix B) .

2. 9 The Pollution Load Index (PLI)

The pollution load index (PLI) provides an assessment of the overall integrity of the study site (which is where the intertidal coastal ecosystems in Iko River estuary) for heavy metals (contaminants) measured in the sediment samples (Benson *et al*., 2016). The PLI is useful for determining the degree of anthropogenic heavy metals or contaminations accumulated in aquatic sediments. It also gives a clear indication of the toxicity status of any specific sediment component of the environment or ecosystem. This is possible as it indicates the number of times the heavy metal level could have exceeded the average background concentration (Benson *et al.*,2020).

The PLI is derived from the contamination factor (CF) and is given by the equation

$$PL1 = \sqrt[n]{C(F1 \times CF2 \times CF3)..CFn}$$

where CF is the contamination factor of a metal, i, and n is the number of metals or contaminants measured at a site (Benson *et al*., 2016; Aljahdali and Alhassan, 2020). The PLI classifies sediments environmental pollution status as follows:

Class 1: < 0 – 0, no pollution,
Class 2: 0 – 1, low degree of pollution,
Class 3: 1 – 2, moderate degree of pollution,
Class 4: 2 – 4 high degree of pollution;
Class 5: 4 – 8 very high degree of pollution; and
Class 6: 6 – 18, extremely high degree of pollution.

2.10 Data analysis

Statistical analysis on whole sediment concentrations of the heavy metals was performed using Minitab 17 statistical software. A two-tailed t-test ($p \leq 0.05$) was used to evaluate the significance of variability between the dry and rainy seasons' heavy metals data sets obtained from the five study locations as described by APHA (2016) and Tuo (2019).

All data obtained in the study were also subjected to analysis of variance (ANOVA) for spatial and seasonal variations, with the level of significance set at $P \leq 0.05$. The statistical analysis of the data was performed using SPSS v.25 software, including mean, standard deviation and other calculations for the data acquired.

3. Results and Discussion

3.1 Results of Heavy Metals in Sediments and Surface Waters

The concentrations of heavy metal ions in the sediment and water from Iko River marine rae presented in Tables 2 and `1 respectively.

The results reveal marked spatial and seasonal variations in heavy metal concentrations between sediments and surface waters within the Eastern Obolo ecosystem. In the sediments, vanadium concentrations ranged widely, from as low as 6.54 mg/kg during the rainy season at L1 to as high as 469.56 mg/kg during the rainy season at L5. Although vanadium was below detection limits (BDL) at several sites, notably at L2 and L4, the extremely high levels recorded at L3 and L5—especially during the rainy season—suggest localized inputs or accumulation processes. It appears that increased surface runoff during the rainy season enhances the mobilization and deposition of vanadium, leading to values that exceed the natural shale standard.

Chromium showed similar spatial variability, with detectable levels at L1 and L2 ranging between 2.15 and 26.79 mg/kg, while remaining BDL at other sites during the dry season. However, during the rainy season, chromium at L5 surged to 163.89 mg/kg, considerably surpassing both the shale reference value and the World Health Organization (WHO) guideline. This variability indicates that chromium may be mobilized episodically, likely due to enhanced leaching or runoff from contaminated areas during heavy rainfall.

Manganese concentrations in sediments were notably high, with particularly extreme enrichment at L5 where levels reached over 21,000 mg/kg during the dry season, far exceeding both the shale reference value and WHO guidelines. Such extraordinary levels suggest either significant anthropogenic influence or geochemical conditions that promote manganese accumulation. The presence of high manganese concentrations in sediments is concerning, as it may adversely affect benthic organisms and alter sediment redox dynamics.

Iron concentrations were consistently elevated across all sites and seasons, with the highest recorded level of nearly 155,000 mg/kg at L3 during the rainy season. These values are well above the natural shale background and WHO standards, reflecting both the natural geochemical makeup of the area and additional industrial inputs. High iron content in the sediments can also influence the sorption and mobility of other heavy metals, thereby affecting their bioavailability.

In contrast, cobalt was below detection limits at all sites, and nickel was detected only at L1 and L2, with levels well below the shale standard. This limited detection indicates that cobalt and nickel are not major concerns in this ecosystem or may originate from more localized sources. Copper was found only at L1 and L2, with values that generally remained below both shale and WHO standards, whereas zinc exhibited elevated concentrations, especially at L3 and L5 during both seasons. At L5, zinc concentrations ranged from approximately 745 to 856 mg/kg, significantly exceeding both the shale standard and WHO guidelines, which raises potential concerns for benthic organisms and suggests persistent industrial inputs. Cadmium was consistently below detection limits in the sediments, indicating that its levels are within acceptable limits and that it does not represent a primary pollutant in this area.

In the water samples, heavy metal concentrations were generally much lower than in the sediments, indicating that the water column acts more as a transient pathway for contaminants rather than a repository. Vanadium was detected in water at L1 and L2 at concentrations of about 5.65 to 6.45 mg/L, but it was below detection limits at other sites. Chromium was not detected in any water samples, suggesting that it is rapidly removed from the water column by adsorption onto particulates or is not mobilized under the prevailing conditions. Manganese and iron were present in the water at low levels compared to their sediment counterparts, implying that most of these metals are retained in the sediments, which act as sinks. Similarly, copper, zinc, and lead were present in trace amounts in the water, further supporting the observation that these metals preferentially partition into the sediment.

Comparing the two matrices, sediments clearly accumulate significantly higher concentrations of heavy metals than water, which is consistent with the role of sediments as sinks for such contaminants. The seasonal variations observed—with many metals showing enhanced concentrations during the rainy season—suggest that increased runoff and leaching contribute to the episodic loading of metals into the sediments. Although the water samples generally exhibited lower concentrations due to dilution and rapid sedimentation, even trace levels may be of

concern over time, particularly for sensitive aquatic species and communities relying on these water resources.

Overall, the elevated levels of heavy metals such as vanadium, chromium, manganese, iron, and zinc in the sediments—often exceeding both natural background (shale) values and WHO guidelines—highlight the significant potential for ecological and human health risks. These findings emphasize the need for targeted monitoring and remediation efforts, particularly at hotspots such as L3 and L5, where contamination is most pronounced. The observed seasonal differences further underline the importance of continuous monitoring programs to capture temporal fluctuations and inform effective environmental management strategies in this industrially influenced region.

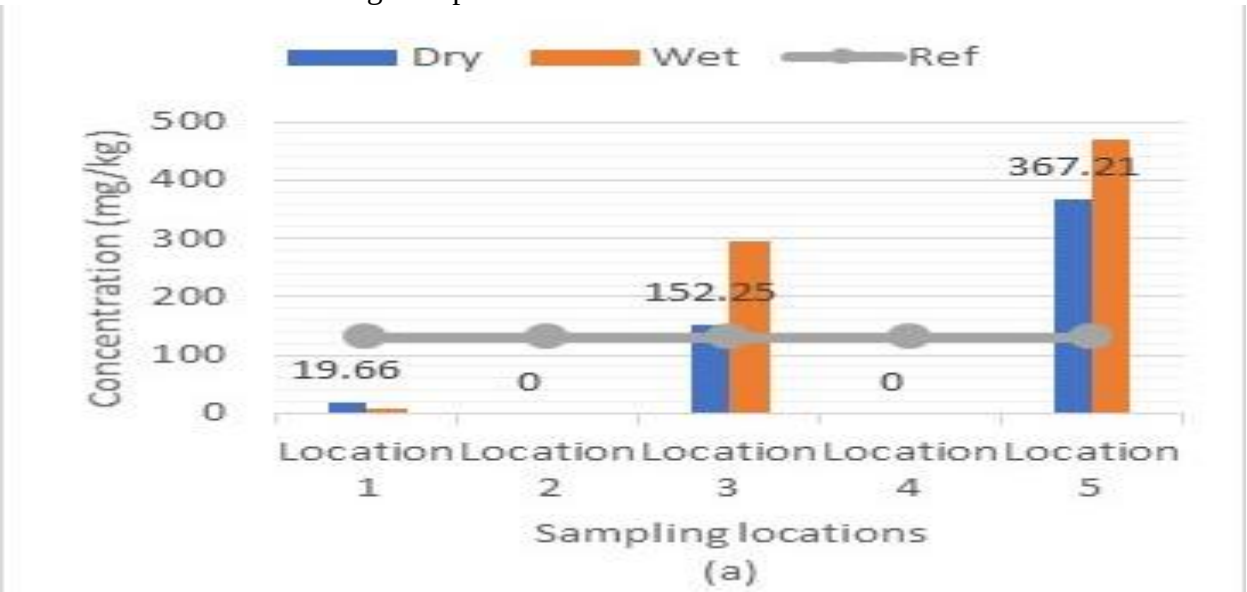

Fig. 2 :Vanadium concentration in sediments

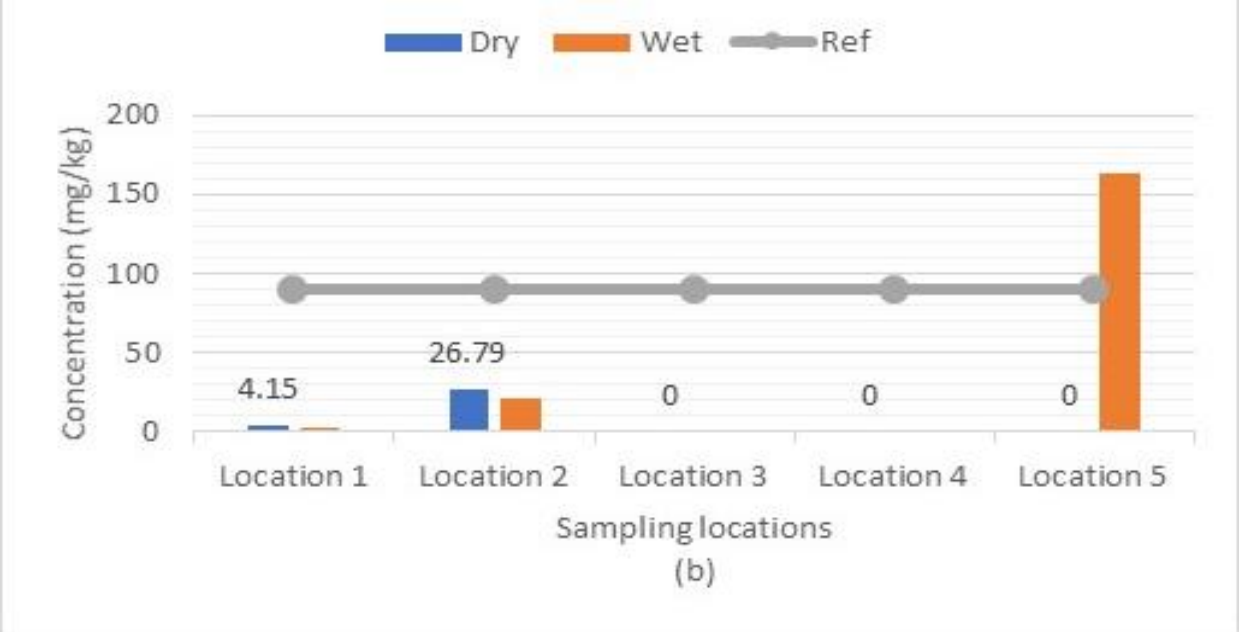

Fig. 3: Chromium concentration in sediments

Table 2 : Results of heavy metal concentrations in sediments from Eastern Obolo ecosystem during dry (D) and rainy (R) seasons

M	L1 (D)	L1 (R)	L2 (D)	L2 (R)	L3 (D)	L3 (R)	L4 (D)	L4 (R)	L5 (D)	L5 (R)	**Shale ***	WHO
	Value	**Value**	**Value**	**Value**	**Value**	**Value**	**Value**	**Value**	**Value**	**Value**		
V	19.66 ±1.00	6.54 ±0.49	BDL	BDL	152.25 ±0.00	296.01 ±0.00	BDL	BDL	367.21 ±0.00	469.56	130.00	
Cr	4.15 ±0.50	2.15 ±0.99	26.79 ±2.00	20.91 ±0.50	BDL	BDL	BDL	BDL	BDL	163.89 ±0.00	90.00	100.00
Mn	228.81 ±0.99	121.81 ±0.99	134.63 ±1.00	178.39 ±1.00	1580.55 ±0.00	2621.97 ±4.93	286.99 ±50.00	93.38 ±0.00	21151.84 ±0.00	1120.84 ±0.00	850.000	20.0
Fe	9509.59 ±76.50	6421.75 ±17.24	13458.24 ±106.50	12845.53 ±159.50	115100.20 ±725.00	154870.39 ±1517.34	36879.90 ±64.04	16215.12 ±14.72	146406.47 ±651.74	144003.45 ±256.16	47200.00	5000.00
Co	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	19.00	___
Ni	4.48 ±0.50	2.38 ±0.49	15.60 ±1.00	12.57 ±0.50	BDL	BDL	BDL	BDL	BDL	BDL	78.00	___
Cu	1.15 ±1.00	0.68 ±0.98	17.69 ±1.00	13.11 ±1.00	BDL	BDL	BDL	BDL	BDL	BDL	45.00	100.00
Zn	28.43 ±0.50	20.56 ±0.00	61.40 ±0.50	58.15 ±0.00	310.30 ±0.00	513.10 ±0.00	258.13 ±0.00	170.38 ±0.00	745.07 ±0.00	856.06 ±0.00	95.00	300.00
Cd	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	0.30	3.00

****M = heavy metal, W = WHO standard, Turekian and Wedepohl (1961) * ; Onjefu *et al.* (2016).**; BDL=Below detection limit.**

Table 3: Results of heavy metal concentrations in water samples from Eastern Obolo ecosystem during dry (D) and rainy (R) seasons

Heavy metals	L1		L2		L3		L4		L5	
	D	**R**	**D**	**R**	**D**	**R**	**D**	**R**	**D**	**R**
V	6.36±0.08	5.93±0.10	6.45±0.02	5.65±0.02	BDL	BDL	BDL	BDL	BDL	BDL
Cr	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Mn	0.64±0.00	0.06±0.00	0.08±0.00	BDL	0.04±0.00	0.45±0.00	0.71±0.00	0.39±0.00	0.95±0.00	0.97±0.00
Fe	34.80±0.15	53.76±0.02	6.34±0.04	1.18±0.02	6.91±0.00	6.78±0.00	6.78±0.00	3.43±0.00	6.45±0.00	7.81±0.00
Co	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Ni	0.03±0.03	0.02±0.03	0.02±0.03	0.02±0.02	BDL	BDL	BDL	BDL	BDL	BDL
Cu	0.71±0.04	0.68±0.02	0.70±0.03	0.68±0.03	BDL	BDL	BDL	BDL	BDL	BDL
Ag	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Zn	0.10±0.00	0.05±0.00	0.09±0.00	0.07±0.00	0.78±0.00	1.21±0.00	0.96±0.00	BDL	2.48±0.00	0.68±0.00
Cd	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Pb	0.01±0.03	0.02±0.06	0.04±0.02	0.03±0.04	BDL	BDL	BDL	BDL	BDL	BDL

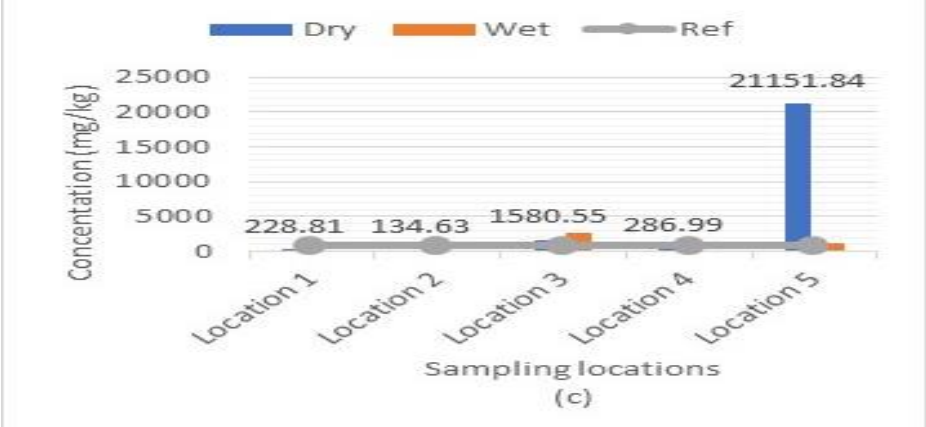

Fig. 4 : Manganese concentration in sediments

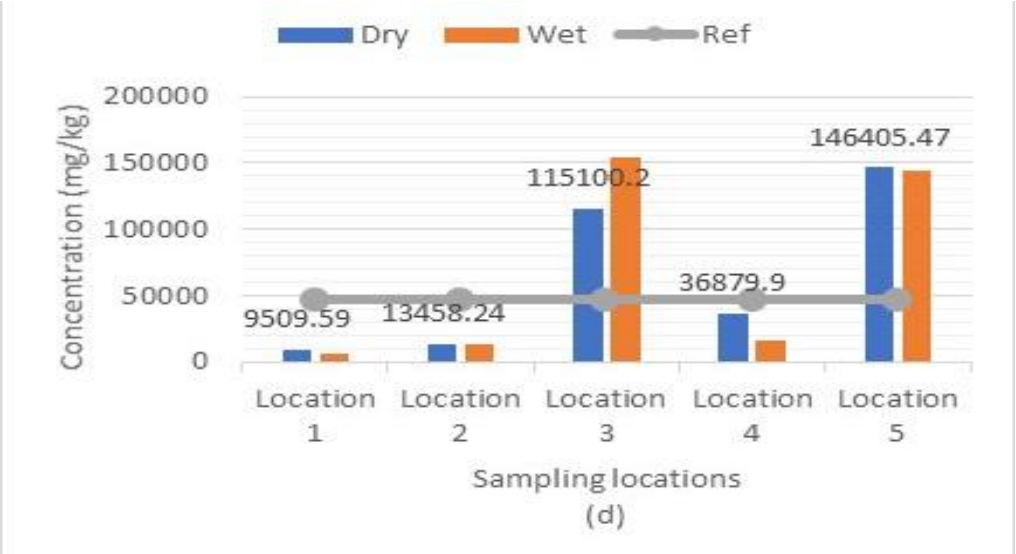

Fig. 5 :Iron concentration in sediment

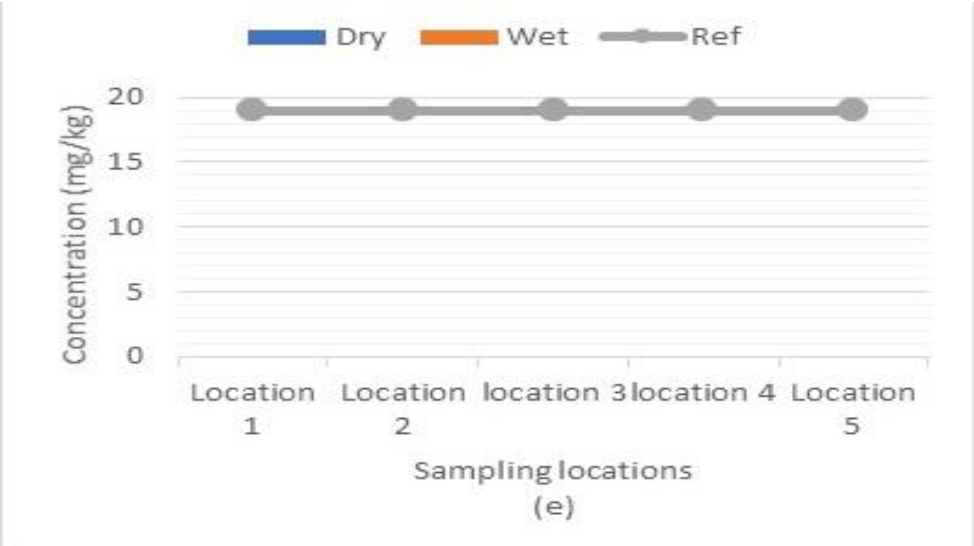

Fig.6 : Cobalt concentration in sediments

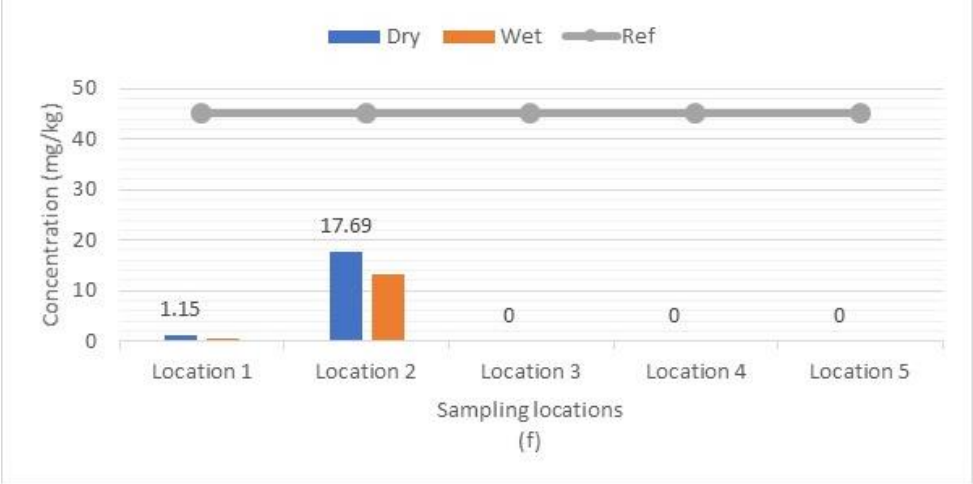

Fig.7 : Copper concentration in sediments

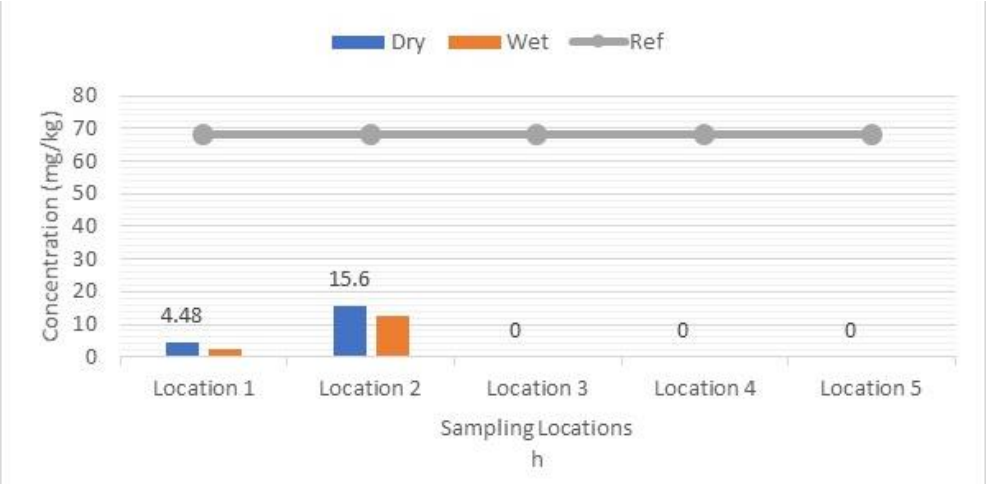

Fig. 8 : Nickel concentration in sediments

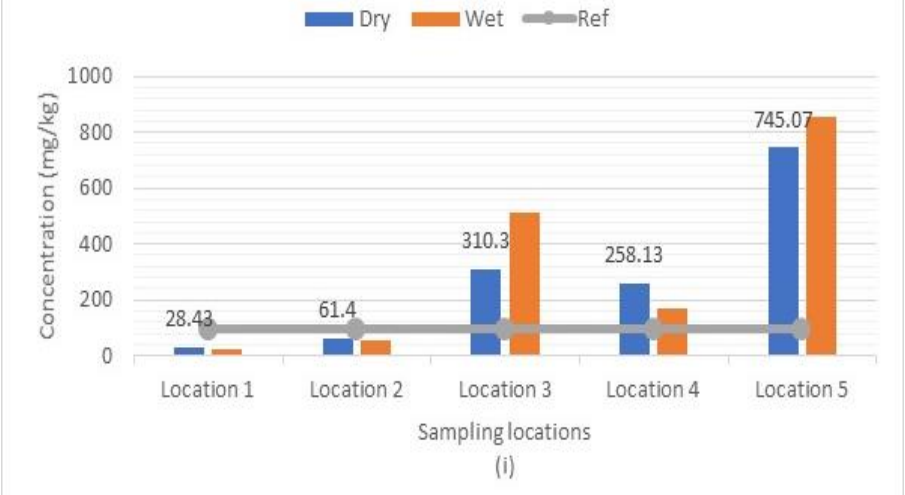

Fig. 9 : Zinc concentrations in sediments

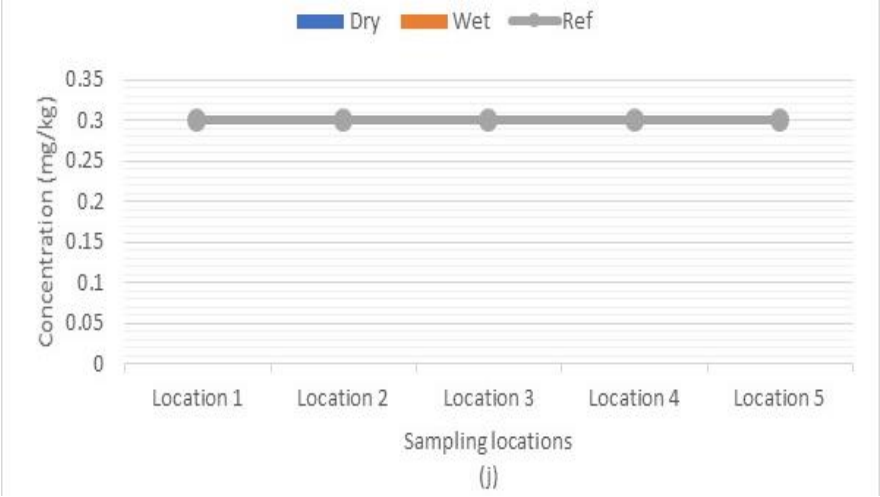

Fig. 10 : Cadmium concentrations in sediments

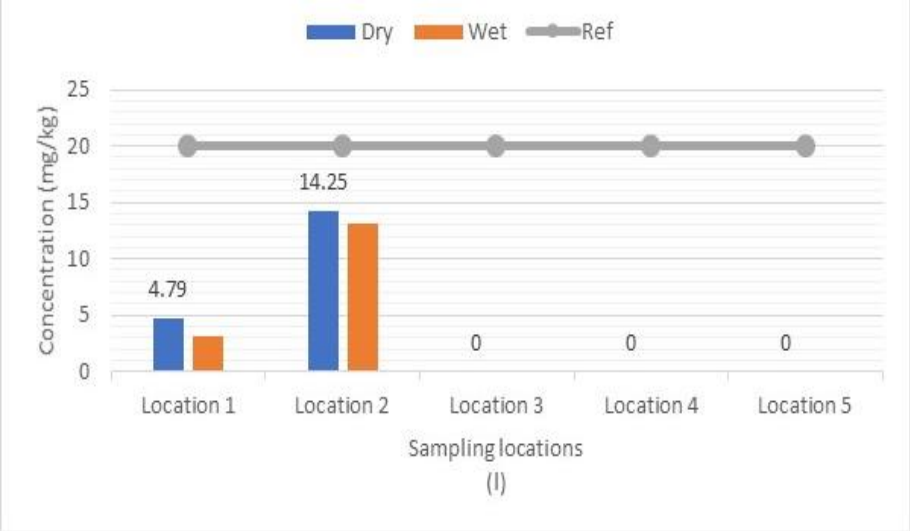

Fig. 11: Lead concentrations in sediments

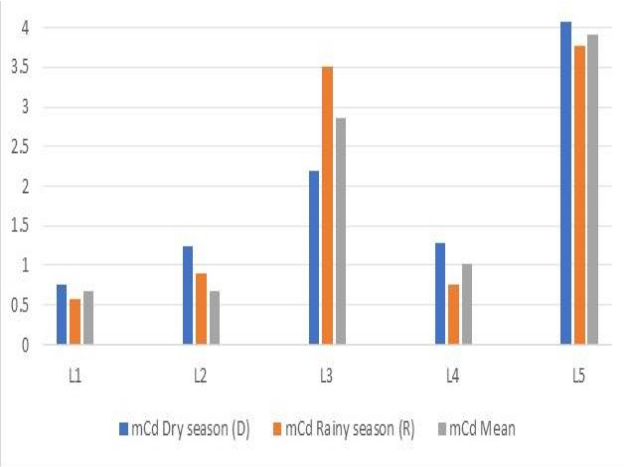

Fig 12: The modified degree of contamination index, mCd, for the sampling sites in sediments

3.2 The Modified Degree of Contamination Index

The modified degree of contamination index, mC_d, in sediment, offers an empirical and generalized form of estimating the overall level of contamination at a given sampling site.

The modified degree of contamination index, mC_d, in sediment is presented in Fig. 12 for both dry and rainy seasons. The mC_d results indicated that L1 has the lowest contamination status (0.59 - 0.75). Again, the results indicated that L5 has ranked above the others, and has a high degree of contamination (3.76 – 4.07). Locations L2, L3 and L4 have low to

moderate degrees of contamination (0.75 – 3.50). Fewer metals were detected at L5. However, it has a high degree of contamination concerning PLI and mC_d due to the high contribution from Zn concentration measured at location 5 (L5) .

3.3 Enrichment Factor (EF)

The Enrichment factor (EF) is presented in Table 4, with an indication of the highest value at L5,due to anthropogenic sources. The other metals have no to low enrichment status at L1. At L2 the metals V, Mn and Ni have EF indicating natural origin for them in that environment. The EF values close to 1.0 indicates a natural (lithogenic) origin for the element being assessed, less than 1.0 indicates a probable mobilization or reduction of the element, and values above 1.0 probably indicate that the element has anthropogenic origin in the environment. (Pandey *e al.*, 2015; Benson *et al*., 2016; Aljahdali and Alhassan, 2020).

Table 4: The enrichment factor (EF) for the heavy metals in sediments

Metals	EF				
	L1	L2	L3	L4	L5
V	0.60	0.00	0.60	0.00	1.05
Cr	0.21	0.95	0.00	0.00	0.59
Mn	1.22	0.66	0.84	0.40	0.63
Fe	1.00	1.00	1.00	1.00	1.00
Co	0.00	0.00	0.00	0.00	0.00
Ni	2.38	0.74	0.00	0.00	0.00
Cu	1.23	1.23	0.00	0.00	0.00
Ag	0.00	0.00	0.00	0.00	0.00
Zn	1.53	2.26	1.52	4.00	2.74
Pb	1.16	2.46	0.00	0.00	0.00

At locations L3, L4 and L5 the EF indicated no enrichment from anthropogenic sources, implying enrichment from natural sources for the metals V, Cr, Mn, Co, Ni, Cu, Ag, Pb, . At these sites, Zn (1.52 - 4.00) has enrichment which suggests probable anthropogenic sources of enrichment or origin.

The EF results (Fig.13) showed that most of the metals being studied have natural origin along the coastline of Eastern Obolo. The probable anthropogenic sources of enrichment for the heavy metals may include petroleum products and related activities, agricultural and domestic wastes due to rapid urbanization, as well as products of corrosion of corrugated iron roofing sheets, oil pipelines, etc due to acid rain as a result of gas flaring (Attah, 2012; Benson *et al*., 2016; Fortoul *et al.*, 2020).

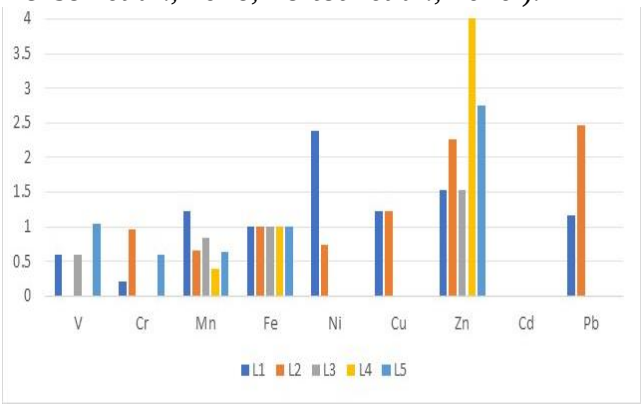

Fig 13: Enrichment factor (EF) for the heavy metals at the study sites in sediments

3.4 The Contamination Factor (CF)

The contamination factors determined in this study are represented in Table 5, for all the study locations (L1 - L5). At L3 and L4 the contamination status varied between low to considerable concerning all the metals. However, L5 indicated a very high contamination status concerning Zn (7.84 – 9.01) in both seasons).

Generally, the CF results show that L1 was the least contaminated despite the numerous metals present there. Also, L5 was found to have the highest contamination status. The highest contamination status of L5 can be accounted for by crude oil spills and wastes from the watershed of an abandoned artisanal crude oil refining site which drains directly into L5 through surface runoff (Etesin *et al.*, 2013; Udo *et al*., 2020).

Table 5: nnnnnnnnnnnnnnnn

Metal	L1 (D)	L1 (R)	L2 (D)	L2 (R)	L3 (D)	L3 (R)	L4 (D)	L4 (R)	L5 (D)	L5 (R)
V	0.15	0.05	–	0.30	1.17	2.28	–	–	2.82	3.61
Cr	0.05	0.05	–	0.23	–	–	–	–	–	1.82
Mn	0.27	0.14	0.16	0.21	1.86	3.08	0.34	0.11	2.53	1.32
Fe	0.20	0.14	0.29	0.27	2.44	3.28	0.78	0.34	3.10	3.05
Co	–	–	–	–	–	–	–	–	–	–
Ni	0.07	0.04	0.23	0.18	–	–	–	–	–	–
Cu	0.03	0.01	0.39	0.29	–	–	–	–	–	–
Ag	–	–	–	–	–	–	–	–	–	–
Zn	0.30	0.22	0.65	0.61	3.27	5.40	2.72	1.79	7.84	9.01
Cd	–	–	–	–	–	–	–	–	–	–
Pb	0.24	0.15	0.71	0.66	–	–	–	–	–	–

3.5 The Pollution Load Index (PLI)

Table 6 presents the Pollution load index (PLI) results for L1- L5 locations in the study area. The results obtained (Fig.14) indicates a low to high contamination status of the sampling sites. Similar to the lgeo index and CF, the PLI shows that L1 (the Atlantic coastline/sand beach) is the least polluted. It also showed L5 (Emeremen River) as the highest polluted site with a high degree of pollution (Aljahdali and Alhassan, 2020). s earlier inferred, L5 was directly contaminated by the abandoned artisanal crude oil refining site located in its catchment watershed. The beach sand at L1 has larger grain sizes, low organic matter content and metal binding sites, thus having a lower retention capacity for heavy metals (Juhl, 2018; Simpson and Kumar, 2016; US EPA, 2007, 2023).

Table 6.: The pollution load index of the study sites, L1 – L5

Location/Site	Dry Season	Rainy Season	Average (PLI)
L1	0.23	0.13	0.18
L2	1.24	1.19	1.22
L3	2.04	3.34	2.69
L4	0.90	0.41	0.66
L5	3.75	3.93	3.84

3. 6 The Geoaccumulation Index (lgeo)

The lgeo index estimated from the present study is represented in Table 7 and indicated that sampling sites L1, L2, L3, L4, and L5 ranged from class 0, 1 and 2 (uncontaminated to moderately contaminated by metals) during the dry and rainy seasons. The highest lgeo index (1.57 – 1.81) was recorded for Zn at L5 (Emeremen river sediments). Uncontaminated lgeo index (Class 0) was recorded for all the metals, except Zn, at L3, L4, and L5 for both seasons (Fig. 14).

The moderate contamination status of L5 may have been greatly influenced by contaminated

surface runoff from the watershed of abandoned illegal artisanal crude oil refining sites of over 10 years which drains directly into L5.

3. 7 Potential ecological risk

The potential ecological risk of heavy metals in sediments of Iko River marine ecosystem is presented in Table 8 . All the heavy metals of concern in this study have low potential ecological risk in the sediments ($E^i_f < 40$, and Ri < 150) at all the sampling sites in both seasons (maximum Ri = 20.44 at L5), as presented in Table 8.

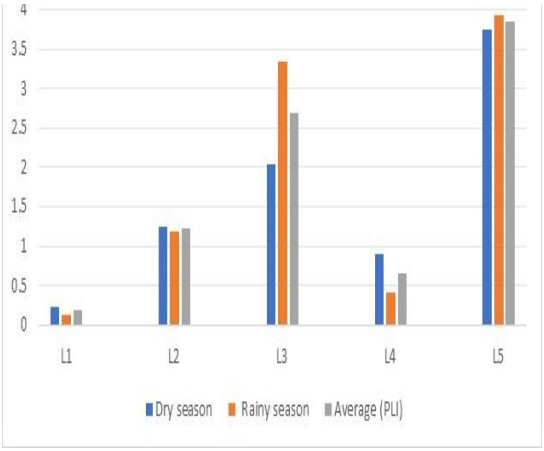

Fig 14 : The pollution load index of the study sites, L1 – L5

Table 7: Geoaccumulation index (lgeo) for sediments along Eastern Obolo coastline

Metal	L1 D	L1 R	L2 D	L2 R	L3 D	L3 R	L4 D	L4 R	L5 D	L5 R
V	0.03	0.01	–	–	0.24	0.45	–	–	0.57	0.72
Cr	9.25×10^{-3}	4.79×10^{-3}	0.06	0.0048	–	–	–	–	0.37	0.51
Mn	0.05	0.03	0.03	0.04	0.0075	0.62	0.07	0.02	0.37	0.51
Fe	0.04	0.03	0.06	0.05	0.0067	0.66	0.16	0.07	0.62	0.61
Co	–	–	–	–	–	–	–	–	–	–
Ni	0.01	7.02×10^{-3}	0.05	0.03	–	–	–	–	–	–
Cu	5.12×10^{-3}	2.90×10^{-3}	0.08	0.06	–	–	–	–	–	–
Zn	0.06	0.04	0.13	0.12	0.66	1.08	0.55	0.36	1.57	1.81
Cd	–	–	–	–	–	–	–	–	–	–
Pb	0.05	0.03	0.14	0.13	–	–	–	–	–	–

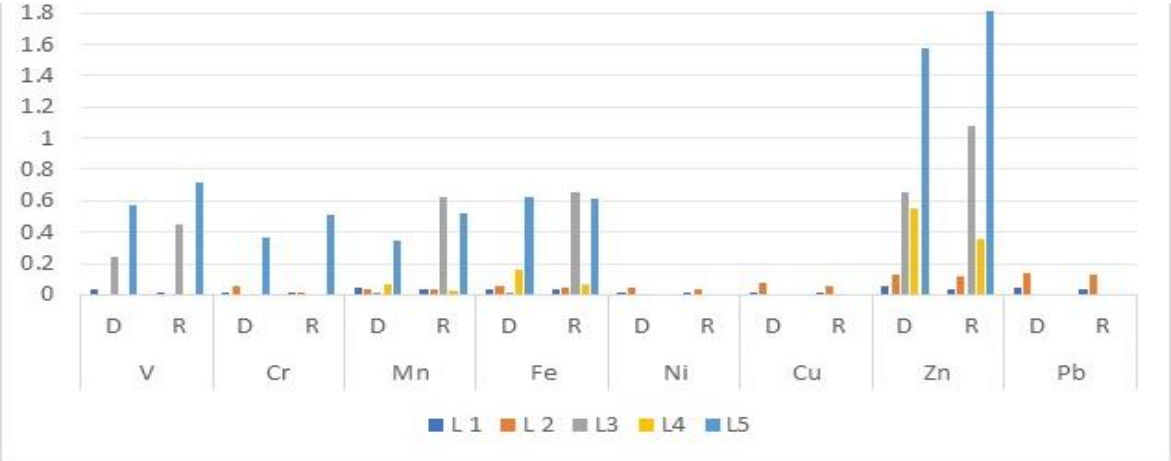

Fig. 14 : Geoaccumulation index (lgeo) for sediments along the Eastern Obolo coastline

Table 8 : The potential ecological risk assessment for the heavy metals in the sediments

Study Location	Calculated E_f^i for individual heavy metals									R_i
	Zn	**Cr**	**Ni**	**Pb**	**As**	**V**	**Cd**	**Mn**	**Cu**	
L1	0.26	0.07	0.27	0.96	4.65	0.20	-	0.21	0.10	6.70
L2	0.63	0.53	1.04	3.43	7.96		-	0.18	1.71	15.48
L3	4.33	-	-	-	-	3.45	-	2.47	-	10.25
L4	2.26	-	-	-	-	-	-	0.22	-	2.48
L5	8.43	3.64	-	-	-	6.44	-	1.93	-	20.44
Min	0.26	0.07	0.25	0.98	4.65	0.20	-	0.18	0.10	2.48
Max	8.43	3.64	1.04	3.43	7.96	6.44	-	2.47	1.71	20.44
Mean	2.58	1.41	0.65	2.21	6.31	3.36	-	0.62	0.91	11.07

This is clearly due to the low levels of highly toxic heavy metals such as Cd and Pb. The present results for PERI are generally in agreement with several previous studies and reported case studies in which Cd alone was found to have considerably spiked the Ri values for the studied sites, into the high-risk zone (Cd alone making over 93 per cent contributions to the site's Ri (Ma and Han, 2020).

In comparison with this study, Sediments from both Senegal and Morocco had the highest concentrations of chromium (Cr), copper (Cu), Pb and cadmium (Cd), which were also above standard limits (threshold effect level, TEL) for Canada (Diop et al. 2015), while the results from this study were lower for these metals. However, the results of zinc determination in sediments from this study were higher than values determined for sediments in Asa River, Nigeria (26.6 – 148.0 mg/kg) and sediments in Dakar coast, Senegal (7.17 – 88.0 mg/kg) (Fagiga *et al.* 2016).

4. 0 Conclusion

The study has established the levels and distributions, as well as ecological and geoaccumulation of V, Cr, Mn, Fe, Ni, Cu, Pb, Co, Cd, in sediments and water from five sampling sites (Atlantic coastline, Iko river, Obolo river, Amadaka river and Emeremen river) of Iko river marine ecosystem, Akwa Ibom State, Nigeria. Except for Cd and Co which were not detected in any of the sample matrices from all the study sites, the heavy metals have varied distributions and abundance ranging from undetected, low to high concentrations across the study sites and media during the dry and rainy seasons. However, the observed temporal and spatial variations of the studied heavy metals in the sediments were not statistically significant ($P \leq 0.05$), implying a common origin for these metals along the study sites. Strong correlations (0.5-0.99) between the other metals and Fe, Mn and Zn were revealed. The order of contamination in sediments was (mg/kg dw) Fe (6421.75) > Mn> Zn> V> Cu > Pb> Cd and Co (which were not detected in all seasons, locations and media). Fe, Mn, Zn,V, were higher than average reference shale values. Natural, but mostly anthropogenic activities, including petroleum production in the area, accounted for the metals enrichment as indicated by the ecological indices (lgeo, E_f, C_f, mCd, PERI, Ri etc).

Fe, Mn, and Zn were found in the water at all the study sites (L1, L2, L3, L4, and L5) at relatively higher concentrations (mg/l) than the other heavy metals, including Co, Cd, and Cr . Most of the metals including Co, Cd, and Cr, were not detected at all the sites in both

seasons. The mean pH of the water, across the five sampling sites ranged from pH 5.75 to 7.40, which was relatively high. The pH values were mostly within the slightly acidic or alkaline range which, according to some studies, favours precipitation and less solubility of heavy metals and thereby determines the quality of the sediment. Low concentrations of the metals in the water compared to the sediments at all the sites were probably due in part to bioaccumulation or bioconcentration of the metal by aquatic biota species (fishes, benthic species, plants like mangrove and seagrasses, etc

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Compliance with Ethical Standards Declaration

Ethical Approval

Not Applicable

Competing interests

The authors declare that they have no known competing financial interests

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Authors' Contribution

All authors participated in the field and laboratory aspects of the work. The draft manuscript was written by the authors under the supervision of the corresponding author.