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Ecological Risk Indices and Statistical Methods for Pollution Evaluation in Contaminated Sediment from Warri River, Southern Nigeria

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Abstract: This study aimed to investigate the pollution level and ecological risk conditions of heavy metals in the sediment of Warri River. Heavy metals determined using the Atomic Absorption Spectrophotometer (AAS) AA 500L model, showed a descending order of Fe>Zn>Cu>Mn>Cr>Pb>Cu. The sediment qualities and ecological indices were assessed using enrichment factor (ER), geo-accumulation index (Igeo), contamination factor (CF), contamination degree (CD), pollution load index (PLI) and potential ecological risk indices (PERI) at three stations: Agbarho, Enerhen and Onwian-Aladja (station 1, 2, and 3). Correlation and hierarchical cluster analysis were utilised to examine the origins of sediment heavy metals. In all sediment samples, Cu and Zn were higher than the threshold-effects level (TEL) values, while Zn was the only metal value higher than the probable-effects level (PEL). Based on the ecological indices, the studied metals were found to have significant considerable enrichment of 5≥20 with respect to Fe, Cu and Zn. The Igeo values of all the trace metals indicated unpolluted to severely polluted status ($0 \leq \text{Igeo} \leq 3$). The CF (>6) and CD (>24) indicate mild to extreme contamination and moderate to extremely high contamination. The ER range (0.05–374.85) indicates low to high ecological risk, while the PLI (>1) values indicate high contamination. Cluster analysis revealed that stations with high contamination levels were linked to contaminants. Thus, the accumulation of heavy metals in the studied sediment is primarily attributed to the anthropogenic activities and probable other environmental factors.

1. Introduction

Aquatic habitats significantly impact the trophic quality of water bodies by facilitating diverse biological processes (Mirandaa *et al*., 2020). However, sediment intrusion into rivers can have direct and indirect detrimental effects on aquatic organisms. These impacts include reduced light penetration, habitat deterioration, suffocation of organisms, and the introduction of absorbed contaminants (Heery *et al.,* 2017). The composition of sediment comprising various particle sizes such as sand, silt, and clay, along with organic matter and toxic contaminants, significantly influences the distribution of species that depend on it for habitat (Zhang *et al.,* 2014). The Niger Delta represents Nigeria's most resource-rich region, characterised by significant hydrocarbon reserves (Ejiba *et al.,* 2016). The extraction of oil and related operations have resulted in a rise in waste generation and its discharge into waterways. A significant quantity of metals, pesticides, furans, polychlorinated biphenyls and phthalates are transported to the sediment annually. The United States Environmental Protection Agency has identified 128 priority pollutants, with 65 per cent primarily or exclusively associated with sediment and biota (Luigi *et al.,* 2015). Metal pollution in aquatic ecosystems has received significant attention owing to its toxicity, persistence, and accumulation in the environment. Heavy metals (HMs) rapidly adhere to particles and become incorporated into the sediment at the bottoms of aquatic environments following their release (El Hammari *et al.,* 2022; Goswami and Neog, 2023). Heavy metals accumulate in soft tissue and are detrimental when not metabolised by the body (Akartasse *et al.,* 2017; Karim *et al.,* 2019; Fu and Xi, 2020). Consequently, they can disrupt normal biological processes in organisms and lead to detrimental effects on various aquatic life forms. Research indicates that river sediments are essential for the absorption and transport of metals in aquatic ecosystems (Al-Afify and Abdel-Satar, 2020; Ali *et al.,* 2022). Owing to the restricted solubility of heavy metals, more than 85% of effluent discharge ultimately accumulates in sediments. These sediments act as the principal repository for heavy metals, which are accumulated by aquatic species and then infiltrate the food web (Miao *et al.,* 2021). This presents considerable ecological and public health challenges, since toxic metals can result in enduring environmental harm and health complications. Restoring and monitoring the biological integrity of our aquatic ecosystems, while ensuring the safeguarding of human health and aquatic organisms, requires the preservation of sediment quality.

Statistical approaches have been extensively applied in recent years to identify possible dangers connected to high concentrations of HM in the evaluation and management of aquatic ecosystems. Several indices have been employed including the geo-accumulation index (Igeo), contamination factor (CF), contamination degree (CD) and enrichment factor (EF), to evaluate the ecological risk and HM contamination (Mahvi *et al.,* 2022). The baseline concentrations of HM constitute the basis for the development of these widely employed pollution indices. Consequently, sediment quality is crucial for assessing the general status of the ecosystem in an aquatic habitat (Kabir *et al.,* 2020). Thus, this paper aims to assess the environmental distribution of HM and the ecological risks posed by contaminated sediments from the Warri River.

2. Methodology

2.1 Study Area

Warri River spans approximately 255 sq km^2 and extends between latitude 5°2' - 6°00' N and longitude 5°24' - 6°2' E. The river originates around Utagba-Uno and flows through Odiete, Agbarho, and other areas in Warri City before draining into the Atlantic Ocean at the Forcardos (Egborge, 1991). This river functions as a source of water supply for household usage. The area is encompassed by many neighbouring towns and industrial establishments such as Chevron Nigeria Limited, Matix Energy Services Limited, and Warri Refinery and Petrochemical Company Limited. The water body of this river receives municipal, industrial, and residential waste from the surrounding communities and industries.

2.2 ***Sampling Stations***

The sampling stations were chosen due to their proximity to areas with human activities. Station 1 is located in Agbarho, in a zone of fishing activities (5^o 24 6^o 21'E and 5^o 21 6^{o}0'N) situated along the river. Station 2 (Enerhen) is in proximity to the Enerhen market (5°47'8.232" N and 5°30' 31.636" E) where the market waste empties into this station. Station 3 (Ovwian-Aladja) is situated behind Premium Steel and Mines Limited, formerly known as Delta Steel Company; this facility discharges effluent directly into the river. (**Figure 1**).

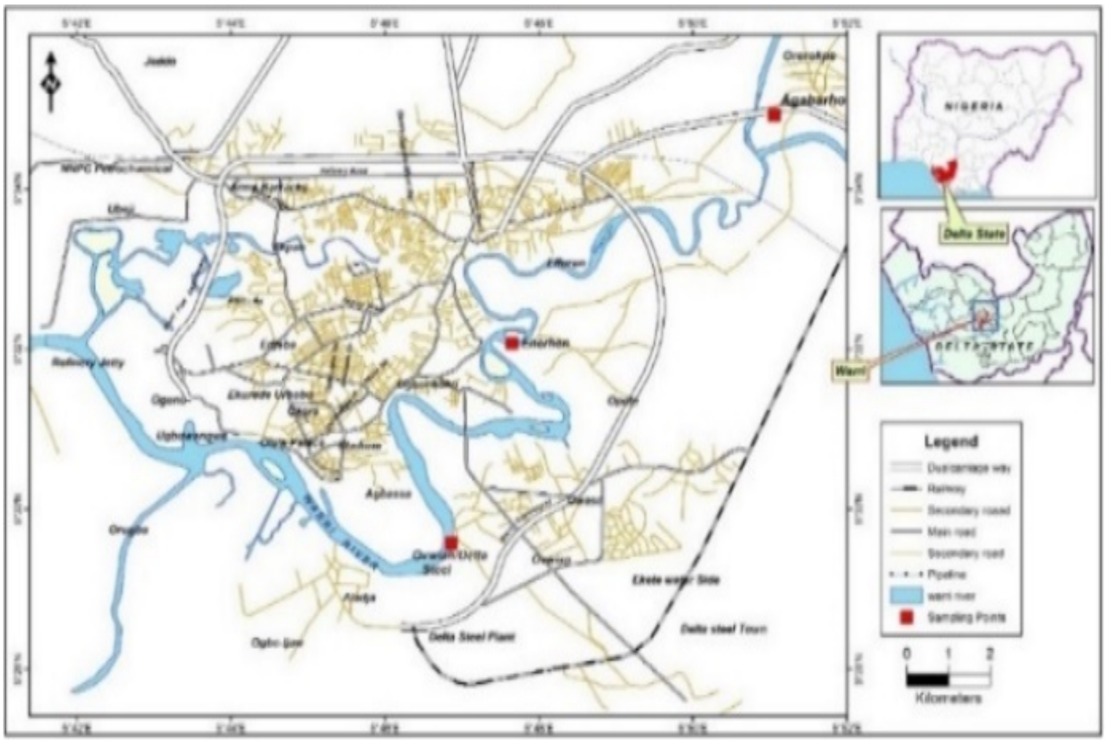

Figure 1. Map of Warri River showing the sampling stations

2.3 Sediment Collection

Fifteen sediment samples (N=15) were taken monthly from January to March 2023 along Warri River, covering majority of the polluted sections in Ovwian-Aladja (station 3), Enerhen (station 2) and Agbarho (station 1). Sediment samples were taken from a depth of 0-10 cm at each selected station with a Van Veen grab. After sampling, the sediment was placed into a labelled polyethene plastic bag transported to the laboratory in an icebox, and preserved at −20 °C (Liu *et al.,* 2008).

2.4 Sample Extraction and Analysis

The samples were allowed to air dry before being pulverised with a mortar. Subsequently, samples were sifted with a 2 mm mesh sieve to aid digestion. The mixture was placed into a microwave at 190°C for 15 minutes and allowed to cool before being washed several times with deionised water until the whole sample was completely removed. After using a white 42 mm filter paper to filter the mixtures, 50 mL of deionised water was added, and the volume was topped up. For heavy metals, the digested samples were analysed via an Atomic Absorption Spectrophotometer (AAS) AA 500L model.

2.5 Quality Control and Assurance

To ensure the accuracy and precision of the analytical results, all chemical analyses were conducted in triplicate. Blanks, duplicates, and certified reference materials (CRM) were used to validate the method. The recovery rates for all metals were within the acceptable range of 95–105%. Calibration curves for each metal were constructed using multi-element standards, and the quality of the data was verified using certified sediment standards.

2.6 Ecological Risk Assessment

2.6.1 Enrichment Factor (EF)

To characterise the effect of human-induced pollution, the following equation in Eqn.(**1**)

$$\textbf{EF} = \frac{(Cm\backslash Cx)\text{sample}}{(Cm\backslash Cx)\text{ background}} \qquad \textbf{Eqn. 1}$$

Cm/Cx represents the ratio of the sample concentration, whereas Cm/Cx denotes the ratio of the background concentration. <2 indicates low; 2≥5 denotes moderate; 5≥20 signifies considerable enrichment; 20≥40 represents strong enrichment; >40 reflects high enrichment.

2.6.2 Geo-accumulation Index (Igeo)

The level of contamination of bottom sediment was assessed using Eqn (**2)**

$$\textbf{Igeo} = log_2\left(\frac{Cn}{1}.5\ Bn\right) \qquad \textbf{Eqn. 2}$$

Cn denotes the concentration of heavy metals in sediment samples; Bn represents the geochemical local baseline concentrations of the metal. The factor of 1.5 indicates the change in the background value. Categorising pollution levels is as follows: < 0 indicates unpolluted; ≤ 1 denotes slight pollution; ≤ 2 denotes moderate pollution; ≤ 3 reflects moderate to severe pollution; ≤ 4 represents severe pollution; ≤ 5 indicates severe to extreme pollution; and > 5 denotes extreme pollution.

2.6.3 Contamination Factor (CF)

Sediment pollution levels were determined using the following **Eqn** (3)

$$\textbf{CF} = \frac{Cm\ sample}{Cm\ background\ \ value} \qquad \textbf{Eqn}\ (3)$$

A CF value less than 1 denotes mild, 1 to 3 shows moderate, 3 to 6 indicates severe, and a CF value more than 6 indicates extreme contamination.

*2.6.4 Contamination`Degree(CD)* `

The formula used for CD was calculated using the Eqn.4 :

$$\textbf{CD} = \sum_{i=0}^{n} \textbf{CF} \qquad \textbf{Eqn}\ (4)$$

Classified contamination is given as minor (< 6), moderate (6 < 12), significant (12 < 24), and extremely high (≥ 24) contamination degrees.

2.6.5 Pollution Load Index (PLI)

The formula used for PLI was estimated using the **Eqn** (5)

$$\textbf{PLI} = (\textbf{CF}_{1x}\, \textbf{CF}_{2x}\ \ldots\ldots..\textbf{CF}_n\ \ 1/n) \qquad \textbf{Eqn}\ (5)$$

where n represents the concentration of metals and CF denotes the contamination factor value.

2.6.6 Potential Ecological Risk Index (PERI)

The toxicity of metals in the sediment was calculated using the **Eqn** (6)

$$\text{PERI} = \sum \text{ERI1} + \text{ERI2} + \text{ERI3} + \text{ERI4} \ \textbf{Eqn}\ (6)$$

Er ≤ 40 implies minimal ecological risk, 40 ≤ 80 suggests moderate, 80 ≤ 160 indicates significant, 160 ≤ 320 indicates high, and >320 indicates extremely high.

2.7 Statistical Analyses

Inter-station comparisons were conducted to determine significant differences between the heavy metals and physicochemical characteristics. Excel (2010) and SPSS 16.0 were used to perform the Duncan Multiple Range (DMR) test and parametric analysis of variance (ANOVA) for significant discrepancies ($p<0.05$). To ascertain whether there was a substantial correlation between heavy metals and the physico-chemical parameters, correlation coefficients were calculated. Blue colour in the visual output denote positive correlation while red colour denote negative correlation. Dark colour signify a greater level of correlation, the darker the colour the stronger the correlation.

3. Results and Discussion

3.1 Physicochemical Characteristics

Table 1 presents the average mean values of the physicochemical parameter in sediment from Warri River. The mean pH values of the collected sediment at the three stations ranged from 2.83-6.12. According to the standard of the WHO these values were not within the acceptable limits of 6.5-8.5. The low pH signifies that the river sediment is acidic, which may influence the diversity of piscivores (Rahel and Nibbelink,1999). A notable disparity in pH across the stations ($p < 0.01$) may be ascribed to the riverbed's geology and the degree of contamination from surface runoff. Shao *et al*. (2016) reported an average pH of 7.3 and 8.3 for surface sediment porewater pH and water column in northern South China Sea deep waters. The observed pH from this study is consistent with Islam *et al.* (2021).The Electrical Conductivity (EC) values for the sediment varied from 102.67 to 2014.67 µS/cm and were not below the WHO recommended limit (700 µS/cm), except for results at station 1. Dissolved ions affect EC and may originate from both natural and man-made sources. A high EC value signifies a substantial concentration of dissolved ions, including inorganic salts. We observed a significant difference ($p < 0.01$) in EC between the sediment samples. Electrical conductivity is a metric that determines the degree of dissolved particles in substances. An imbalance of ions in soil or water could bring about some physiological disorders in plants and animals (Gavrilescu, 2021**).**

Table 1: Physicochemical Characteristics of Sediment of Warri River

Parameters	Station 1 X±SD	Station 2 X±SD	Station 3 X±SD	p-value
Ph	6.12^{a} ±0.45	2.83^{b} ±0.25	6.12^{a} ±0.45	p<0.01

EC (us/cm)	102.67[c] ±51.39	8430.67[a] ±569.62	2014.67[b] ± 1266.04	$p<0.01$
Organic Carbon (%)	0.67[c] ±0.26	11.13[a] ±0.15	7.07[b] ±1.95	$p<0.01$
Organic Matter (%)	1.16[c] ±0.44	19.24[a] ±0.26	12.22[b] ±3.38	$p<0.01$
Total Nitrogen (%)	0.09[b] ±0.05	1.58[a] ±0.50	0.95[a] ±0.40	$p<0.05$
THC	34.33[b] ±3.38	230.65[a] ±66.28	59.38[b] ±37.27	$P<0.05$
Sand (%)	91.76±6.02	76.72±9.42	78.99±9.66	$p>0.05$
Silt (%)	1.60[c] ±0.54	15.17[a] ±3.47	8.67[b] ±1.34	$p>0.05$
Clay (%)	6.64±5.51	6.64±5.51	12.34±8.36	$p>0.05$

The organic carbon ranged from 0.67-11.13 % and organic matter from 1.16 % in station 1 to 12.22 % in station 2. The values were lower than those recorded for organic matter in the study of the sediment physicochemical characteristics at Maruba Dam Reservoir, Machakos, Kenya (Luvai *et al.,* 2022). The total nitrogen ranged from 0.09-1.58 % in station 2 with the highest value of 1.58 %, while station 1 had the lowest value of 0.09 %. The nitrogen content in sediment offers essential insights about the quality of sedimentary organic matter. The values found in this study were lower compared to a previously published study (Feng *et al.*, 2022). The low level of macronutrient nitrogen in this river indicates heightened human activity, particularly at station 2. Total Hydrocarbon Content (THC) of the sediments ranged from 34.33 and 230 mg/kg. Station 2 (Enerhen) had the highest value of 230 mg/kg, while the lowest value of 34.33 mg/kg was obtained from station 1 (Agbarho). The high concentrations of total hydrocarbons obtained in sediment from station 2 may be attributed to illegal refinery operations. This may also have arisen due to intermittent discharge from oil pipelines and installations around this location.

Sediment particle analysis of Warri River was dominated by sand particles, which ranged from 76.72-91.76 %. The silt and clay fractions in the sediments varied from 1.60% to 15.17% and from 6.64% to 12.34%, respectively. This is similar to the investigation of Bodo Creek in the Niger Delta that reported sand particle dominance (Vincent-Akpu *et al.,* 2015) but contrary to the reports of Nwaja Creek (Adowei and Bale, 2023). Individual sampling sites showed that station 2 had a greater silt component (15.17%) than station 3 (8.67%) and station 1 (1.60%). The percentage clay content was higher in station 3 (12.34 %) than in station 2 (8.11%) and station 1 (6.64) respectively. The proportions of sand, clay, and silt particles in bottom sediments were not the same, with major significant differences observed for silt particles. Hence, this may be attributed to the geological diversity in this catchment area.

3.2 Concentration of heavy metals

The overall HM concentration (**Table 2**) across the sampling stations varied from 86.27-775.41 mg kg^{-1} for Fe,0.00- 0.53 mg kg^{-1} for Pb,0.78-3.20 mg kg^{-1} for Cr, 0.00-0.12 mg kg^{-1} for Cu,10.59-66.93 mg kg^{-1}, 38.85-374.85 mg kg^{-1} for Zn and 2.67-64.99 mg kg^{-1} for Mn. The heavy metal concentration profile in the sediment was in descending order, Fe>Zn>Cu>Mn>Cr>Pb>Cu. Fe exhibited the greatest variation in sediment concentration across all sampled stations. High metal concentrations were found at station 2, with the exception of Pb, while station 3 had the highest mean levels in all heavy metals analysed. This might be related to the human activities observed in those two stations.

Table 2 indicates that the concentration of HM obtained from the bottom sediment did not exceed the background levels except for Cu and Zn at station 2 and station 3. Heavy metal levels in the various stations were below the Sediment Quality Guidelines (SQGs) by the United States Environmental Protection Agency (USEPA) (Macdonald *et al.*, 1996). Cu and Zn were the only metals in stations 2 and 3 that had values higher than the threshold-effects level (TEL), while Zn was the only metal value higher than the probable-effects level (PEL) in all sediment samples. The fact that stations 2 and 3's TEL values for Cu and Zn were higher than PEL indicates that these stations are moderately toxic.

Table 2: Concentration of heavy metals in sediment of Warri River

3.3 Enrichment Factor (EF)

HM mg/kg	Station 1 x±SD	Station 3 x±SD	Station 2 x±SD	p-value	WSQG a	ChSQG b	CSQG c	TEL d	PEL f
Fe	86.27 c ±7.99	775.41 a ±217.82	508.33 b ±9.80	p<0.05	4.72	-	-	-	-
Pb	0.00 b ±0.00	0.47 a ±0.20	0.53 a ±0.15	p<0.05	20	60	300	30.2	112
Cr	0.78 b ±0.16	3.20 a ±1.21	2.23 a ±0.16	p<0.05	-	87	200	52.3	160
Cd	0.00±0.00	0.12±0.13	0.08±0.08	p>0.05	0.3	22	0.3	0.68	4.21
Cu	10.59 b ±2.06	66.93 a ±38.52	55.71 a ±26.11	p<0.05	45	91	100	18.7	108
Zn	38.85 b ±11.87	374.85 a ±101.67	323.21 a ±57.12	p<0.05	95	360	250	124	271
Mn	2.67 b ±2.80	64.99 a ±12.38	49.87 a ±4.56	p<0.01	850	770	-	-	-

The results of the enrichment factor (EF) in the bottom sediments (Table. 3) exhibited the following order from highest to lowest: Mn>Zn>Fe>Cu>Cr>Pb. Negligible levels of contamination (EF<2) were detected for Pb and Cr. Stations 2 and 3 showed a moderate enrichment (2<5) of Pb and Cr, and a high enrichment (5≤20) of Cu and Zn. A depletion of metal enrichment was observed in station 1, except Mn. This metal is unlikely to come from natural sources since the EF values are above 60. Mn had the highest enrichment (EF>40), which exhibited exceptionally high levels of enrichment at the station. The stations with the highest Mn enrichment were stations 2 and 3.

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HM	Station 1	Station 2	Station 3	All samples

Table 3: **Enrichment factor values of measured metals in Warri River**

Fe	1.09	9.83	6.44	5.79
Pb	0.04	1.87	2.13	1.35
Cr	1.26	5.17	3.60	3.34
Cu	1.21	7.67	6.38	5.09
Zn	1.34	12.9	11.17	8.49
Mn	2.54	61.89	47.49	37.31

3.4 Geo-accumulation Index (Igeo)

The findings of the estimated Igeo in (Table. 4) indicate that all the metals at station 1 fell into grade 0, suggesting an unpolluted status. The calculated Igeo values for station 2 and station 3 were < 4. It may be inferred from the values that the two stations have mild to moderate levels of Pb, Fe, Cu, Cr, and Zn contamination. The Mn, Igeo is > 4 in station 3 and < 6 in station 2, denoting a high level of pollution in the bottom sediment**.**

Table 4: Geo-accumulation index values for sediment samples from the Warri River

HM	Station 1	Station 2	Station 3	All samples
Fe	-0.46	2.71	2.10	1.45
Pb	-5.23	0.32	0.51	-1.47
Cr	-0.25	1.78	1.26	0.93
Cu	-0.31	2.35	2.09	1.38
Zn	-0.16	3.11	2.89	1.95
Mn	0.76	5.37	4.98	3.70

3.5 Potential Ecological Risk

Table 5 depicts the ecological risk rating for heavy metals in Warri River sediments. With the exception of Pb and Cr, all heavy metals in the stations appear to be present at considerable levels of pollution, as indicated by their values, in which the values were greater than 40. Furthermore, the heavy metals were ranked in order of probable ecological risk index as follows: Zn > Cu > Mn > Cr > Pb. In addition, a higher concentration of Zn and Cu ranged from 40 to 400, signifying their elevated pollution levels in the sampled locations. Station 1 and station 2 were found to have the lowest and highest values, respectively. Similarly, the lowest known levels of the metals, except Cu, may have caused the lowest PER at station 1.

Table 5: Potential Ecological Risk values for sediment samples from the Warri River

HMs	Station 1	Station 2	Station 3
Pb	0.05	2.67	2.33
Cr	1.57	4.47	6.41
Cu	52.95	278.53	334.67
Zn	38.85	323.21	374.85
Mn	2.67	49.87	64.99
Total	96.08	658.74	783.25

3.6 Contamination factor (CF) contamination degree (CD) and pollution load index (PLI)The calculated contamination factor for sediment in Warri River is provided in (table 5). Average heavy metals ranked in order of their highest CF is Mn>Zn>Fe>Cu>Cr>Pb, respectively. Table 6 shows the mean CD and PLI values for heavy metals found in Warri River sediments. Furthermore, the two sampling locations were found to have CD (> 24). Based on the average CD values, the stations' pollutant levels are arranged as follows: 2 > 3 > 1. Pollution Load Index values are presented in (Table. 6). The PLI results ranged from 0.78 to 9.14. The average PLI value was computed as 5.78. The PLI for the heavy metals in the sediments exceeded 1 in stations 2 and 3, with the exception of station 1. Significant anthropogenic impacts on sediment quality are indicated by sediments with greater PLI values (PLI > 1), whereas substantial anthropogenic activity is not suggested by sediments with lower PLI values (PLI < 1).

Table 6: Contamination Factor (CF), Contamination Degree (CD) and Pollution Load Index (PLI) index values for sediment samples from designated station (ST) the Warri River

ST	Fe	Pb	Cr	Cu	Zn	Mn	CD	PLI
1	1.09	0.04	1.26	1.21	1.34	2.54	7.49	0.78
2	6.44	2.13	3.60	6.38	11.17	47.49	77.22	7.42
3	9.83	1.87	5.17	7.67	12.96	61.89	99.38	9.14

3.7 Correlation Analysis

pH correlated positively with sand while negatively with EC, organic carbon, organic matter, N, silt, Fe, Pb, Cr, Zn, and Mn (Fig. 2). A positive correlation is shown by the colour blue, whereas a negative correlation is indicated by the colour red. The square box indicates a significant correlation, while the colours without the square signify an insignificant correlation. Electrical conductivity exhibits a positive and significant correlation with organic carbon, organic matter, N, silt, Fe, Cr, Zn and Mn as organic carbon and organic matter exhibit similar parameters as EC except with Pb.

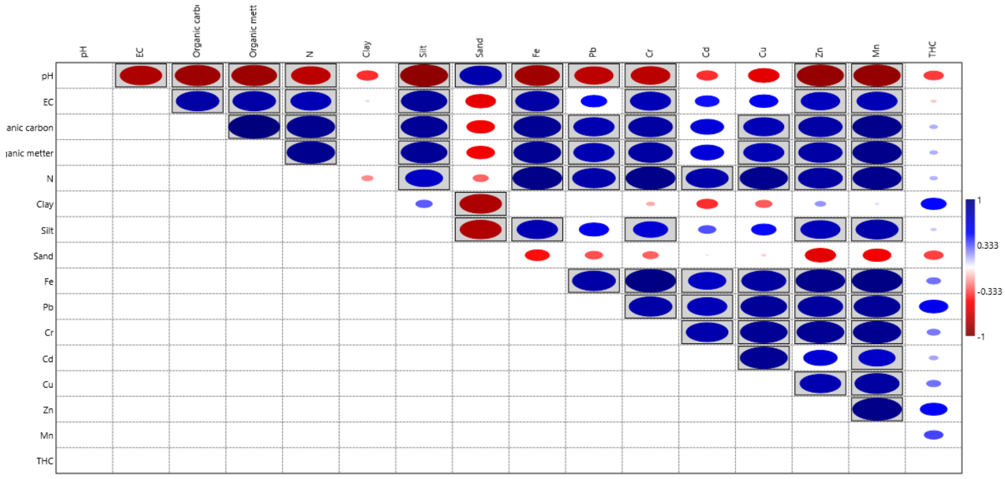

Figure 2: **Pearson correlation analysis between heavy metals and other parameters in bottom sediment**

3.8 *Cluster analysis, Ward-Euclidean similarity and distance indices*

The Euclidean similarity and distance indices for each study location are displayed in (Table. 7). Based on the concentrations of all parameters examined in the bottom sediments of the Warri River, Figure 3 represents the dendrogram for cluster analysis. Three separate groups, station 1, station 2, and station 3, are formed from the sampled areas. The dissimilarity between places was 1989.463, 8363.728, and 6424.0856, according to the Euclidean dissimilarity and distance index.

Table 7: **: Euclidean similarity and distance indices across the stations**

Stations	1	2	3
1	0	1989.4628	8363.7281
2	1989.4628	0	6424.0856
3	8363.7281	6424.0856	0

The dendrogram's clustering indicates that the conditions that prevailed in station 1 throughout the sample period were more comparable to those that were observed at station 2. Furthermore, the conditions that prevailed at station 1 and station 2 differed greatly from those that prevailed at station 3. The physical and chemical properties of the bottom sediments at three sites along the Warri River displayed a distinct variance based on the cluster combinations.

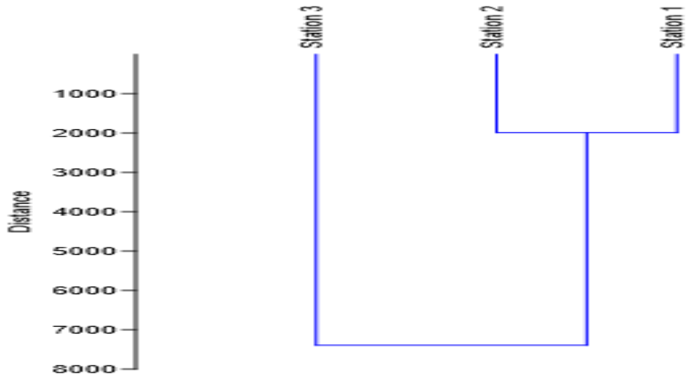

Figure 3: **Dendogram for cluster analysis based on distance indices on sampled station**

Conclusion

The quality of bottom sediment is critical to the river's long-term viability. In general, heavy metal concentrations were greater in locations with significant anthropogenic impact. The selected ecological risk assessment approach revealed that the river is exposed to a high environmental risk owing to metal contamination. This is an indication of pollution, and polluted wastewater is being discharged uncontrolled into the natural catchment of the Warri River without prior treatment by industries and communities in the region. To attain a cleaner environment in Nigeria, regulatory and enforcement authorities should intensify efforts to monitor the compliance of various companies with set effluent restriction rules.

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Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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