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(RESEARCH ARTICLE)

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Polyaromatic hydrocarbon distribution, source apportionment and ecological risks in the sediment of okpare river, olomu, delta State, Nigeria

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Abstract

A significant quantity of contaminants is produced primarily by anthropogenic activities degrades water quality. Toxic pollutants in aquatic systems can be stored in sediment, which may then impact aquatic life, humans who consume food from these systems, and the water column itself. This study examines the distribution, source apportionment, and ecological risks of Polyaromatic Hydrocarbon (PAHs) in the sediment of Okpare River, Olomu, Delta State, Nigeria. Samples were collected over 12 months, encompassing both the wet (May 2022 to October 2022) and dry seasons (November 2022 to April 2023). A total of eighty-four samples were obtained from seven sampling stations positioned 100 meters apart along the Okpare River, following standard methods and procedures. The sediment had mean values of Naphthalene (0.0005 mg/kg, 0.0018 mg/kg), Benz(a)anthracene (0.0067 mg/kg, 0.015 mg/kg), Benzo(a)pyrene (0.036 mg/kg, 0.027 mg/kg), Indeno (1,2,3-cd) pyrene (0.023 mg/kg and 0.028 mg/kg) and Benzo (g,h,i) perylene (0.026 mg/kg, 0.033 mg/kg) during the wet and dry seasons. PAHs (5 rings) were found in higher percentages in the sediments in both seasons. Diagnostic ratio results indicated that PAHs in the sediment may be attributed to both pyrogenic and petrogenic origins. Ecological risk assessment of PAHs in the sediment showed that the concentration of all the 16 priority PAHs were < ERL value. Although the are higher concentrations of 5rings PAHs in the sediment, they do not pose a significant ecological risk at present. Nevertheless, ongoing monitoring is essential to safeguard the health of the aquatic ecosystem and those dependent on it.

Keywords: Sediment; Heavy metals; Apportionment; Diagnostics Ratio

1. Introduction

Water bodies possess a natural capacity to counteract the detrimental effects of contaminants discharged into them. However, when anthropogenic activities and other natural sources produce an overwhelming quantity of pollutants, water becomes degraded, occasionally surpassing human utilization thresholds. Contaminants exceeding these limits result in the pollution of water within the aquatic system (Boyd, 2020). Deforestation and expanded land utilization represent the inevitable consequences of urbanization and population growth, impacting both biodiversity and the quality of surface water (John, 2020). The excessive use of land and deforestation expose the topsoil, facilitating the erosion of loose soil particles into water bodies through water flow and wind. This leads to increased sedimentation, ultimately resulting in heightened turbidity in aquatic systems (Tundu *et al.* 2018). Elevated turbidity impedes sunlight from reaching the water column's bottom, causing a reduction in photosynthesis and, subsequently, a decrease in dissolved oxygen (Wen *et al*. 2007; Boyd, 2015).

Toxic pollutants received by water bodies may dissolve in the water column, adhere to suspended sediment, or settle as sediment at the bottom of the water bodies (Adesuyi *et al.* 2016). Sediments constitute a complex mixture of particulate compounds such as sand, silt, clay, and organic substances (Reuther, 2009). Sediments serve as a reservoir in the aquatic system, potentially becoming a source of toxic pollutants to the water column, aquatic organisms, and

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humans consuming these organisms from the water body (Adesuyi *et al.* 2016). Therefore, it is imperative to conduct sediment characterization during water quality assessment. Particle size, pH, organic carbon content, nitrate, and phosphorus influence the interaction and dynamics of contaminants within the sediment (Adesuyi *et al*. 2016). Particle size plays a crucial role in contaminant adsorption in the sediment matrix because fine particles, like silt and clay, possess a larger surface area, adsorbing more contaminants than coarse particles such as sand and gravel with a smaller surface area (Adesuyi *et al.* 2016). According to Yanhao *et al.* (2018), a study evaluating the effect of pH on the release of heavy metals from polluted sediment demonstrated that acidic pH conditions are more likely to release heavy metals like cadmium, nickel, and copper back into the water column. Total organic carbon is a pivotal parameter for estimating the level of organic contaminants in sediments (Palvos *et al*. 2015). Organic carbon in aquatic systems primarily originates from natural sources like plant and animal decomposition and anthropogenic sources such as fertilizer use in farms and the discharge of organic-rich wastewater into the environment (Palvos *et al.* 2015).

The global focus on the contamination of water bodies with heavy metals, notably lead, chromium, mercury, and cadmium, has intensified. This concern arises from the inherent toxicity, non-biodegradability, environmental persistence, and potential for accumulation in organisms through biomagnification, even at low concentrations (Alprol *et al.* 2021; Lata *et al.* 2022). The surge in heavy metal pollution is attributed to the discharge of untreated industrial wastewater, expanded mining operations, degradation of underground pipes, agricultural activities, and illegal refinery practices (Anyanwu *et al*. 2018).

Many settlements in the Olomu kingdom, Delta state, Nigeria, depend heavily on the Okpare River, a tributary of the Niger Delta. It provides the main source of water for irrigation, home use, fishing, and leisure activities. Regretfully, oil leaks from illicit bunkering operations in the Okpare community have had a substantial negative impact on the river (Onajite and Ovie, 2022). Understanding the source, distribution, and ecological danger of polycyclic aromatic hydrocarbons—of which crude oil is a source—is essential for creating effective mitigation plans and preserving the Okpare River's ecosystem's health as well as the welfare of its human inhabitants.

2. Materials and Method

2.1. The study area

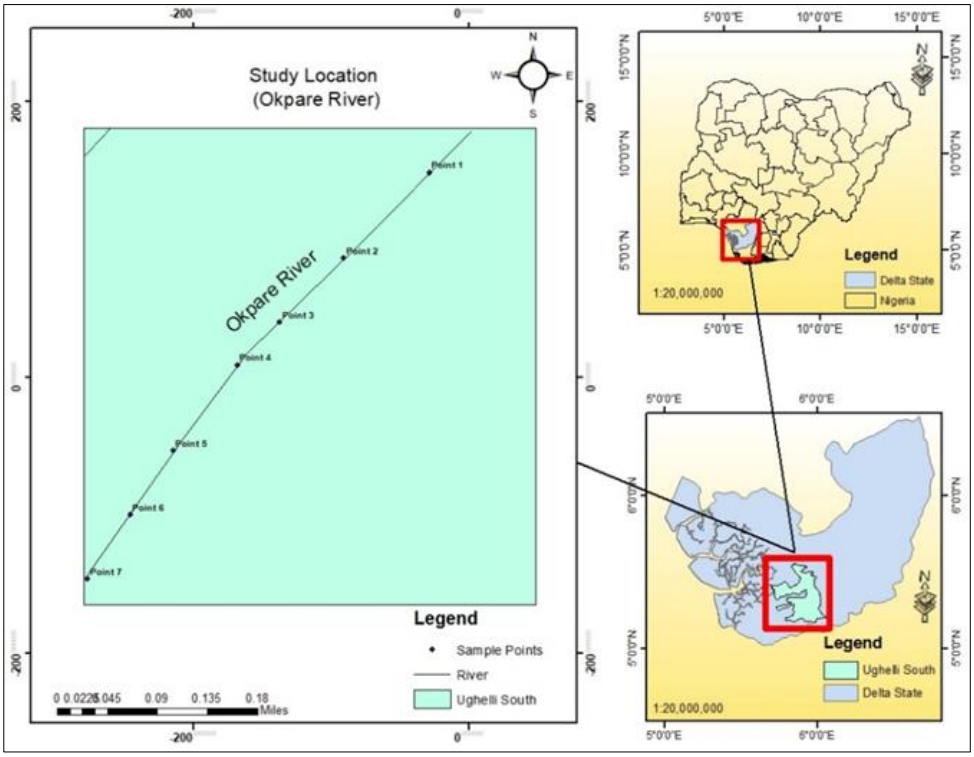


Figure 1 Map of the Study Area

The Okpare River rises in Umuaja, Umutu, and drains into the Atlantic Ocean near the Forcados Estuary (Onajite and Ovie, 2022). The study area lies between latitudes 05^27N'47.0 and N05^27'28.0 and longitudes E005^54'19 and E005^54'3. The river flows from the northwest to the southeast. The study area has two seasons: the wet season, which runs from May to October with a brief dry spell in August, and the dry season, which goes from November to April and is marked by a dusty haze from the northeast winds (Ekanem & Nwagbara, 2005; Diejomaoh and Okoro, 2024). Figure 1 presents the map of the study area showing the sampling points.

2.2. Sampling of Sediment

The Eckman sediment grab was used to collect sediment samples. During a 12-month period that included both the wet (May 2022 to October 2022) and dry (November 2022 to April 2023) seasons, sediments were collected from the seven sampling locations spaced 100 meters apart along the Okpare river. Eighty-four samples in total were taken from seven sampling locations. The sediment samples were put into polyethylene bags (Inyang *et al.* 2018). All samples were preserved at 4°C and transported to the laboratory.

2.3. Analytical Methods

Extraction of Sediment Samples for PAHs was done Using Soxhlet extraction Method (APHA 6440 B). 100 g of sediment sample was weighed, air dried and sieved. The sediment sample was wrapped in filter paper placed in a thimble, and then loaded into the main chamber of the Soxhlet extractor, and extracted with dichloromethane over a period of 24 hr.

This was followed up by column chromatography for cleanup. The sediment extracts were transferred into a 10 mm ID × 30 cm chromatographic column packed with 10 g activated silica gel slurry with about 2 cm anhydrous sodium sulphate layer on top. The column was eluted with 20 mL of dichloromethane to obtain the aromatic fraction. The eluates were concentrated to about 2 mL with a rotary evaporator at 30 °C. All the cleaned sample extracts were analyzed using a gas chromatography – Flame Ionization Detector (GC - FID) (Inyang *et al.* 2018).

2.4. Quality Control and Quality Assurance

The analytical method's ability to produce valuable and reliable analytical data were confirmed by calculating the percentage recovery, limit of detection (LOD), and limit of quantification (LOQ). The percentage recovery was determined after ten samples underwent analysis by adding a known concentration of analyte to the sample matrix and subsequently analyzing it. The percentage that recovered ranged from 82.1% to 110%. Continuous dilution and standard solution analysis were used to establish LOD until the lowest concentration was reached at a signal to noise ratio of 3. Likewise, LOQ was ascertained, standard solution was continuously diluted and analyzed until the lowest concentration was found at a signal ratio of 10.

2.5. Statistical Analysis

The data generated underwent descriptive statistical analysis to obtain the mean of the PAHs concentration. The sediment quality guideline method and the diagnostic ratio calculations were computed with Microsoft Excel Office 365.

2.6. Ecological Risk Assessment

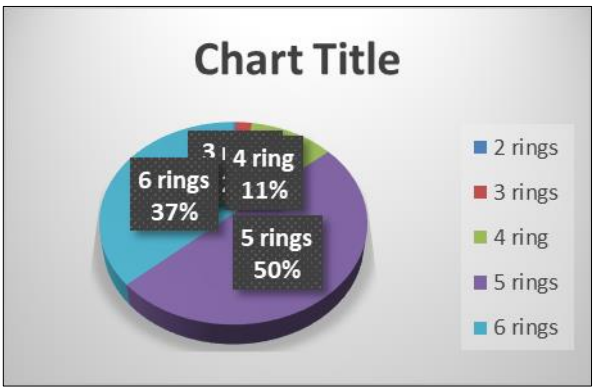
Sediment quality guidelines (SQGs) are quantitative chemical concentration values intended to forecast negative impacts on biological resources, preserve biological resources, or do both (Richard and Christopher, 2002). SQGs provide two guideline values: effects range low (ERL) and effects range median (ERM), established using the 10th and 50th percentiles, respectively, in a database of increasing concentrations associated with adverse biological effects (Dong *et al*. 2021). ERM is the chemical concentration at which harmful consequences would frequently occur, whereas ERL represents the chemical concentration below which harmful effects on aquatic life is considered low, according to Long and Morgan, (1991). Concentration values between ERL and ERM suggest a potential effects range within which effects may occur either intermittently or irregularly (Dong *et al*. 2021). The effect range low values and effect range high values of the 16 priority PAHs, recommended by MacDonald *et al*. 2002 are presented in Table 1.

Table 1 Values of ERL and ERM (µg/kg) for individual PAHs (MacDonald *et al.* 2002)

Polycyclic Aromatic Hydrocarbons	Effect Range Low µg/kg	Effect Range High µg/kg
Naphthalene	160	2100
Acenaphthylene	16	500
Acenaphthene	44	640
Fluorene	19	540
Phenanthrene	240	1500
Anthracene	85	1100
Fluoranthene	600	5100
Pyrene	665	2600
Chrysene	384	28000
Benzo(a)anthracene	261	1600
Benzo(b)fluoranthene	320	1880
Benzo(k)fluoranthrene	280	1620
Benzo(a)pyrene	430	1600
Indeno(1,2,3-cd) pyrene	-	-
Dibenz(a,h)anthracene	63.4	260
Benzo(g,h,i)perylene	430	1600

3. Result and discussion

3.1 Distributions of Individual Polycyclic Aromatic Hydrocarbons in the Sediment



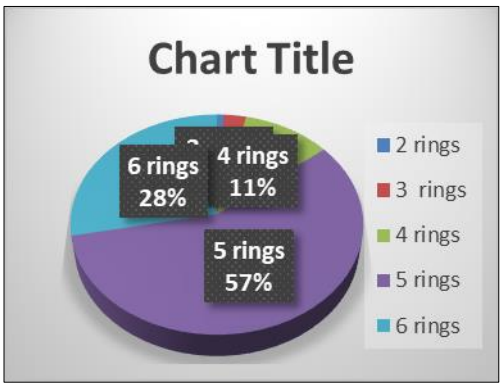


Figure 2 PAHs Distribution During the Wet Season

Figure 3 Distribution During the dry Season

The concentrations of Naphthalene had mean values of 0.0005 mg/kg and 0.0018 mg/kg in the wet and dry seasons respectively. Acenaphthylene levels had mean concentrations of 0.0005 mg/kg and 0.0017 mg/kg in the wet and dry seasons respectively. Meanwhile, Acenaphthene concentrations had average values of 0.0005 mg/kg and 0.0015 mg/kg in the wet and dry seasons respectively. Fluorene concentrations had mean levels of 0.0005 mg/kg and 0.0012 mg/kg in the wet and dry seasons respectively. Phenanthrene concentrations had mean values of 0.001 mg/kg and 0.0015 mg/kg during the wet and dry season. Anthracene concentrations had average values of 0.0012 mg/kg and 0.0015 mg/kg during the wet and dry seasons respectively. Fluoranthene concentrations had mean values of 0.002 mg/kg and 0.0025 mg/kg during the rainy and dry season. Pyrene levels had average values of 0.002 mg/kg and 0.0025 mg/kg during the wet and dry seasons respectively.

Chrysene levels had average values of 0.0028 mg/kg and 0.0032 mg/kg. Benz(a)anthracene levels had average values of 0.0067 mg/kg and 0.015 mg/kg. Benzo(b)fluoranthene concentrations had average values of 0.012 mg/kg and 0.024 mg/kg during the wet and dry seasons respectively. Dibenz(a,h)anthracene had the in the highest mean concentration in the surface water during the wet season. Benzo(k)fluoranthene concentrations had mean values of 0.014 mg/kg and 0.046 mg/kg during the wet and dry season. Benzo(a)pyrene levels had average values of 0.036 mg/kg and 0.027 mg/kg during the wet and dry seasons respectively. Indeno (1,2,3-cd) pyrene levels had average values of 0.023 mg/kg and 0.028 mg/kg. Dibenz (a, h) anthracene levels had average values of 0.036 mg/kg and 0.029 mg/kg. Benzo (g,h,i) perylene concentrations had average values of 0.026 mg/kg and 0.033 mg/kg during the wet and dry seasons respectively. Two categories of PAHs were identified from the measurements in this study: 2 to 3 rings (low molecular weight, LMW), four rings, and 5 to 6 rings (high molecular weight, HMW). The distribution of PAHs in the Okpare River in the wet season was in the order 5 rings > 6 rings > 4 rings > 3 rings > 2 rings (Figure 2 and Figure 3). High molecular weight (HMW) PAHs were present in high concentration in the sediment in the study area in both season (Figure 2 and Figure 3). Sogbanmu *et al*. (2019) reported higher ∑PAHs values in the range of 302-1290 µg kg-1 in a study conducted in Lagos lagoon, Nigeria. Tongo *et al*. 2013 reported higher range 5.25 to 573.33 µg/kg- from Ovia River, Nigeria. Eguvbe reported higher mean concentrations of PAHs in the range N.D - 2,654 µg/kg (wet season) and N.D to 3,513 µg/kg (dry season) from a study conducted in selected creeks in Delta state.

Table 2 Distribution of Polycyclic Aromatic Hydrocarbons in the Sediment during the wet and dry Season

		Wet Season		**Dry Season**	
Polycyclic aromatic Hydrocarbons	**Number of Rings**	**Range (mg/kg)**	**Mean (mg/kg)**	**Range (mg/kg)**	**Mean (mg/kg)**
Naphthalene	2	0.00 – 0.002	0.0005	0.00 – 0.006	0.0018
Acenaphthylene	3	0.00 – 0.002	0.0005	0.00 – 0.007	0.0017
Acenaphthene	3	0.00 – 0.001	0.0005	0.00 – 0.007	0.0015
Fluorene	3	0.00 – 0.002	0.0005	0.00 – 0.006	0.0012
Phenanthrene	3	0.00 – 0.002	0.001	0.00 – 0.009	0.0015
Anthracene	3	0.00 – 0.004	0.0012	0.00 – 0.009	0.0015
Fluoranthene	4	0.00 – 0.006	0.002	0.00 – 0.015	0.0025
Pyrene	4	0.00 – 0.006	0.002	0.00 – 0.014	0.0027
Chrysene	4	0.00 – 0.012	0.0028	0.00 – 0.006	0.0032
Benz(a)anthracene	4	0.00 – 0.026	0.0067	0.00 – 0.028	0.015
Benzo(b)fluoranthene	5	0.00 – 0.048	0.012	2.00 – 0.055	0.024
Benzo(k)fluoranthrene	5	0.00 – 0.052	0.014	8.00 – 0.028	0.046
Benzo(a)pyrene	5	0.00 – 0.103	0.036	9.00 – 0.061	0.027
Indeno(1,2,3-cd) pyrene	5	0.003 – 0.039	0.023	1.00 – 0.055	0.028
Dibenz(a,h)anthracene	6	0.001– 0.089	0.036	3.00 – 0.071	0.029
Benzo(g,h,i)perylene	6	0.002– 0.064	0.026	0.00 – 0.079	0.033

3.2 Ecological Risk Assessment Results of PAHs in the Sediment

The PAHs concentration(µg/kg) obtained in the study area are compared to sediment quality guideline values of ERL and ERM (Table 2 and Table 3), the mean concentrations of the 16 priority PAHs, ranging from 0.50 – 35.83 µg/kg in the wet season and 0.16 – 45.50 µg/kg in the dry season, were all below the ERL values (Table 3). This indicates that the potential for PAHs levels in the study area to harm aquatic life is low.

Table 3 Toxicity Risk Assessment of the Sediment in the Study Area

		Wet Season		Dry Season			
PAHs	**Abbreviation**	**Range**	**Mean (µg/kg)**	**Range**	**Mean (µg/kg)**	**ERL (µg/kg)**	**ERM (µg/kg)**
Naphthalene	Nap	N.D – 2.00	0.50	N.D – 6.00	1.83	160	2100
Acenaphthylene	Acy	N.D – 2.00	0.50	N.D – 7.00	0.16	16	500
Acenaphthene	Ace	N.D – 1.00	0.50	N.D – 7.00	1.50	44	640
Fluorene	Flu	N.D – 1.00	0.50	N.D – 6.00	1.16	19	540
Phenanthrene	Phe	N.D – 4.00	1.00	N.D – 9.00	1.50	240	1500
Anthracene	Ant	N.D – 4.00	1.16	N.D – 9.00	1.50	85.3	1100
Fluoranthene	Flt	N.D – 6.00	2.00	N.D – 15.00	2.50	600	5100
Pyrene	Pyr	N.D – 6.00	2.00	N.D – 14.00	2.66	665	2600
Chrysene	Chr	N.D – 12.00	8.50	N.D – 6.00	3.16	384	2800
Benzo(a)anthracene	BaA	N.D – 26.00	6.66	N.D – 28.00	14.83	261	1600
Benzo(b)fluoranthene	BbF	N.D – 48.00	12.33	N.D – 55.00	24.30	320	1880
Benzo(k)fluoranthrene	BkF	N.D – 17.00	13.66	2 – 180.00	45.50	280	1620
Benzo(a)pyrene	BaP	3 – 103.00	35.83	8 – 61.00	26.83	430	1600
Indeno(1,2,3-cd) pyrene	InP	3 – 37.00	23.33	9 – 55.00	28.33	-	-
Dibenz(a,h)anthracene	DBA	4 – 89.00	36.16	3 – 0.07	28.50	63.4	260
Benzo(g,h,i)perylene	B(ghi)p	2 – 64.00	26.33	3 – 0.8	32.50	430	1600

Note: ERL – Effect Range Low; ERM – Effect Range Medium; ND – Not Detecte

Table 4 The Results of Diagnostic Ratio of PAHs in the Study Area

	Diagnostic Ratio Guideline Values		**Calculated Diagnostic Ratio**	
Diagnostic Ratio	**Petrogenic**	**Pyrogenic**	**wet**	**Dry**
Ant/Ant+Phe	< 1	> 1	0.54	0.50
InP/InP + B(ghi)p	< 0.2 - < 0.5	> 0.5	0.47	0.46
Flt/Flt+Pyr	< 0.4 – 0.5	> 0.5	0.50	0.48
B(a)P/B(ghi)P	> 0.6	< 0.6	1.36	082
LMW/HMW	> 1	< 1	0.03	0.04
B(a)A/ B(a)A+Chr	< 2 – 0.35	> 0.35	0.70	0.82

Note: Ant - Anthracene; Phe – Phenanthrene; InP - Indeno(1,2,3-cd) pyrene; B(ghi)p - Benzo(g,h,i)perylene, Flt - Fluoranthene, Pry – pyrene, B(ghi)p - Benzo(g,h,i)perylene; B(a)A - Benz(a)anthracene; B(a)P - Benzo(a)pyrene; Chr – Chrysene; LMW – Low Molecular Weight; HMW – High Molecular Weight

3.3 Source Apportionment

The diagnostic ratios for the 16 priority PAHs analyzed in the study area during both the wet and dry seasons are presented in Table 4.12 and Table 3. The calculated Ant/Ant+Phe ratios during both seasons were > 0.1, indicating a potential source of PAHs from pyrogenic source. The InP/InP + B(ghi)p ratios during both seasons was within the range > 0.2 - < 0.5, suggesting petrogenic origin. Flt/Flt+Pyr ratio values were between 0.4 and 0.5 in both seasons, implying

that the PAHs may originate from petrogenic source. B(a)P/B(ghi)P ratios were consistently > 0.6 in both seasons, indicating a likely source of PAHs from petrogenic. The LMW/HMW values during both seasons were < 1, suggesting that the PAHs may be from pyrogenic sources. Additionally, B(a)A/B(a)A+Chr values in both seasons were > 0.35, indicating that the origin of PAHs in the study area may be attributed to the pyrogenic origin.

4 Conclusion

The distribution of polyaromatic hydrocarbons, source allocation, and ecological risks are investigated in the Okpare River sediment in Olomu, Delta State, Nigeria. Compared to low molecular weight PAHs, high molecular weight PAHs were detected in higher proportions; 5-ring compounds predominated in the sediment during the rainy season and 6-ring compounds during the dry season. Very low ecological assessment results suggest that there may not be any harmful effects of PAHs in the research area. Both pyrogenic and petrogenic origins are probably connected to the source of PAHs in the investigated area.

Compliance with ethical standards

Acknowledgments

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Disclosure of conflict of interest

No conflict of interest to be disclosed.

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