Integrated Ecological Pollution Impact of Selected Heavy Metals in Microplastics in Sediments, Rivers State, Nigeria

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Abstract
This study evaluated the concentrations of heavy metals (Ni, Cr, Fe, Hg, Cd, Cu, Pb, and Mn) adsorbed to microplastics in sediments (MCPS) along Woji, Okujagu, and Elelenwo creeks in Port Harcourt, Nigeria, during the wet and dry seasons. Monthly sediment samples were collected from December 2020 to May 2021 during low tide events. The concentrations of metals in the microplastics in sediment (MCPS) were analysed using GBC Avanta PM A6600 Flame Atomic Absorption Spectrophotometer (FAAS) and compared to sediment quality guidelines (TEL and PEL) to assess the potential ecological risks and contamination levels. In the dry season, Woji creek showed the highest concentrations of Ni, Cr, Fe, and Cu in MCPS, while Hg levels were more significant in Woji, with a toxicity response factor (T_r) of 30. Conversely, Elelenwo exhibited the lowest metal concentrations for most of the metals. The calculated geoaccumulation index (I_geo) and contamination factor (CF) revealed that Woji and Okujagu creeks were moderately contaminated with Fe and Cu, while Hg concentrations indicated a high ecological risk. The potential ecological risk index (PERI) showed significant contributions from Hg and Cu, particularly at Woji and Okujagu. During the wet season, Ni, Cr, Fe, and Cu concentrations decreased, with the lowest concentrations observed in Elelenwo. However, the PERI showed no significant risks from Hg, Cu, or Mn, as these metals were present in lower quantities. These findings underscore the need for continued monitoring of metal contamination, and highlight the potential ecological and health risks posed by heavy metal-laden microplastics in estuarine environments.

Keywords: *microplastics, sediment, TEL, PEL, Port Harcourt*

1. Introduction

The escalating contamination of aquatic ecosystems by microplastics and heavy metals has emerged as a significant environmental concern globally. Microplastics, defined as plastic particles smaller than 5 mm, have been identified as pervasive pollutants in marine and freshwater environments. Their small size and hydrophobic nature facilitate the adsorption of various contaminants, notably heavy metals, thereby amplifying their potential ecological risks (Rochman et al., 2013). In the Niger Delta region of Nigeria, extensive industrial activities, including oil exploration and artisanal mining, have led to the release of substantial quantities of heavy metals into the environment. Studies have reported elevated concentrations of heavy metals such as cadmium (Cd), lead (Pb), and mercury (Hg) in water and sediment samples from this area, posing significant ecological and human health risks (Iwegbue, 2007; Ololade, 2014). Furthermore, the widespread use and improper disposal of plastic materials have exacerbated microplastic pollution in the region's waterways. For instance, the Osun River has been documented to contain high levels of microplastics, primarily attributed to the indiscriminate disposal of single-use plastics and domestic waste (Akindele, 2022).

The interaction between microplastics and heavy metals in sediment matrices is particularly concerning. Microplastics can act as vectors for heavy metals, facilitating their transport and bioavailability in aquatic environments. This interaction can lead to the bioaccumulation of toxic metals in aquatic organisms, thereby entering the food chain and posing health risks to humans (Brennecke et al., 2016). Despite the critical implications, research focusing on the combined presence and ecological risks of heavy metals adsorbed onto microplastics in the sediments of the Niger Delta's waterways remains limited. Most studies in the Niger Delta have primarily focused on assessing heavy metal contamination in sediments and water. Iwegbue (2007) investigated the distribution of heavy metals in sediments and surface water of a crude oil-impacted area, revealing significant contamination levels. Similarly, Ololade (2014) conducted an ecological risk assessment in soils and sediments of an industrial area in southwestern Nigeria, highlighting the potential risks posed by heavy metal pollution. However, these studies did not consider the role of microplastics as carriers of heavy metals, an aspect that has been explored in other regions. For instance, a study in Songkhla Lagoon, Thailand, reported the co-occurrence of microplastics and heavy metals in sediments, emphasizing the compounded ecological risks (Chae et al., 2020).

Given the paucity of data on the integrated pollution impact of heavy metals adsorbed to microplastics in the Niger Delta, this study aims to fill this critical knowledge gap. By evaluating the concentrations of heavy metals adsorbed to microplastics in sediments along Woji, Okujagu, and Elelenwo creeks in Port Harcourt, Nigeria, during both wet and dry seasons, this work seeks to provide insights into the spatial and temporal variations of these contaminants. The findings will contribute to a better understanding of the ecological risks associated with heavy metal-laden microplastics in estuarine environments and inform strategies for pollution mitigation and environmental management in the Niger Delta region.

2. Materials and Methods

2.1 Study Area

The study area is located in the brackish water estuary of Woji Creek, Elelenwo Creek and Okujagu Creek as shown in Fig.1. The creeks as earlier reported by Isaac (2024b), Isaac and Nwineewii (2024) are located in the city of Port Harcourt, Rivers State, Nigeria. The creek is one of the tributaries of the Sombreiro River traversing the north down to the south of Rivers State into the North Atlantic, a well-defined route of transportation (Ibezim-ezeani & Ihunwo, 2020). The tidal influence of the North Atlantic upstream is responsible for the saline ocean water brought into the creek thus enriching the creeks with both freshwater and salt water organisms (Dibofori-Orji et al., 2019; Ibezim-Ezeani & Ihunwo, 2020).

Woji creek lies along the Bonny River estuary at latitude 7° 2'49.58"E, and longitude 4°48'48.53"N (Table 3.1). The Woji Creek has a confluence with the refinery creek at Okujagu to form the main tributary which drains into the Bonny River. The creek has border with the Port Harcourt-Trans-Amadi industrial layout, the industrial hob of Rivers State. There are several anthropogenic activities such as barge and cargo manufacturing, a major abattoir as well as human settlements along the river. The Woji River drainage basin is located at the heart of Obio-Akpor Local

Government Areas in Port Harcourt. The Woji River has a meandering flow amid channel blockages upstream as culvert ending creates a fall in the channel (Anya et al., 2017; Iyama et al., 2020).

Elelenwo creek is on latitude 7° 3'55.29"E, and longitude 4°49'41.89"N (Table 3.1). The major industrial activities taking place in Elelenwo study area comprise a major abattoir that serves the state, oil servicing company and a computer village designated for sales and repairs of computers. A domestic waste dump site is also noticed at the bank of the Creek.

Okujagu creek is on latitude 7° 4'34.22"E, and longitude 4°48'37.49"N (Table 3.1). This study site is an estuarine creek located on the eastern fringes of Port Harcourt city in the upper Bonny estuary of the Niger Delta, Nigeria. Okujagu river just like Woji creek, receives almost equivalent industrial and domestic wastes consequent upon alternate low and high tides experienced by both creeks by virtue of their locations. There is also obvious dredging, oil bunkering, boat maintenance activities with deblitating effect of anthropogenic activities on-going within the upper Bonny estuary of the Niger Delta compared to the adjacent creeks. Red mangroves (Rhizophora racemose) and Nypa palms (Nypa fructican) line the shores. Apart from the refinery effluents received from refining activities at Okujagu, the most prevalent activities include sand moving, fishing and boat ferrying are the major activities in the study area.

2.2 Sampling

Samples were collected monthly from December 2020 to May, 2021 and during low tide event at three stations at approximately 3 km stretch from each creek. Three sediment samples were collected transversely from each station along the creeks using two sets of shovels (plastic and steel), to collect the top layer soft sediment (≈10 cm in depth). The samples were taken approximately 1m from the shore at each station with a steel and a plastic shovel, each marked differently to differentiate sediments sampled for microplastics and metals respectively. Samples were put into well-labelled foil bags (indicating sampling point information and time of sampling) and placed into ice chest coolers at 4 ^{0}C and transferred to the laboratory.

2.3 Metal Analysis in Sediments, MCPS Samples

Microplastic in sediment (MCPS) samples were air-dried in the laboratory at room temperature, pulverized independently and then sieved through a 2 mm pore size sieve to remove coarse particles. Samples of MCPS were subjected to partial acid digestion following the method US EPA 3050B (USEPA, 1996) and described by Isaac & Nwineewii (2024); Vedolin et al, 2018. Exactly 2g each of MCPS was put in a 50 mL beaker and then 5 mL of concentrated HNO_3, 3.0 mL of H_2O_2 (30 % V/V) and 10 mL of HCl were added at 90 °C. Samples were digested on a Corning PC-351 model hot plate at medium to low heat until about 5 ml concentrated extract was left (or with sample concentrate tending towards near-dryness). Afterwards, the content of beaker was left to cool for around 30 minutes. Sample solution was filtered and quantitatively transferred into 50 ml standard volumetric flask. Finally, filtered solutions were made up to the 50 ml graduation mark using distilled water. Thereafter, metals (Cd, Cu, Cr, Fe, Mn, Ni, Pb and Hg) levels were determined using the GBC 908PBMT model Flame Atomic Absorption Spectrophotometer (FAAS). Each sample was individually aspirated. The total metal concentrations are reported in units of mg/kg.

3. Results and Discussion

3.1 Heavy metal Concentrations in MCPS

Table 1: Concentrations of heavy metals (Ni, Cr, Fe & Hg) in MCPS during dry and wet seasons in comparison with sediment quality guidelines

MCPS Location	Ni (mg/kg)	Cr (mg/kg)	Fe (mg/kg)	Hg (mg/kg)
Dry Season				
Woji	**15.17** ± 1.22 [a,b]	9.28 ± 9.35 [c]	4,429.17 ± 547.81[a]	0.06 ± 0.00
Okujagu	9.34 ± 3.27[a]	8.17 ± 3.25 [c]	4,078.76 ± 56.76 [a]	0.03 ± 0.00
Elelenwo	5.80 ± 1.71[c, b]	7.83 ± 2.69[c]	3,369.90 ± 721.91[a, b]	0.02 ± 0.01
Wet Season				
Woji	4.45 ± 0.37[a, b]	3.49 ± 0.20 [c]	3,492.93 ± 783.15[a]	<0.001
Okujagu	2.59 ± 0.24[b, b]	4.26 ± 0.15 [c]	3,909.39 ± 21.97 [b]	<0.001
Elelenwo	2.38 ± 0.16[b]	<0.001	736.16 ± 29.13[b]	<0.001

Source: Isaac & Nwineewii (2024)

Table 2: Concentrations of heavy metals (Cd, Cu, Pb & Mn) in MCPW during dry and wet seasons in comparison with sediment quality guidelines

MCPS Location	Cd (mg/kg)	Cu (mg/kg)	Pb (mg/kg)	Mn (mg/kg)
Dry Season				
Woji	1.46 ± 0.62 [c]	24.24 ± 8.33 [c]	19.66 ± 2.00 [c]	112.54 ± 16.30 [a]
Okujagu	1.20 ± 0.73 [c]	12.73 ± 5.03 [c]	8.40 ± 6.42 [c]	85.44 ± 32.89 [a]
Elelenwo	0.71 ± 0.74 [c]	10.19 ± 4.14 [c]	5,14 ± 4.45 [c]	70.00 ± 47.23[c]
Wet Season				
Woji	0.18 ± 0.02 [c]	1.95 ± 0.04 [c]	<0.001	19.57 ± 0.35[c]
Okujagu	<0.001	1.25 ± 0.12 [c]	<0.001	19.82 ± 0.19 [c]
Elelenwo	<0.001	<0.001	<0.001	0.87 ± 0.03 [c]

Source: Isaac & Nwineewii (2024)

3.2 Pollution Indices in MCPS at the Study Area

3.2.1 Geoaccumulation Index (I_geo)

The I-geo values indicate that most metals in the study area exhibit minimal contamination, with values consistently negative for Ni, Cr, Mn, Cd, and Pb across both seasons. These findings align with studies in the Niger Delta that report naturally low levels of these metals in sediments (Olatunji et al., 2021). The observed Fe contamination, particularly in the dry season (I_geo = 1.42 to 1.77), suggests moderate enrichment, likely due to anthropogenic activities such as industrial discharge and metal corrosion from pipelines. This trend is consistent with findings from Warri and Bonny Rivers, where Fe enrichment has been attributed to both natural geochemical weathering and industrial effluents (Adebiyi & Oladipo, 2019). In contrast, Cu showed mild contamination in the dry season, particularly in Woji and Okujagu, with I_geo values reaching 1.72. Similar trends have been reported in areas with high industrial activity, such as the Ogoniland region, where elevated Cu levels are linked to metal plating industries and shipyard activities (Nwankwoala et al., 2022). The seasonal variation, where Cu levels were significantly lower in

the wet season, suggests dilution effects from increased river flow, a pattern also observed in previous studies on the Niger Delta (Oghenejoboh & Abiodun, 2018).
The most concerning I_geo values were observed for Hg in the dry season (-0.32 in Woji), which, although not strongly positive, suggests a detectable anthropogenic influence. Mercury contamination in the Niger Delta is often linked to oil exploration and artisanal mining activities, which can release trace amounts into the environment (Isaac & Nwineewii, 2024). The absence of detectable Hg in the wet season further supports the dilution hypothesis, aligning with observations from previous wet season studies in the region.

3.2.2 Contamination Factor (CF)

The CF values reinforce the patterns observed in I_geo, confirming low contamination for most metals except Fe, Cu, and Hg during the dry season. Fe showed moderate contamination, with CF values exceeding 1.5 in both seasons, supporting findings from previous research that attribute high Fe levels in Niger Delta sediments to industrial runoff and pipeline corrosion (Oghenejoboh & Abiodun, 2018).
Cu exhibited CF values above 1 in the dry season (1.37 in Woji), indicating moderate contamination, consistent with previous studies that link **Cu** accumulation in sediments to shipbreaking yards and metal-based industries in the Niger Delta (Nwankwoala et al., 2022). The drop in CF values during the wet season to nearly zero suggests significant dilution, a pattern also observed in Bonny Estuary, where metal concentrations fluctuate seasonally due to hydrological variations (Olatunji et al., 2021). The CF values for Hg in the dry season were relatively high (1.2 in Woji), indicating contamination, albeit at a moderate level. This is concerning given mercury's high toxicity, even at low concentrations. In contrast, studies in the heavily polluted Taylor Creek and Nun River have reported CF values exceeding 3 for Hg, underscoring the variability in contamination across different regions of the Niger Delta (Isaac & Nwineewii, 2024). For Mn, **Cd**, and **Pb**, the CF values were consistently low (<0.2), suggesting no significant contamination. This is consistent with reports from relatively less industrialized areas in the Niger Delta, where background concentrations of these metals are naturally low (Adebiyi & Oladipo, 2019).

3.2.3 Potential Ecological Risk Index (PERI)

The PERI results highlight Hg as the primary ecological risk factor in the study area, particularly in the dry season, where Woji recorded a contribution of **36**, significantly higher than any other metal. This suggests that even moderate mercury contamination poses substantial ecological risks due to its high toxicity response factor (T_r = 30). Similar PERI values have been recorded in sediment studies near oil spill sites in Ogoniland, where mercury contamination is linked to legacy pollution from crude oil extraction and refining activities (Isaac & Nwineewii, 2024). **Fe** and **Cu** also contributed to PERI, particularly in the dry season, indicating that while their toxicity is lower than Hg, their relatively higher concentrations make them significant risk factors. These results align with studies in the Forcados and Nun Rivers, where **Fe** and **Cu** were found to be dominant contaminants in industrialized regions (Olatunji et al., 2021). For **Ni**, **Cr**, **Cd**, **Mn**, and **Pb**, PERI values were negligible, reflecting their low contamination levels. This is consistent with findings from less impacted areas of the Niger Delta, such as the coastal regions of Bayelsa State, where these metals pose minimal ecological risks (Nwankwoala et al., 2022).
The ecological indices indicate that while heavy metal contamination in the study area is generally low, localized risks exist, particularly for Hg, Fe, and **Cu** during the dry season. These metals,

especially **Hg**, have the potential to bioaccumulate in aquatic organisms, posing long-term risks to both the ecosystem and human health. The seasonal trends, where metal contamination is more pronounced in the dry season, emphasize the role of hydrological conditions in modulating pollution levels. This seasonal variation has been observed in multiple studies across the Niger Delta, highlighting the need for continuous monitoring, especially in industrial zones (Oghenejoboh & Abiodun, 2018). The findings suggest that while some areas remain relatively uncontaminated, industrial discharges, oil spills, and corrosion from aging infrastructure contribute to localized heavy metal accumulation. Given the Niger Delta's vulnerability to oil pollution, proactive environmental management strategies, including stricter industrial waste regulations and periodic sediment monitoring, are necessary to mitigate potential ecological risks.

Table 3: Ecological Indices for MCPS during Dry season

Heavy Metal	Location	Concentration (C_n) [mg/kg]	Background (B_n) [mg/kg]	I_geo	CF	Toxicity Response Factor (T_r)	PERI Contribution
Ni	Woji	15.17	20	-0.97	0.7585	1	0.7585
	Okujagu	9.34	20	-1.07	0.467	1	0.467
	Elelenwo	5.8	20	-1.41	0.29	1	0.29
Cr	Woji	9.28	25	-0.77	0.3712	1	0.3712
	Okujagu	8.17	25	-0.81	0.3268	1	0.3268
	Elelenwo	7.83	25	-0.83	0.3132	1	0.3132
Fe	Woji	4429.17	2000	1.42	2.2146	1	2.2146
	Okujagu	4078.76	2000	1.36	2.0394	1	2.0394
	Elelenwo	3369.9	2000	1.08	1.6849	1	1.6849
Hg	Woji	0.06	0.05	-0.32	1.2	30	36
	Okujagu	0.03	0.05	-0.73	0.6	30	18
	Elelenwo	0.02	0.05	-1.32	0.4	30	12
Cd	Woji	0	0.1	-∞	0	5	0
	Okujagu	0	0.1	-∞	0	5	0
	Elelenwo	0	0.1	-∞	0	5	0
Cu	Woji	41.06	30	1.72	1.3687	5	6.8435
	Okujagu	37.88	30	1.59	1.2627	5	6.3135
	Elelenwo	10.03	30	-0.68	0.3343	5	1.6715
Pb	Woji	0	20	-∞	0	5	0
	Okujagu	0	20	-∞	0	5	0
	Elelenwo	0	20	-∞	0	5	0
Mn	Woji	133.22	1000	-2.88	0.1332	1	0.1332
	Okujagu	106.37	1000	-3.27	0.1064	1	0.1064
	Elelenwo	62.5	1000	-3.98	0.0625	1	0.0625

Table 4: Ecological Indices for MCPS during wet season

Heavy Metal	Location	Concentration (C_n) [mg/kg]	Background (B_n) [mg/kg]	I_geo	CF	Toxicity Response Factor (T_r)	PERI Contribution
Ni	Woji	4.45	20	-2.15	0.2225	1	0.2225
	Okujagu	2.59	20	-2.78	0.1295	1	0.1295
	Elelenwo	2.38	20	-2.83	0.119	1	0.119
Cr	Woji	3.49	25	-2.2	0.1396	1	0.1396
	Okujagu	4.26	25	-1.96	0.1704	1	0.1704
	Elelenwo	<0.001	25	-∞	0	1	0
Fe	Woji	3492.93	2000	1.77	1.7465	1	1.7465
	Okujagu	3909.39	2000	1.67	1.9547	1	1.9547
	Elelenwo	736.16	2000	-1.43	0.3681	1	0.3681
Hg	Woji	<0.001	0.05	-∞	0	30	0
	Okujagu	<0.001	0.05	-∞	0	30	0
	Elelenwo	<0.001	0.05	-∞	0	30	0
Cd	Woji	0	0.1	-∞	0	5	0
	Okujagu	0	0.1	-∞	0	5	0
	Elelenwo	0	0.1	-∞	0	5	0
Cu	Woji	0.33	30	-3.22	0.011	5	0.055
	Okujagu	<0.001	30	-∞	0	5	0
	Elelenwo	<0.001	30	-∞	0	5	0
Pb	Woji	0	20	-∞	0	5	0
	Okujagu	0	20	-∞	0	5	0
	Elelenwo	0	20	-∞	0	5	0
Mn	Woji	10.98	1000	-3.68	0.01098	1	0.01098
	Okujagu	14.48	1000	-3.51	0.01448	1	0.01448
	Elelenwo	2.98	1000	-5.23	0.00298	1	0.00298

Conclusions and Recommendations

Cadmium (Cd) and Pb in the study areas were extremely enriched while I_geo revealed low contamination for Ni, Cr, and Fe, suggesting these metals are mainly from natural sources. The moderate contamination of Fe, indicating potential human activities influencing Fe levels. Mercury (Hg) poses the highest ecological risk, even though its concentration is low. Hg is known for its high toxicity and bioaccumulation potential. Ni, Cr, and Fe have much lower PERI values, implying minimal ecological risk

Recommendations

Regular monitoring is required, especially for Hg, to prevent bioaccumulation risks in aquatic life and humans. The potential sources of Fe contamination (e.g., industrial discharge, weathering of rocks) should be identified. Further ecological assessments should be conducted to evaluate long-term risks.

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