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Spatial distribution of organochlorine pesticides contamination in the galma river sediment, Nigeria

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ABSTRACT

This study investigated chlorinated pesticide contamination in Galma River sediments, Zaria, Nigeria, focusing on its spatial distribution and ecological risks. Despite global bans on many organochlorine pesticides (OCPs), residues persist in Nigerian rivers, posing significant environmental and public health threats. Sediment samples from 10 locations along the river were analyzed using gas chromatography-mass spectrometry (GC-MS), revealing widespread contamination by 18 OCPs. Among these, Hexachlorobenzene (HCB), α-HCH, and Heptachlor epoxide were found in the highest concentrations, with HCB ranging from 46.29 to 167.21 ng/g, α-HCH from 31.56 to 159.4 ng/g, and Heptachlor epoxide peaking at 142.03 ng/g. Ecological risk assessments identified major contamination hotspots at locations P5, P6, and P8, where Heptachlor epoxide concentrations exceeded safe limits, indicating severe risks to aquatic life. Historical pesticide use was evident in P10, where elevated levels of p, p'-DDD and p,p'-DDT were found. Clustering analysis revealed three distinct contamination patterns, highlighting areas requiring urgent remediation. This study highlights the need for better regulation, continuous monitoring, and targeted remediation efforts to mitigate the ongoing risks posed by pesticide contamination in northern Nigeria's agricultural regions.

1. Introduction

Modern agriculture has significantly benefited from the use of agricultural chemicals, which help manage pests, diseases, and weeds, thereby enhancing food quality and production [1]. However, the widespread and indiscriminate use of certain pesticides, particularly organochlorine pesticides (OCPs), has led to severe environmental and public health concerns [2]. Due to their resistance to degradation, OCPs, like dichlorodiphenyltrichloroethane (DDT),

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Hexachlorobenzene (HCB), and Lindane, persist in the environment for extended periods. These substances can accumulate in soils and sediments and enter aquatic ecosystems through bioaccumulation and biomagnification, posing significant threats to aquatic life and human health [3]. Due to their harmful nature, OCPs have been banned or heavily restricted in many countries, including Nigeria. However, their use continues in some regions due to farmers' reliance on these chemicals to boost agricultural yields, insufficient enforcement of regulations, and the availability of cheaper alternatives. In Nigeria, where agriculture is a major economic activity, pesticide use remains prevalent to maintain productivity. Consequently, OCP residues persist in various environmental compartments, particularly in water bodies near agricultural areas [4]. This issue is especially pronounced in rural areas where communities depend on these water sources for fishing and irrigation, raising concerns about long-term effects on biodiversity, water quality, and human health.

Most research on OCP contamination in Nigeria has focused on southern regions, particularly urban and industrialized areas with high pollution levels [4,5]. In contrast, northern Nigeria, where subsistence farming is widespread and pesticide use is common [6], has received less attention despite the potential for significant contamination due to agricultural runoff and improper pesticide disposal [7]. The Galma River in northern Nigeria, crucial for local agriculture and communities, is one such water body potentially at risk. The river supports extensive crop cultivation and is heavily used for irrigation, drinking water, and fishing, making its health vital to local socio-economic stability [8].

This study is the first to systematically assess OCP contamination in the sediments of the Galma River. Previous research has examined other rivers in Nigeria, but there is a notable gap in understanding the contamination in northern Nigeria and the Galma River specifically. By filling this gap, the study provides critical data for future risk assessments, policy development, and mitigation strategies to protect aquatic ecosystems and public health.

The study employed advanced Geographic Information Systems (GIS) for spatial analysis of contamination patterns and used the Risk Quotient (RQ) method for ecological risk assessment. This approach highlights contamination hotspots and assesses potential environmental hazards. Focusing on sediments is essential, as they serve as long-term pollutant sinks and can re-suspend contaminants into the water. The findings have broader implications for sediment management and pollution control in agricultural regions. In essence, this research offers new insights into OCP contamination in northern Nigeria, highlights the need for regulatory focus, and provides a foundation for future studies and policy-making efforts to mitigate pesticide pollution in rural and agricultural areas.

2. Material and Methods

2.1 Study area

The Galma River is a significant water body located in northern Nigeria, particularly within Kaduna State. It originates from the highlands of the Jos Plateau, flowing south-westwards through Zaria and its surrounding agricultural areas before eventually joining the Kaduna River. The river spans a substantial length, providing essential water resources for domestic, agricultural, and industrial uses in the region. It serves multiple purposes for the local population, including as a source of drinking water, irrigation, and fishing. The river's catchment area falls within the northeastern part of the Kaduna River basin. This agriculturally intensive region sees considerable crop cultivation on both sides of the riverbanks throughout the year.

Figure 1 shows the map of the study area, which involves ten strategically selected sampling points along the Galma River to comprehensively assess the occurrence and distribution of chlorinated pesticides in the river's sediments. These points span a range of geographic locations, providing a broad overview of the river's condition. These sampling points were carefully chosen to cover a diverse range of environmental conditions and land use practices along the Galma River.

Figure 2 presents the Land Use and Land Cover (LULC) classification for the Galma watershed, aimed at distinguishing different land use types

within the area. The classification categorizes the land into distinct classes [9]. This classification provides a comprehensive understanding of the region's landscape and its various uses.

2.2 Sample Collection

The sampling was conducted in January 2024, during the dry season. A sediment grab sampler was used to collect samples from ten locations along the length of the river, ranging from upstream (P1) to downstream (P10). Any extraneous materials, such as stones, leaves, and shells, were manually removed. The sediment samples were individually wrapped in labeled aluminum foil, stored in a cooler with ice, and transported to the laboratory, where they were refrigerated at 4°C.

2.3 Sample preparation, treatment and extraction of sediment

In the laboratory, sediment samples were first air-dried at ambient temperature, then pulverized using a pre-cleaned porcelain mortar and pestle and sieved to ensure uniform particle size. To remove residual moisture and enhance extraction efficiency, 10 g of the dried sediment was mixed with 10 g of anhydrous sodium sulfate. A solvent mixture of n-hexane and acetone in a 1:1 ratio was added to the sample in volumes of 20-30 mL. The Soxhlet extraction technique was used, where the sample was continuously refluxed for 4-6 hours, ensuring the efficient recovery of chlorinated pesticides. After extraction, the solvent layer was filtered through a combination of anhydrous sodium sulfate and glass wool to remove any particulate matter and moisture. The extracts are then concentrated using a rotary evaporator until reduced to about 1 mL.

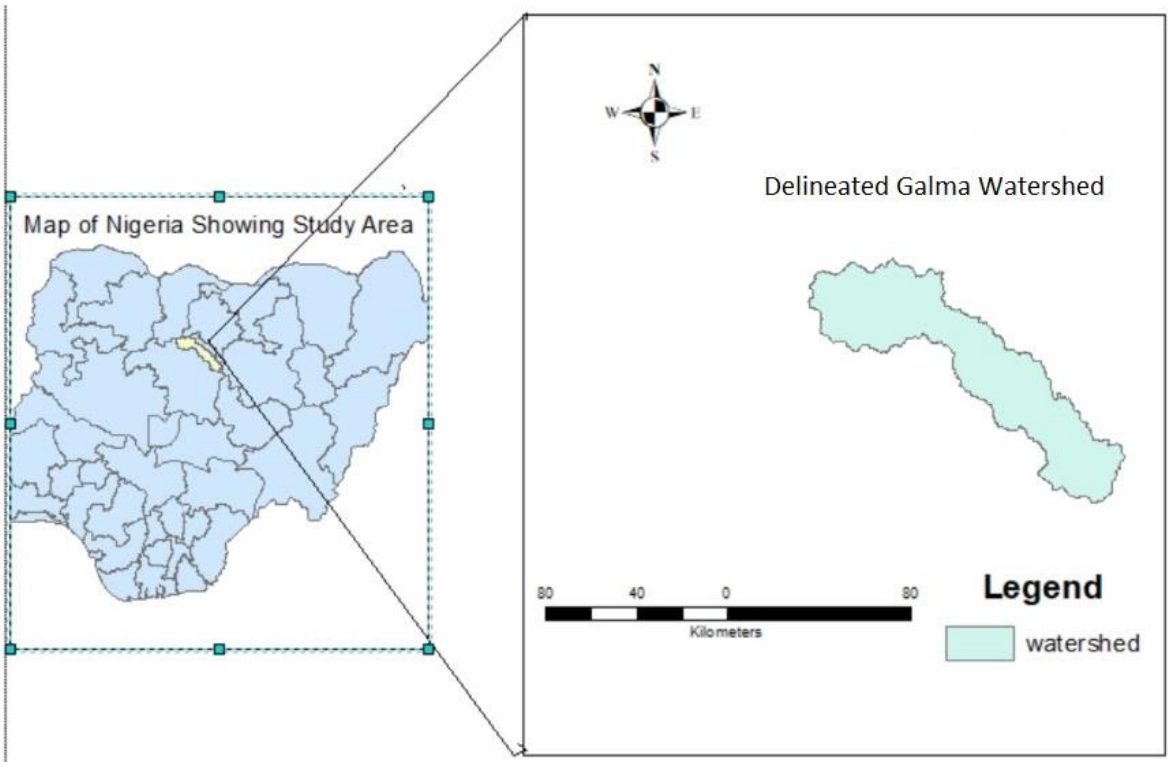

Fig. 1. Map of the study area.

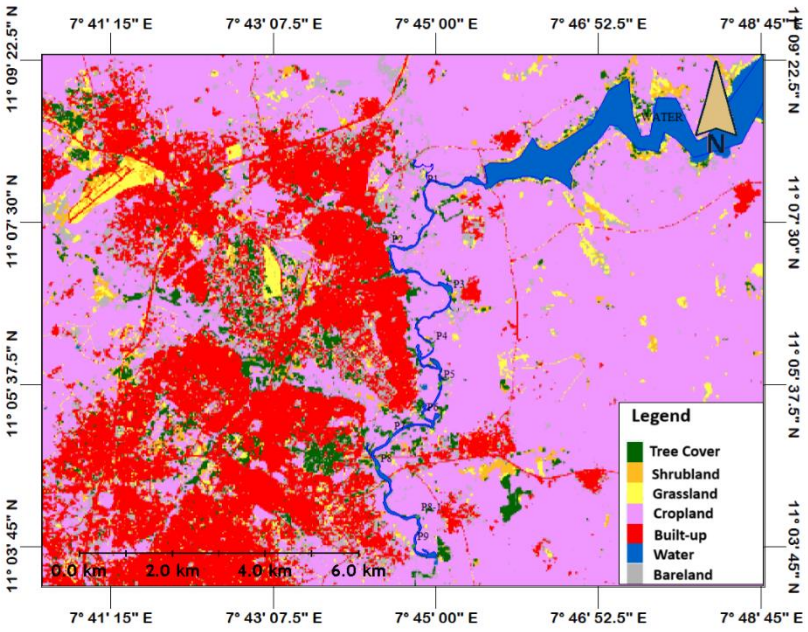

Fig. 2. Land use land cover map showing sampling locations.

Clean-up was achieved by passing the extract through a silica gel column with a hexane-acetone mixture to elute the pesticides. The eluates were further concentrated to 0.5mL for analysis. Finally, internal standards decafluorobiphenyl was added before the sample underwent gas chromatography (GC-MS) for specific identification and quantification of chlorinated pesticides. This method was based on EPA 3540B [10,11].

2.4 Analytical Determination

The procedure for analyzing organochlorine pesticides (OCPs) was carried out as described by [5] and [12]. In detail, the sediment samples were first dried using a vacuum freeze-drying method. A total of 50 g of homogenized river sediment was subjected to the Soxhlet extraction using a mixture of 200 ml of dichloromethane and acetone in a 1:1 (v/v) ratio for 72 hours. Afterward, the sample extract was concentrated to around 2 ml using a rotary evaporator. To purify the hexane extract, an alumina/silica gel glass column containing 1 cm of anhydrous sodium sulfate was employed. Fractions containing polychlorinated biphenyls (PCBs) and organochlorine pesticides (OCPs) were eluted using a dichloromethane/hexane mixture (3:7, v/v), and the collected solution was evaporated under a gentle nitrogen stream to a volume of 5 ml. The final residue was dissolved in 1 ml of hexane, sealed, and prepared for analysis by gas chromatography.

For the quantification of OCPs in sediment samples, a Hewlett-Packard 5890 Series II gas chromatograph equipped with an electron capture detector (ECD) was used. The chromatographic separation was carried out using a fused-silica capillary column (30 m × 0.32 mm × 0.52 µm). The GC conditions were as follows: the initial temperature was set from 70°C to 280°C at a rate of 5°C/min and held at 280°C for 20 minutes. The detector and injector temperatures were set at 300°C and 270°C, respectively. Helium was used as the carrier gas at a flow rate of 1.5 ml/min, with nitrogen serving as the makeup gas at a flow rate of 60 ml/min [12].

2.5 Quality Control

A robust Quality Assurance and Quality Control (QA/QC) procedure was followed to ensure accuracy in the analysis of the chlorinated pesticides [13,14]. In brief, an internal standard concentration of 100 µg/L was maintained across all samples to normalize analyte responses. The surrogate standard used was 2,4,5,6-tetrachloro-m-xylene, added at a concentration of 10 µg/L to each sample prior to extraction. This surrogate helped assess the extraction efficiency and matrix effects by ensuring that analyte recovery rates were within acceptable limits—75% to 125% for quantification and 65% to 135% for qualification. Instrument calibration was achieved using multi-level calibration curves, with correlation

coefficients (r^2) above 0.99 to ensure precision. Matrix spikes and replicate samples further validated the method, with any deviations in recovery triggering method adjustments. This QA/QC approach ensured that the analysis was both reliable and aligned with international standards. Sample preparation techniques were critically evaluated for their impact on quantitative outcomes, including limits of detection (LOD), sensitivity, and limit of quantification (LOQ). The method development phase was identified as pivotal in ensuring analytical accuracy, as differences in environmental conditions, equipment, and reference values can significantly influence data variability. Clear definitions of the limits of qualification and quantification were established to standardize measurements across analyses.

2.6 Data Analysis

Spatial distribution of chlorinated pesticides (OCPs) was analyzed using ArcGIS version 10.2. All statistical analyses were conducted using Jupyter Notebook with Python libraries, including NumPy, pandas, seaborn, and SciPy. Boxplots were generated to visualize the distribution and variability of contaminant concentrations across different sampling sites. A correlation matrix was used to assess relationships between organochlorine pesticide (OCP) concentrations and associated p-values calculated to determine statistical significance. Hierarchical clustering analysis was performed to identify potential groupings of sampling sites based on contamination patterns. The risk quotient (RQ) method assessed ecological risks to aquatic organisms, calculated using the measured concentrations of OCPs and their predicted no-effect concentrations (PNEC). This method provided insights into the potential hazards posed by OCPs in the river sediment. If RQ < 1, the risk is considered low or negligible. If RQ ≥ 1, it indicates a potential ecological risk, meaning further investigation or mitigation measures might be required.

3. Results and Discussion

3.1 Spatial Distribution of Chlorinated Pesticides

The analysis of sediment samples from various locations in Galma river revealed a widespread contamination by chlorinated pesticides. A total of eighteen compounds were detected across the sampling sites, with varying concentrations noted in each location as shown in Table 1.

HCB was consistently found in high levels across various sites, with P1 (167.21 ng/g) and P5 (156.73 ng/g) recording the most elevated concentrations, both significantly exceeding the effects range-median (ERM) threshold of 46 ng/g [15]. All ten sites exceeded the effects range-low (ERL) threshold of 1.58 ng/g, suggesting potential ecological risks. Conversely, its levels were relatively lower in P8 (46.29 ng/g) and P9 (56.41 ng/g), though still above the ERM threshold, hinting at possible local environmental influences that may be contributing to the lower retention of HCB in those regions [16]. The concentration of α-HCH fluctuated across different points, with the highest recorded value at P5 (92.75 ng/g), while it was not detected at P6 and P10. This trajectory indicated a possible accumulation process that could be associated with certain environmental or human-related activities. Trans-chlordane presents considerable fluctuations, featuring a notable spike at P9, which suggests a specific local factor responsible for the high concentration levels. Heptachlor was most prominent in P7, where it reached 132.46 ng/g, while P8 recorded a lower concentration of 81.45 ng/g. The highest Heptachlor level was observed at P7, indicating a localized peak at this point. Additionally, other compounds such as p,p'-DDD, p,p'-DDE, and p,p'-DDT showed significant peaks in P10, especially p,p'-DDD and p,p'-DDT, which display higher concentrations relative to other sites. This indicated that P10 served as a hub for these pesticides, potentially attributable to past pesticide usage or contamination incidents unique to that area.

3.2 OCP Concentration Patterns

Figure 3 illustrates the variability in organochlorine pesticide (OCP) concentrations across the ten locations along the Galma River, revealing notable spatial differences in contamination patterns.

Locations P3, P5, P9, and P10 displayed wide concentration ranges and multiple outliers, suggesting high variability and potential episodic pollution events

Table 1. Concentrations of OCPs detected across the 10 sampling sites

OCP (ng/g)	P1	P2	P3	P4	P5	P6	P7	P8	P9	P10
HCB	167.21	92.55	125.48	78.93	156.73	102.35	88.25	46.29	56.41	138.76
α-HCH	76.85	38.3	49.12	69.51	92.75	n.d	56.92	31.57	83.45	n.d
β-HCH	97.28	89.23	82.44	87.14	n.d	148.23	48.49	77.44	97.54	102.63
γ-HCH	142.57	63.36	113.46	73.85	83.16	71.85	93.31	132.76	103.66	85.41
δ-HCH	95.64	122.85	n.d	52.42	95.62	85.7	72.34	113.53	47.54	75.43
Heptachlor	48.62	79.5	85.22	92.14	47.69	46.82	132.46	81.45	124.74	56.44
Heptachlor epoxide	33.87	49.52	56.43	78.7	78.22	124.58	142.04	53.92	n.d	72.91
Oxychlordane	71.59	113.46	78.95	32.58	53.52	67.41	75.42	103.55	n.d	47.83
Trans-chlordane	97.54	145.02	94.53	48.46	68.13	84.54	91.53	124.06	158.23	101.33
Cis-chlordane	56.99	49.73	66.96	61.29	39.27	92.32	32.55	94.74	76.25	89.54
Cis-nonachlor	61.33	64.28	129.58	83.75	82.29	76.51	56.38	96.83	48.22	123.76
Trans-nonachlor	87.23	98.83	72.16	131.57	76.59	84.13	113.46	71.52	31.57	108.75
α-endosulfan	100.25	84.24	114.52	82.31	52.42	97.83	82.31	46.82	87.15	75.25
o,p'-DDE	58.71	36.49	66.71	162.39	82.13	76.42	99.43	118.23	48.72	86.58
p,p'-DDE	60.57	51.63	34.66	47.61	50.42	n.d	42.57	40.28	110.25	98.72
o,p'-DDD	112.37	62.45	157.83	n.d	129.46	102.85	63.22	51.47	54.64	46.83
p,p'-DDD	49.75	85.43	90.24	63.42	114.33	56.83	86.25	78.12	98.74	131.23
p,p'-DDT	79.15	98.72	76.41	93.25	46.73	113.55	92.75	89.14	76.92	114.83
Total OCP	1497.52	1425.59	1494.7	1339.32	1349.46	1431.92	1469.68	1451.72	1304.03	1556.23

Note: OCP: Organochlorine pesticides, HCB: Hexachlorobenzene, HCH: Hexachlorocyclohexane (α, β, γ, δ refer to isomers), DDE: Dichlorodiphenyldichloroethylene, DDD: Dichlorodiphenyldichloroethane, DDT: Dichlorodiphenyltrichloroethane, n.d: Not detected, P1–P10: Sampling points

. These fluctuations might be associated with periodic agricultural runoff, industrial discharges, or runoff inputs that introduce contaminants into the river system [17]. In contrast, P2 and P4 exhibited narrower interquartile ranges (IQRs) with fewer outliers, indicating more stable contamination levels. The presence of outliers at P9 and P4, however, suggested that localized pollution spikes occurred, potentially due to intermittent agricultural activities or sediment disturbances. The highest median concentrations were observed at P9 and P10, suggesting that these locations might experience sustained OCP inputs, posing persistent ecological risks despite the less extreme variability [18].

The occurrence of outliers in multiple locations highlights the sporadic nature of contamination, which may pose acute risks to aquatic life [17]. These contamination spikes could result from intensive pesticide applications or environmental factors such as rainfall-induced runoff, which redistributes pollutants within the river system. The patterns observed in the boxplot suggested that while some locations experienced fluctuating contamination, others exhibited sustained OCP levels that still posed ecological concerns. This variability showed the necessity for targeted monitoring and remediation efforts, particularly in hotspot locations with high median concentrations (P9, P10) and frequent outliers (P3, P5, P9, P4), to mitigate both long-term and episodic contamination risks.

Additionally, clustering was employed to analyze the concentration patterns of organochlorine pesticides (OCPs) across the different locations. Clustering is a statistical method used to group data points based on similarities, allowing patterns or trends to emerge within complex datasets. The approach helps identify potential sources, environmental behaviours, and associated risks, offering insights for targeted monitoring and remediation efforts. Three distinct clusters were identified (Figure 4) based on normalized OCP concentrations. Cluster 2, including HCB and p,p'-

DDD, represented OCPs with consistently high concentrations, likely due to widespread historical use or strong resistance to environmental degradation. These OCPs posed significant risks of bioaccumulation and trophic transfer, making them priority targets for remediation. Cluster 0 included OCPs with moderate and stable concentrations, suggesting legacy pollution or diffuse sources from past agricultural practices. Most of the OCPs (14) fell within this cluster.

While less critical than Cluster 2, these OCPs still warrant regular monitoring to prevent long-term ecological impacts. Cluster 1 encompassed the OCPs with low or sporadic concentrations, possibly linked to reduced usage, faster degradation rates, or isolated contamination sources. Although these posed a lower immediate risk, their sporadic presence usually indicates the need for careful observation in vulnerable ecosystems.

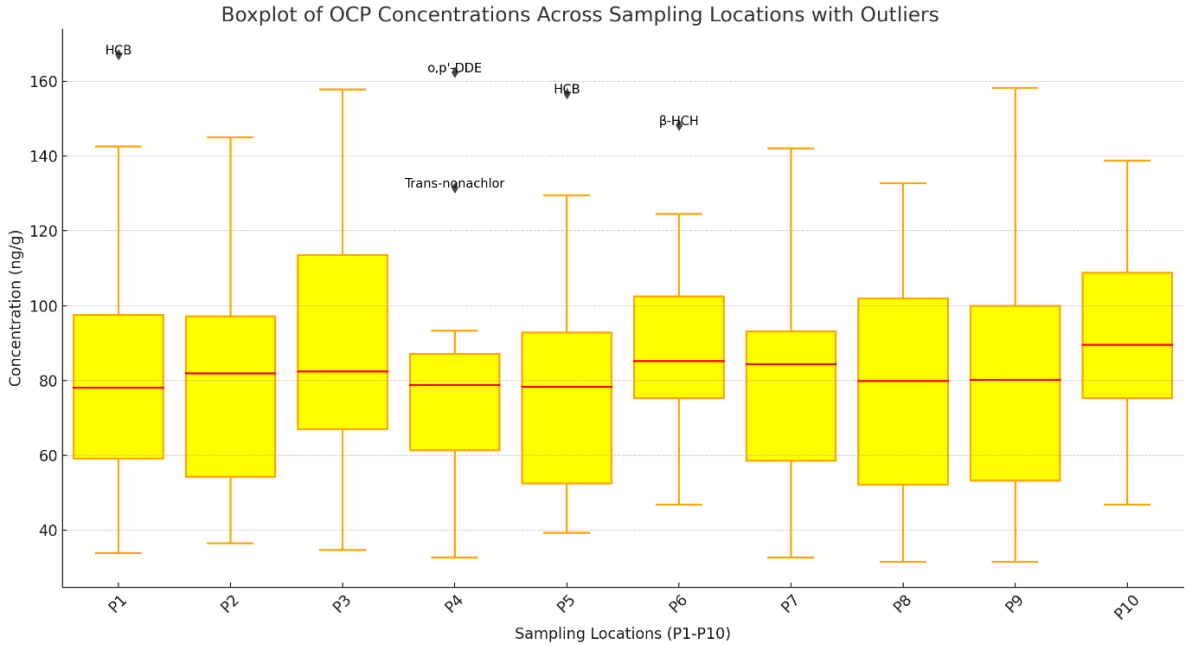

Fig. 3. Boxplot diagram of OCP concentrations across locations.

Figure 5 revealed several important trends in the correlations between OCP levels across different locations. High positive correlations were observed in some areas, indicating that OCP levels tend to rise or fall together. For instance, P5 and P6 exhibited a strong positive correlation, suggesting similar contamination patterns or shared sources affecting both sites. Similarly, P2 and P4 showed a notable correlation, which might point to a common contamination source or similar environmental factors influencing both areas. On the other hand, low or negative correlations were evident in locations such as P3 and P10, where OCP levels were weakly or inversely correlated with other locations. This suggested that the factors impacting OCP concentrations in these areas differed significantly from those influencing other sites. Clusters of correlated locations also emerged from the analysis. For example, P1, P5, and P6 formed a distinct cluster of highly inter-correlated sites, suggesting that these areas might be part of the same environmental system or were exposed to contaminants from a shared source. Locations that exhibit high correlations might require collective management strategies if they share contamination sources or similar risk profiles. Conversely, locations with low correlations might need more individualized approaches based on their unique contamination patterns. The analysis of p-values for the correlations between OCP levels across locations (Figure 6) revealed important insights regarding the statistical significance of these relationships.

Locations with low p-values (< 0.05) indicated statistically significant correlations, meaning that the relationship between OCP levels at these sites was unlikely due to random chance. In contrast, high p-values (> 0.05) suggested that any observed correlation might be coincidental, driven by random variation rather than a meaningful connection. For example, P1 and P5 exhibited a p-value of 0.055561, which was close to the significance threshold, indicating a possible correlation.

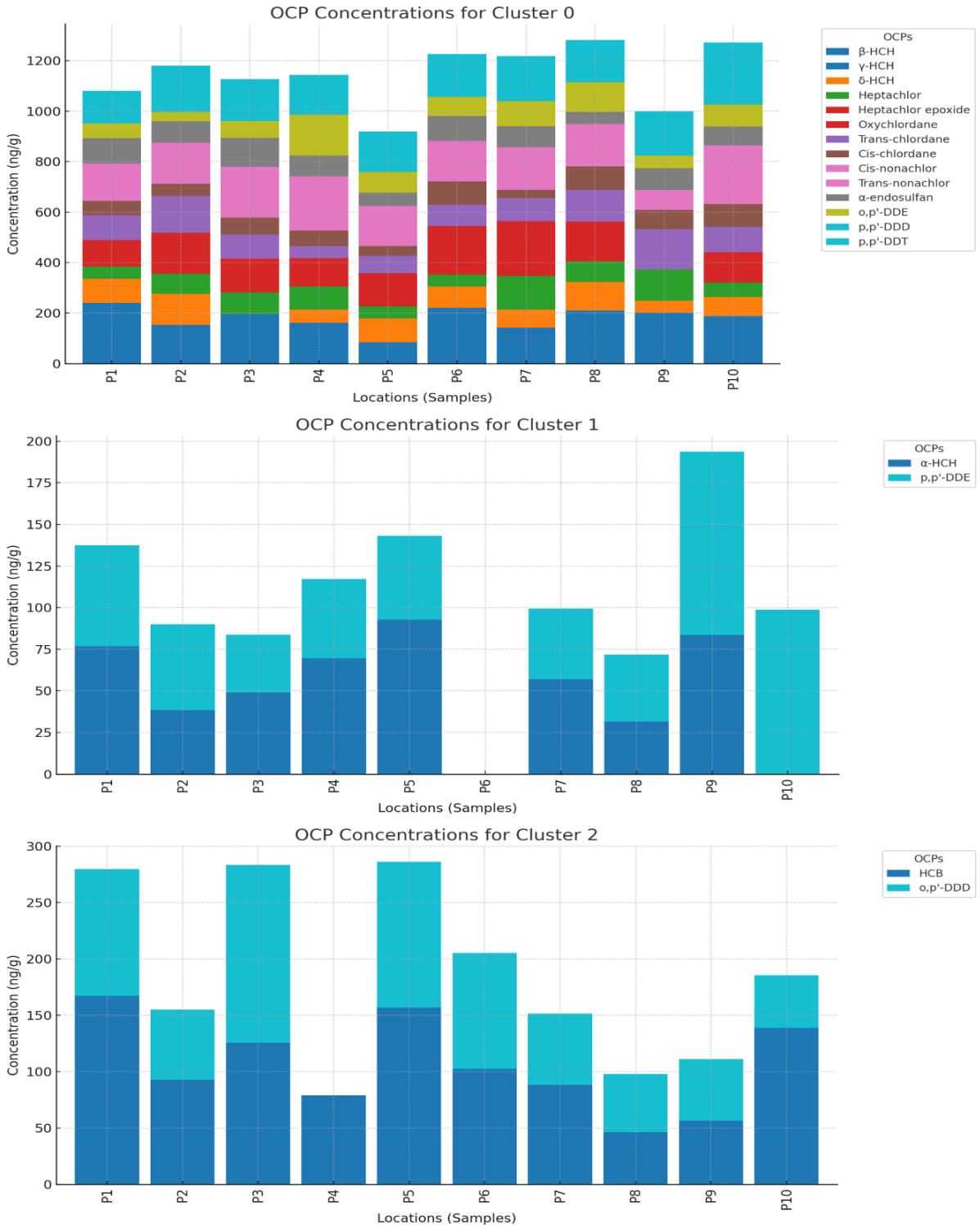

Fig. 4. Grouping of OCP concentrations into clusters.

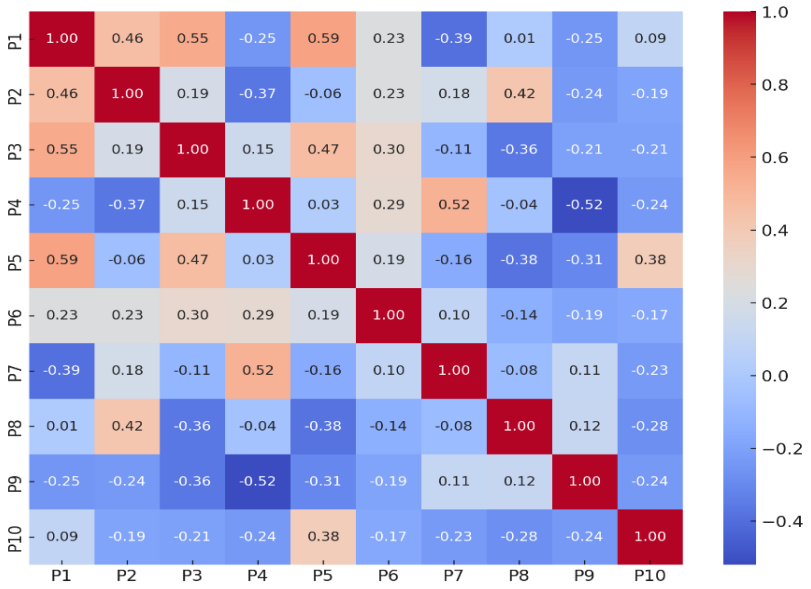

Fig. 5. Correlation matrix of OCP concentrations across locations.

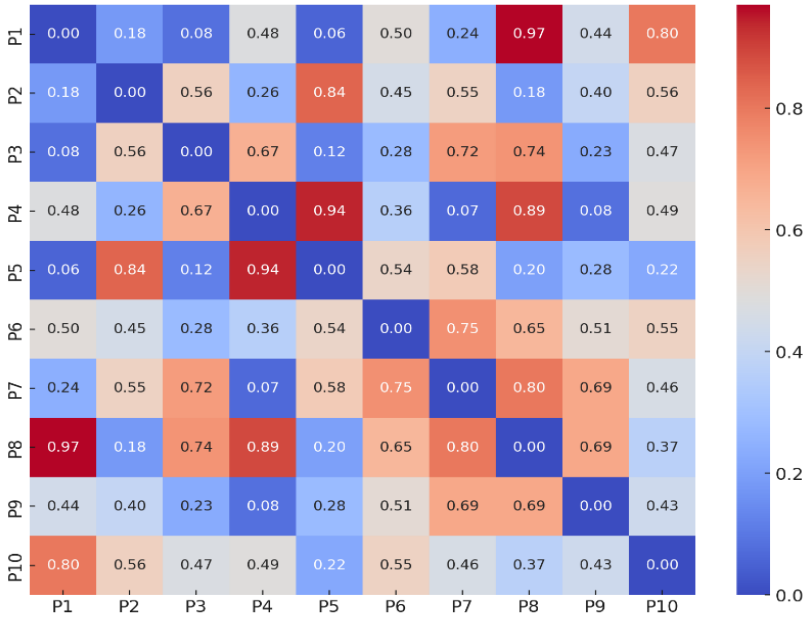

Fig. 6. P-values for correlation between locations.

Similarly, P4 and P7 showed a slightly higher p-value (0.066971), suggesting a potential, though not definitive, relationship between OCP levels in these areas. While some locations, like P1 and P3, had p-values approaching significance (0.081071), the majority of location pairs did not exhibit strong statistical associations. In general, most correlations between locations appeared to be statistically insignificant, as indicated by p-values exceeding the 0.05 threshold. This suggested that OCP levels at most locations were not strongly related to one another, implying that contamination might be driven by localized sources or independent environmental factors. This was similar to the findings by [19] who also demonstrated that the spatial distribution of OCPs varied significantly across sites, driven by differing input sources and physicochemical interactions rather than uniform contamination. However, the few location pairs with p-values near significance, such as those between P1 and P5 or P4 and P7, might indicate areas where OCP levels were more closely linked, potentially due to shared contamination sources or similar environmental

conditions. These trends suggested that while broad, uniform contamination was unlikely, targeted investigation into near-significant correlations could uncover common factors influencing OCP distribution. Consequently, environmental management efforts might benefit from site-specific interventions, with a focus on understanding localized contamination sources to guide remediation strategies.

3.3 Ecological Risk Assessment

An ecological risk assessment was performed using the risk quotient (RQ) method, which involved comparing the detected concentrations of organochlorine pesticides (OCPs) with their predicted no-effect concentrations (PNEC). This was performed by calculating the RQ for the detected OCP based on the equation: RQ = measured concentration/PNEC [20] PNEC values were obtained from the Norman Ecotoxicology Database (see http://www.norman-network.com/nds/ecotox/lowestPnecsIndex.php) which contains PNEC values for over 40,000 substances, mostly derived from QSAR toxicity data for four groups: protists, fish, algae, and Daphnia. QSAR-based PNECs are calculated by dividing the lowest no observable effect concentration (NOEC) from these groups by a factor of 1000. For this study, PNEC values were downloaded from the NORMAN Network on October 20, 2024, as shown in Table 2.

In the context of this ecological risk assessment, the findings from the calculated Risk Quotients (RQs) across multiple organochlorine pesticides (OCPs) and locations (Figure 7) revealed several concerning patterns regarding potential environmental risks. Chemicals like α-HCH, β-HCH, γ-HCH, δ-HCH, Heptachlor and Heptachlor epoxide consistently exhibited extremely high RQ values, often exceeding thresholds by several orders of magnitude (RQ > 1,000). This indicated an alarming level of environmental risk for these compounds at most locations. These high values suggested potential bioaccumulation or persistence in the environment, which could have detrimental effects on local ecosystems [21]. The highest RQ values were consistently observed for Heptachlor epoxide across multiple locations, particularly in P5, P6, and P8. These extreme values indicated that the concentration of Heptachlor epoxide significantly exceeded safe environmental limits, posing a severe toxicological risk to both aquatic and terrestrial ecosystems. Its persistence and high toxicity make it a critical concern, necessitating immediate mitigation efforts in these locations to reduce potential long-term damage. In comparison, Trans-nonachlor and Cis-nonachlor showed moderately elevated RQ values, often ranging between 10 and 30. While these chemicals did not present the same acute threat as Heptachlor epoxide, they still posed a significant ecological risk, especially to soil and sediment-dwelling organisms.

Table 2. PNEC values for target OCP chemicals

OCP	HCB	α-HCH	β-HCH	γ-HCH	δ-HCH	Heptachlor	Heptachlor epoxide	Oxychlordane	Trans-chlordane
PNEC (ng/g)	9.38	0.001	0.001	0.001	0.001	0.0025	0.000088	6.97	6.97
OCP	Cis-chlordane	Cis-nonachlor	Trans-nonachlor	α-endosulfan	o,p'-DDE	p,p'-DDE	o,p'-DDD	p,p'-DDD	p,p'-DDT
PNEC (ng/g)	6.97	3.82	3.82	3.19	11.5	2.2	14.5	2	1

Note. PNEC: predicted no-effect concentrations

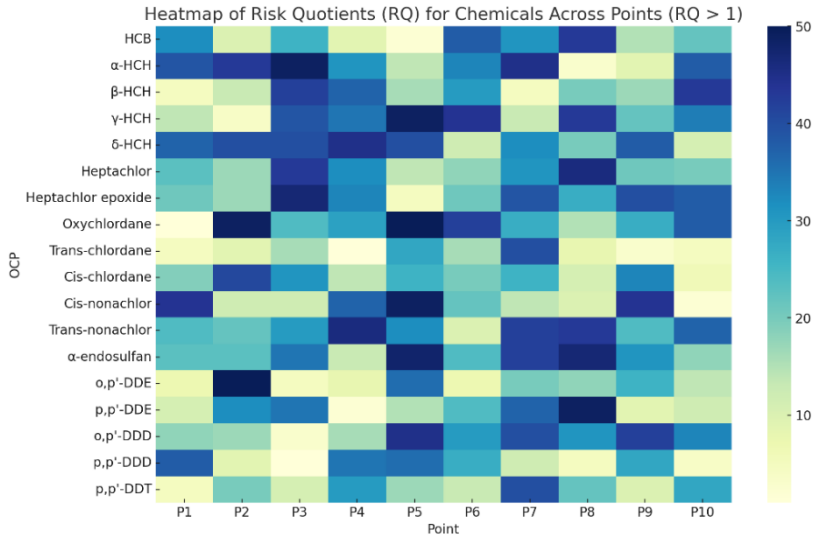

Fig. 7. Heatmap of Risk Quotients (RQ) for OCPs across locations.

Locations P5 and P6 stood out as contamination hotspots, with consistently high RQ values for multiple chemicals, including Heptachlor epoxide, γ-HCH, and Trans-nonachlor. This concentration of high-risk chemicals indicated a compounded environmental burden that could result in long-term ecological damage if not addressed. P10 also exhibited significantly elevated RQ values for p,p'-DDD and p,p'-DDT, both of which exceeded 100. Given the legacy of DDT usage and the persistence of its toxic metabolites in the environment, targeted remediation might be necessary in this area to minimize exposure risks. On the other hand, P1, P2, and P4 generally showed lower RQ values across most chemicals, though occasional spikes in α-HCH and Heptachlor epoxide highlighted areas of concern. While these locations might not exhibit the same level of contamination as others, the presence of certain high-risk chemicals highlighted the potential for localized ecological risks, especially given the persistent and mobile nature of these OCPs.

3.4 Correlation with Land Use

The study also identified a strong correlation between land use types and pesticide concentrations in sediments. Figure 2 shows a land use land cover map displaying a description of the major land use types by location.

***Pristine and Low-Impact Areas*:** P1, located in the upstream reaches of the Galma River with minimal human disturbance, served as a baseline for pesticide contamination. Despite its relatively pristine condition, the concentration of HCB (167.21 ng/g) remained notably high (Figure 8). This could be due to atmospheric deposition or long-range transport from surrounding areas. However, pesticide levels, in general, were lower than those observed in more agriculturally or industrially impacted sites.

Agricultural Impact: Sites P2 and P6, located in agricultural zones, showed moderate to high pesticide concentrations, particularly for compounds like HCB, α-HCH, and Heptachlor epoxide. P2, slightly downstream from P1 and influenced by small-scale farming, showed noticeable increases in HCB (92.55 ng/g) and α-HCH (38.30 ng/g), reflecting runoff from agricultural activities. P6, located in a predominantly agricultural area, showed significantly higher concentrations of multiple OCPs, particularly Heptachlor epoxide (124.58 ng/g), which was indicative of the intense pesticide usage in the region.

Intensive Agriculture and Tributary Impact: At P3, pesticide concentrations were high across multiple OCPs, including HCB (125.48 ng/g) and α-HCH (49.12 ng/g). The tributary likely acted as a conduit for pesticide runoff, amplifying the contamination in the river.

Semi-Rural and Mixed Land Use: P4, a semi-rural site influenced by both agriculture and small-scale industrial runoff, displayed moderate contamination levels. The OCP concentrations here were relatively balanced, with HCB (78.93 ng/g) and α-HCH (69.51 ng/g) showing notable levels.

Similarly, P9, a mixed land-use site, exhibited contamination from both residential and agricultural sources, particularly in the form of p,p'-DDD (98.74 ng/g) and p,p'-DDT (114.83 ng/g), suggesting a cumulative impact from both agricultural runoff and residential waste.

Urban and Industrial Areas: P5, located near a densely populated village, showed contamination from both domestic waste and agricultural runoff. The concentrations of Heptachlor epoxide (78.22 ng/g) and o,p'-DDD (129.46 ng/g) were significant, reflecting the compounded pollution from urban activities. P8, located near an industrial zone, showed contamination from industrial effluents. While agricultural residues contributed, industrial activities seemed to play a more dominant role at this site, as seen with high levels of compounds like cis-nonachlor (96.83 ng/g) and trans-chlordane (124.06 ng/g).

***Downstream Accumulation*:** Finally, P10, located at the confluence of the Galma and Kaduna Rivers, reflected the cumulative impact of all upstream activities. As expected, pesticide concentrations were among the highest, particularly for legacy pesticides like p,p'-DDD (131.23 ng/g) and p,p'-DDT (114.83 ng/g). This suggested long-term contamination from both agricultural and industrial sources that had accumulated downstream.

The correlation between land use and OCP concentrations in the Galma River showed the critical impact of agricultural, industrial, and urban activities on river sediment quality. Sites with intensive agriculture and industrial activities exhibited the highest pesticide levels, while more pristine or less disturbed areas showed comparatively lower contamination. These findings highlight the need for targeted environmental management, focusing on reducing pesticide runoff and improving waste management practices, particularly in agricultural and industrial zones.

3.5 Comparison with Other studies

The findings of this study on chlorinated pesticide contamination in the sediments of the Galma River were consistent with previous research conducted in various regions of Nigeria and globally, although some notable differences in pesticide levels and contamination patterns could be observed based on local land use, agricultural practices, and environmental conditions.

***Comparison with Studies in Southern Nigeria*:** Several studies conducted in southern Nigeria, particularly around urbanized and industrial areas, have documented similar trends of organochlorine pesticide (OCP) contamination. For instance, [22] and [23] reported high concentrations of OCPs, particularly HCB and DDT metabolites, in river sediments, similar to the patterns observed in the Galma River. Like our study, these studies found that downstream locations, especially near industrial and agricultural zones, exhibited significantly higher pesticide levels. However, the concentrations reported in southern Nigeria tended to be higher in urbanized areas, reflecting the influence of industrial effluents, which were less prevalent in northern Nigeria's rural agricultural zones.

***Comparison with Global Studies on Agricultural Runoff*:** Globally, studies from regions with intensive agriculture have also documented high levels of OCPs in river sediments. Research in Southeast Asia [24,25] and South America [26] has shown similar contamination profiles, with compounds like HCB, α-HCH, and β-HCH consistently appearing as major pollutants in river systems affected by agricultural runoff. For example, the study conducted in the Dagu Drainage River reported a maximum HCB concentration of 1,315 ng/g, with an average of 240 ng/g across the river [27]. In addition, the study by [28] in the Lum Stream agricultural region found HCB concentrations in sediments ranging from 0.032 to 0.631 ppm (32 to 631 ng/g) even higher than those observed in our study. This highlights the global persistence of OCPs, particularly in regions with extensive pesticide use, and reinforces the fact that agricultural runoff is a significant contributor to sediment contamination.

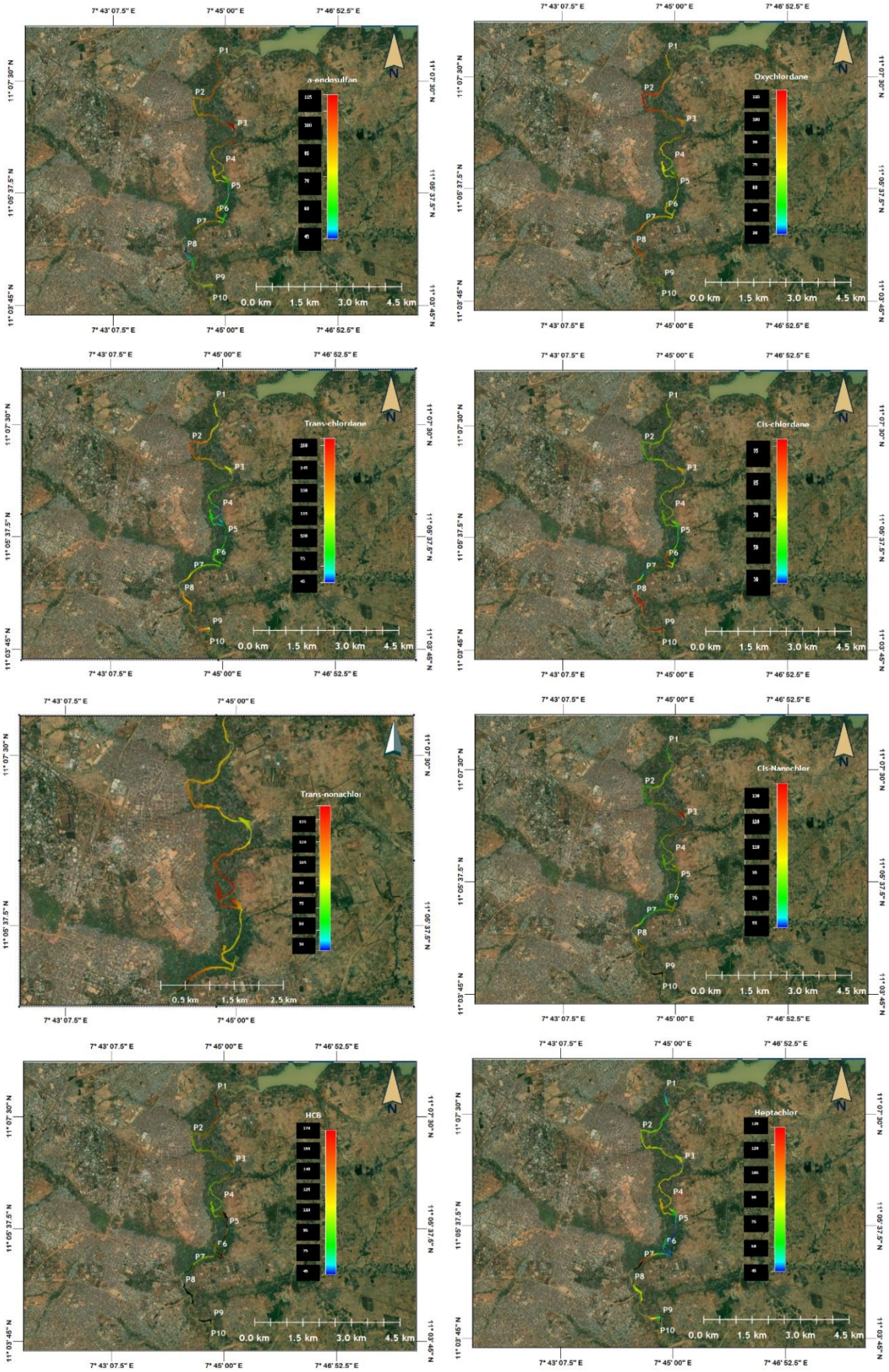

Fig. 8. Maps showing the spatial distribution of selected OCPs along the Galma river.

Legacy Pesticide Use in Rural and Agricultural Zones: A significant finding of this study was the persistence of legacy pesticides, such as p,p'-DDT and its metabolites, in the sediments, particularly at P10, where concentrations were among the highest (e.g., p,p'-DDD: 131.23 ng/g). This was consistent with the findings of [4], who documented the continued presence of DDT and its metabolites in southern Nigeria, even decades after their ban. Similar observations have been made in other African regions, including East Africa, where DDT residues are still detected in agricultural and aquatic environments despite bans on their use [29].

Risk Assessment Approaches: The use of the Risk Quotient (RQ) method for ecological risk assessment in this study had parallels with other global studies. For example, studies conducted by [30] and [31] used RQ analyses to assess the ecological risks posed by pesticide contamination in river sediments in agricultural regions. Like our findings, their studies identified hotspots where pesticide concentrations significantly exceeded safe ecological thresholds, especially for compounds like HCB, which was highly toxic and persistent in the environment. The elevated RQ values for these compounds in P5, P6, and P8 indicated similar patterns of ecological risks as observed in other agricultural river systems, where pesticide use remained high despite regulatory restrictions.

Differences in Contamination Levels Between Regions: A notable difference between this study and some other regional studies is the generally lower overall pesticide concentrations in northern Nigeria. While southern Nigeria's rivers are more heavily impacted by urbanization and industrial discharges, northern Nigeria's rivers, like the Galma River, show moderate contamination levels primarily linked to agricultural runoff. This is in line with research from the Sahel region [32], which found that agricultural activities, particularly those related to pesticide-intensive crops, contributed significantly to pesticide levels in rural water bodies, but urban-related contamination was less pronounced.

In summary, the contamination patterns observed in the Galma River sediments are broadly consistent with global and regional studies on pesticide contamination, particularly in agricultural zones. However, the extent of contamination in northern Nigeria appears to be influenced more by agricultural runoff than by urban or industrial sources, distinguishing it from findings in more industrialized regions of the country. The persistence of legacy pesticides like DDT highlights the long-term environmental impact of historical pesticide use, while the elevated ecological risks identified in certain hotspots point to ongoing threats to aquatic ecosystems.

4. Conclusions

This study highlights significant contamination of the Galma River sediments by chlorinated pesticides, with widespread presence of 18 OCPs, including high concentrations of HCB, α-HCH, and Heptachlor epoxide. Locations P5, P6, and P8 were identified as major contamination hotspots, where concentrations of Heptachlor epoxide exceeded safe ecological thresholds, posing serious risks to aquatic organisms. Notably, P10 showed elevated levels of legacy pesticides like p,p'-DDD and p,p'-DDT, indicating historical pesticide use that continues to affect the river's health. The clustering analysis revealed distinct contamination patterns, with some areas showing consistently high levels of certain OCPs while others exhibited sporadic spikes. These findings show the complexity of pesticide contamination in agricultural regions, where both long-term and episodic pollution events contribute to environmental hazards. The ecological risk assessment, particularly for Heptachlor epoxide, emphasizes the need for immediate intervention in hotspots to mitigate the potential long-term damage to local ecosystems. This research calls for stronger regulatory measures, improved monitoring, and targeted remediation efforts to reduce the persistence and impact of these hazardous chemicals in the river, safeguarding both the environment and public health in northern Nigeria's agricultural regions.

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