Journal of Advances in Chemical Sciences
ISSN 3121-6730
Volume 3, Number 1, 2026

Assessment of Organochlorine Pesticide Contamination in Ogun River Tributary, Ibadan, Nigeria

[1]Adegun, A. O., [1]Okesesan, J. O., [1]Amoo, P. O., [1]Adewuyi, G.O., [2]Ayorinde, G.O., [3]Akande, O.O. and [4]Adediran K.O.

[1]Department of Chemistry, University of Ibadan, Ibadan, Nigeria

[2]Department of Chemical Sciences, Kola Daisi University, Ibadan, Nigeria

[3]Department of Biological Sciences, Environmental Sciences and Toxicology Unit, Lead City University, Ibadan, Nigeria

[4]Department of Biological Sciences, Lead City University, Ibadan, Nigeria

*Corresponding Author: ayodeji2k15@gmail.com

Received 27th May, 2026 **Accepted** 5th June, 2026, **Published online** 12th June, 2026

ABSTRACT

This study aimed to validate an analytical method and apply it to the determination of organochlorine pesticide (OCP) residues in water and sediment samples collected from an Ogun River tributary (Odo Ona River), Ibadan, Nigeria. Surface water and sediment samples were collected, extracted, cleaned up, and analyzed using gas chromatography–mass spectrometry (GC–MS). Method validation was performed by evaluating linearity, recovery, limits of detection (LOD), and limits of quantification (LOQ). The developed method showed satisfactory analytical performance with coefficient of determination (R^2) ranging from 0.9572 to 1.0000. Percentage recoveries ranged from 72% to 105%, indicating acceptable accuracy. The LOD values ranged from 0.0191 µg L^{-1} to 7.5387 µg L^{-1}, while the LOQ values ranged from 0.0579 µg L^{-1} to 22.8446 µg L^{-1}. Application of the validated method to environmental samples revealed total OCP concentrations of 12.28 µg L^{-1}, 1.39 µg L^{-1}, and 0.90 µg kg^{-1} in upstream water, downstream water, and sediment samples, respectively. Endosulfan ether was the predominant residue detected, particularly in upstream water samples. The study demonstrated that the validated method is suitable for monitoring OCP residues in aquatic environments. The concentrations detected exceeded recommended guideline values, indicating potential environmental contamination and the need for continuous monitoring and strengthened regulatory enforcement to protect aquatic ecosystems and public health.

Keywords: Organochlorine pesticides, tributaries, validation, sensitivity, environmental monitoring.

Introduction

Organochlorine pesticides (OCPs) are a group of chlorinated compounds that were extensively used worldwide for agricultural and public health purposes because of their effectiveness, low cost, and broad-spectrum activity against insect pests [1–3]. OCPs belong to the class of persistent organic pollutants (POPs), which are characterized by their resistance to environmental degradation, long-range transport potential, and tendency to bioaccumulate in living organisms [4,5]. Although the production and use of many OCPs have been restricted or banned under the Stockholm Convention, their persistence in environmental matrices and continued application in some developing countries remain important environmental concerns [6–9]. OCP residues can enter aquatic ecosystems through agricultural runoff, atmospheric deposition, industrial discharges, and improper disposal practices, leading to contamination of surface waters and sediments [10–14].

The presence of OCPs in the environment has attracted considerable attention because of their potential adverse effects on both ecosystems and human health. Exposure to these compounds has been associated with respiratory disorders, skin irritation, headaches, nausea, and convulsions, while chronic exposure may result in endocrine disruption, reproductive abnormalities, developmental disorders, neurological impairment, and increased cancer risk [15–17]. Previous studies have also reported that exposure to Dieldrin during pregnancy may interfere with hormonal regulation and fetal development, whereas Chlordane has been linked to diabetes, respiratory infections, migraines, and neurological disorders [18–20]. Due to their lipophilic nature, OCPs accumulate in aquatic organisms and may be transferred through the food chain, thereby increasing the risk of exposure to humans and wildlife [21–26].

Many OCPs are among the so-called “dirty dozen,” a group of highly toxic and environmentally persistent chemicals initially targeted for elimination under the Stockholm Convention [27]. Despite international regulations, residues of these compounds are still detected in environmental samples across many developing countries, including Nigeria, where inadequate enforcement of pesticide regulations and the possible availability of banned products under different trade names may contribute to their continued occurrence in the environment [28,29].

Several studies have reported the occurrence of OCP residues in rivers, reservoirs, sediments, fish, and agricultural soils in different parts of Nigeria [9,10,21]. However, information regarding the occurrence, distribution, and contamination status of OCPs in Ogun River tributaries, particularly Odo Ona River in Ibadan, remains limited.

Consequently, there is insufficient information on the extent of contamination and the potential environmental risks associated with these pesticides in this important water resource. Addressing this knowledge gap is essential because the river serves domestic, agricultural, and economic functions for surrounding communities.

Therefore, this study was undertaken to determine the occurrence and concentration of organochlorine pesticide residues in water and sediment samples collected from Odo Ona River, a tributary of the Ogun River. The specific objectives were to identify possible sources of OCP contamination within the study area, quantify their concentrations in surface water and sediment samples, validate the analytical method for their determinations, and compare the measured concentrations with relevant national and international guideline values. The findings of this study will provide valuable information on the contamination status of OCPs in the study area and contribute to ongoing efforts aimed at protecting aquatic ecosystems and public health. The structures of some of the investigated OCPs are shown in Figure 1.

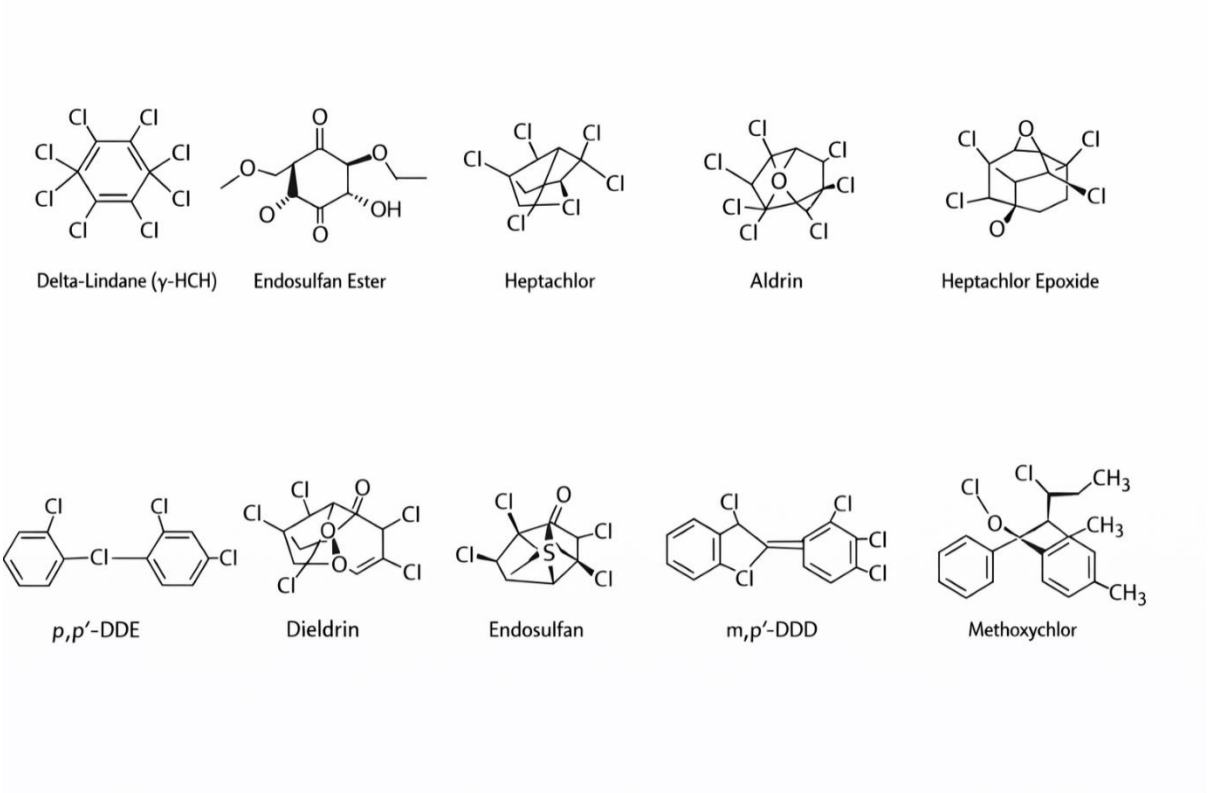

Figure 1: Structures of investigated organochlorine pesticides (OCPs)

Materials and Methods

Study Area

The study area is located along a tributary of the Ogun River at Idi-Ayunre, near the Cocoa Research Institute of Nigeria (CRIN), Ibadan, Southwestern Nigeria. The area lies approximately at latitude 07°29′16″ N and longitude 03°54′44″ E.

Idi-Ayunre is situated within the humid tropical zone characterized by distinct wet and dry seasons, with the wet season typically occurring between April to October, while the dry season extends from November to March. The region experiences relatively high annual rainfall and moderate temperatures, which favour agricultural activities. The Ogun River and its tributaries serve as important sources of water for domestic use, irrigation, and other agricultural purposes in the surrounding communities. The area surrounding the sampling sites is largely dominated by agricultural activities, particularly cocoa cultivation and experimental farming conducted by the Cocoa Research Institute of Nigeria. As part of routine agricultural management practices, pesticides are commonly applied to protect cocoa plants and other crops from insect pests and diseases that may reduce crop yield and quality.

Runoff from agricultural fields, especially during rainfall events, can transport pesticide residues into nearby water bodies and sediments. Consequently, tributaries of the Ogun River in this area may serve as potential sinks for pesticide contaminants originating from agricultural activities. In addition, human activities such as farming, irrigation, and occasional domestic uses of the river may contribute to the introduction of various contaminants into the aquatic environment. Given the agricultural importance of the area and the proximity of the river to pesticide application zones, the study area provides a suitable environment for assessing the occurrence and distribution of organochlorine pesticide residues in water and sediment matrices.

Collection, Handling and Storage of Surface Water Samples

Twenty-five surface water samples were collected by grab sampling from upstream and downstream locations of Odo Ona River using a 2.5 litres Winchester amber bottles. The sampling was performed at a surface depth. The surface water samples were collected according to the procedure described by Adegun *et al.* [10]. To prevent the introduction of air into the samples, the bottles were filled to the brim. This measure ensured that the collected water samples were free from air bubbles, which could potentially affect the accuracy of subsequent analyses. Prior to collecting the water samples, 5 mL of 1 mol dm^{-3} HCl was added. This step was taken to adjust the pH of the samples and prevent degradation.[11]. Upon arrival at the laboratory, the

water samples were stored in a refrigerator set at 4°C until the day of extraction. This controlled storage environment helped maintain the stability of the samples until further analysis [30].

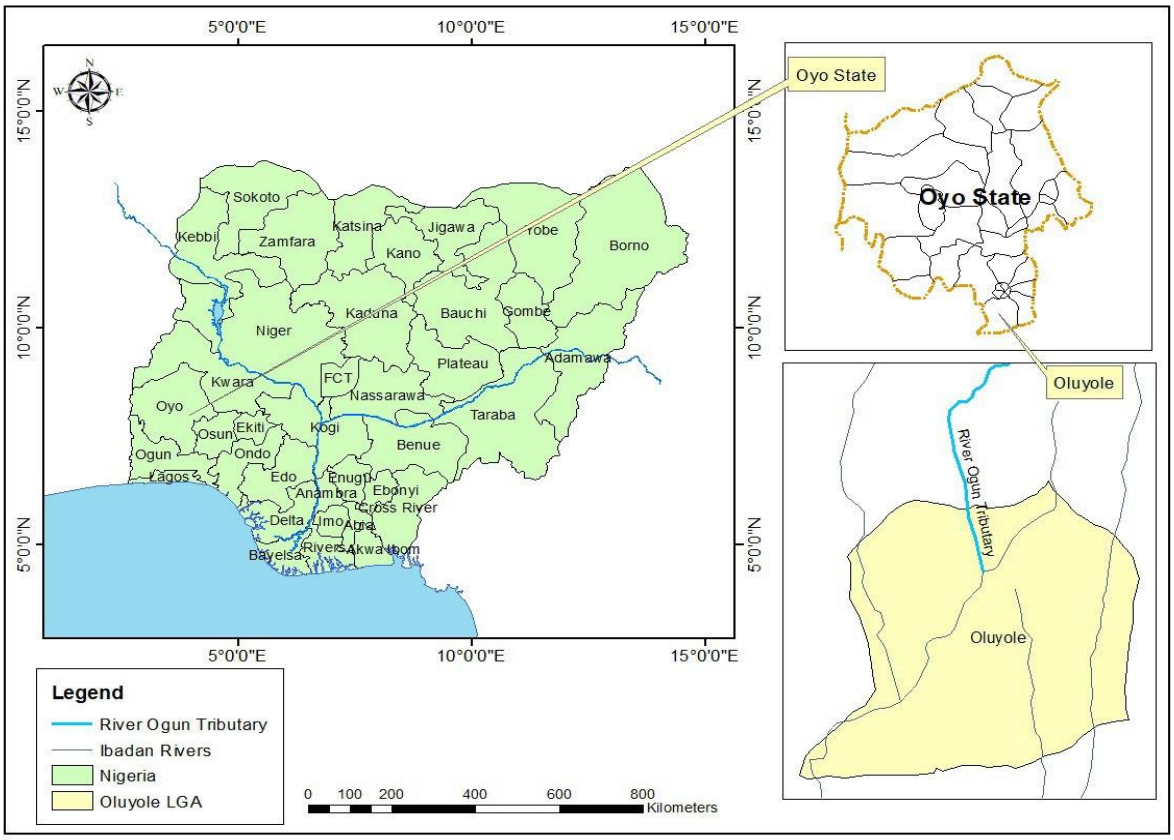

Figure 2: Map of River Ogun tributaries

Collection, Handling and Storage of Sediment Samples

. The sampling depth ranged from 0 to 5 cm. Sediment samples were collected and handled according to Cheung *et al.* [31]. After collection, the sediment samples were thoroughly mixed to ensure homogeneity. The sediments were air-dried naturally for a period of 3 to 4 days [32]. To ensure uniformity, the samples were passed through a 250 µm stainless steel sieve. Before undergoing subsequent processing, the samples were stored in pre-cleaned aluminium foil to preserve their integrity. The samples were then stored in pre-cleaned aluminium foils and transported in an ice-chest. The foils were specifically prepared to minimize contamination [15,17,23]. In order to maintain the quality of the samples and prevent any potential degradation, the foiled sediment samples were kept in a fridge at low temperatures until they were ready to be analysed.

Extraction of OCPs Residues from Surface Water Samples

The determination of OCPs residue was performed using a liquid-liquid extraction as described by Adegun *et al.* [10]. Debris and suspended materials were removed from the water samples by pre-filtration. This involved passing the samples through 0.45 µm fibre glass filters. The filters effectively separated and retained the unwanted particles, ensuring a cleaner and clearer sample for subsequent analysis. Twenty millilitres volume of a 1:1 mixture of n-hexane and dichloromethane was added to 500 mL separating funnel containing 100 mL of the filtered water sample. The mixture was vigorously shaken and then allowed to stand. As a result, two distinct layers formed: an organic phase and an aqueous phase. Once complete separation of the two layers was achieved, the organic phase (containing the target pesticide residues) was carefully drained into 250 mL conical flask. The aqueous phase was subjected to re-extraction once more in order to maximize the extraction efficiency. This was accomplished by repeating the process with an additional 20 mL of the extracting solvent [2,4,10].

Extraction of OCPs Residues from Sediment Samples

A weight of 5 g of the dried and sieved sediment samples was measured and placed into a previously cleaned extraction bottle, along with 30 mL of an extracting solvent consisting of a 1:1 ratio of n-hexane and dichloromethane (DCM). The samples were then subjected to extraction using an ultrasonic bath system for 10 mins. The resulting extract was separated from the sample and transferred to a new, pre-cleaned conical flask, where it was treated with another solvent. For the second extraction, 10 mL of solvent was used, and this process was repeated twice. The two extracts were combined for clean-up.

Clean-up of surface Water and Sediment Extracts

The extracts were purified by column chromatographic method using silica gel as adsorbent. Twenty millilitres of the concentrated extract was placed into a column, which had been prepared by plugging it with glass wool and the silica gel was subsequently topped with a 2.5g layer of anhydrous sodium sulphate. The purpose of the anhydrous sodium sulphate was to eliminate any remnant water from the extract. The extracts containing the pesticides was transferred onto the silica gel column. The extracts were eluted with 10 mL portions of hexane. This elution process allowed for the separation and quantitative collection of the target compounds. The eluate, containing the purified pesticides, was collected into a round bottom flask for concentration. In order to concentrate the eluates, evaporation to dryness was carried out using a rotary evaporator at very low temperature so as not to affect the analytes. This

process involved removing the solvent until only the dry pesticide residues remained prior to Gas chromatography-mass-spectrometry analysis (GC-MS) [32].

Optimized GC-MS condition

An Agilent gas chromatograph coupled to a mass spectrometer was used with triple axis detector equipped with an auto injector (10 µL syringe) and helium as a carrier gas was used for the analysis of the cleaned extracts in triplicates. All the chromatographic separations were performed on capillary column having the specification: length; 30m, internal diameter (0.25 mm), thickness; 0.25µm, treated with phenyl methyl siloxane [10]. Other optimised to Gas chromatography-mass-spectrometry analysis conditions were ion source temperature 240 °C, interface temperature; 200 °C, pressure: 15.2 psi in split mode with split ratio 1:50 with injection temperature of 300 °C. The temperature was raised to 240 °C at the rate of 20 °C/min. and held for 5 minutes with solvent delay of 5 minutes. The total elution time was 46.5 minutes [31].

Recovery and Precision Studies (Method Accuracy and Repeatability Evaluation)

Recovery experiments were conducted to evaluate the accuracy and extraction efficiency of the analytical method for the determination of selected organochlorine pesticides in the studied matrices [3,6,9]. Analyte-free samples were spiked with known concentrations of pesticide standards at different levels to cover the expected range of analytes in real samples. The spiked samples were subjected to the same extraction, clean-up, and analytical procedures as the actual samples [18].

The percentage recovery for each analyte was calculated by comparing the measured concentration with the corresponding spiked concentration. Acceptable recovery ranges were considered within 70–120%, indicating good method accuracy [14,18,24].

Precision of the method was assessed in terms of repeatability (intra-day precision) and reproducibility (inter-day precision). Repeatability was evaluated by analyzing multiple replicates ($n \geq 3$–5) of spiked samples within the same day under identical experimental conditions. Reproducibility was determined by analyzing similarly prepared samples on different days. Precision was expressed as the relative standard deviation (RSD, %), calculated from replicate measurements. RSD values below 15% were considered indicative of acceptable precision [20,24,28].

Determination of LOD and LOQ

The limit of detection (LOD) and limit of quantification (LOQ) were determined based on the signal-to-noise ratio approach, where LOD was defined as the concentration corresponding to a signal-to-noise ratio of 3:1, and LOQ as 10:1[22,24,28]. Alternatively, LOD and LOQ were calculated from the standard deviation of the response and the slope of the calibration curve using the expressions LOD = 3.3σ/S and LOQ = 10σ/S, where σ represents the standard deviation of the response and S is the slope of the calibration curve [16-19].

Quality Assurance and Quality Control

Quality assurance and quality control measures included the analysis of procedural, method, and solvent blanks to identify potential contamination and background interference. Instrument performance was routinely verified using quality control standards to ensure analytical stability and consistency. Method accuracy was evaluated through recovery studies using spiked samples, while precision was assessed based on relative standard deviation from replicate analyses under intra- and inter-day conditions [16-18]. All samples were analyzed in batches alongside calibration standards, blanks, and spiked samples to maintain data reliability and overall analytical quality throughout the study.

Results and Discussion

Method Validation

Table 1 presents some of the parameters for testing the performance of the developed method for analysing organochlorine pesticides in the River Ogun tributary. The key performance metrics for each pesticide, including correlation coefficient (R^2), limit of detection (LOD), and limit of quantification (LOQ). These metrics were crucial in evaluating the reliability and sensitivity of the analytical method. The coefficient of determination, R^2 values ranged from 0.9572 to 1.0000, indicating that the method demonstrates excellent linearity for most of the pesticides analysed.

A correlation coefficient close to 1 for Methoxychlor suggests a strong linear relationship between the concentration and the instrument's response. This is crucial for accuracy in quantifying pesticide levels. For instance, Endosulfan and p'p -DDE have slightly lower R^2 values (0.9572 and 0.9714, respectively), indicating a somewhat weaker but still acceptable linearity. Gao *et al.*, analysed organochlorine pesticides in river water, also reported high R-values typically exceeding 0.98, similar to this finding, thus affirming that the method is well-validated in terms of linearity. The percentage recoveries obtained for the pesticides ranged from 72% to 105%. The lowest recovery was observed for Endosulfan ether (72%),

while the highest recovery was recorded for Aldrin (105%). Other pesticides showed satisfactory recoveries, including Delta-lindane (87%), Heptachlor (96%), Heptachlor epoxide (89%), p,p'-DDE (99%), Dieldrin (97%), Endosulfan (102%), m,p'-DDD (94%), and Methoxychlor (90%). These values fall within the generally acceptable range of 70-120% for pesticide residue analysis, indicating good extraction efficiency and reliability of the analytical procedure [6,8,12,18,20]. The results therefore confirm that the method is suitable for the determination of organochlorine pesticides in environmental samples such as water and sediments.

The LOD values range from as low as 0.0191 µg/L (for Methoxychlor) to 6.1082 µg/L (for p'p -DDE). The LOQ span from 0.0579 (for Methoxychlor) to 22.8446 µg/L (for Endosulfan). Methoxychlor stands out with very low LOD and LOQ values, indicating that the developed method is highly sensitive for detecting and quantifying this pesticide at very low concentrations. In contrast, p'p -DDE and Endosulfan exhibit relatively high LOD and LOQ values, which suggests lower sensitivity for these compounds. This could be due to the chemical properties of these pesticides or the nature of the analysis method used.

Comparatively, the LOD and LOQ values for endosulfan in studies by Adir Cembranel *et al.*, [34], have LODs around 1.5 ng/mL and LOQs around 5 ng/mL respectively which aligns with the levels obtained from this study. This supports the reliability of the validated method for detecting pesticides in River Ogun tributary, though sensitivity could be improved for certain compounds. Delta-Lindane, Aldrin, and Heptachlor have moderate LOD and LOQ values, which are comparable to findings by Zhang *et al.*, [33] where LOD values for similar organochlorine pesticides were around 1-2 ng/mL.

This confirmed that the validated method was appropriate for environmental monitoring of these pesticides. Methoxychlor's exceptionally low LOD and LOQ are notable. In a study by Adir Cembranel *et al*.,[34]. LODs for similar pesticides were reported to be around 0.05 ng/mL, which is in line with the values obtained in this study. This indicated that the developed method was quite sensitive for Methoxychlor detection. p'p -DDE and Endosulfan ether show much higher LOD and LOQ values, which may suggest that the method might require further optimization to improve sensitivity for these pesticides, especially given that Adir Cembranel *et al.*, [34] reported lower LODs for similar compounds in river systems. The high R^2 values and generally acceptable LOD and LOQ values indicated that the developed method is suitable for monitoring organochlorine pesticide contamination in the River Ogun tributary.

Table 1: Validation of Developed Method Parameters

Pesticides	**Coefficient of Determination (R^2)**	**Recovery (%)**	**LOD** (µg/L)	**LOQ** (µg/L)
Delta- Lindane	0.9984	87	1.4362	4.3522
Endosulfan Ether	0.9998	72	0.493	1.4940
Heptachlor	0.9978	96	1.6553	5.0162
Aldrin	0.9985	105	1.3902	4.2127
Heptachlor epoxide	0.9987	89	1.2968	3.9229
p'p -DDE	0.9714	99	6.1082	18.5096
Dieldrin	0.9983	97	1.4902	4.5157
Endosulfan	0.9572	102	7.5387	22.8446
m'p -DDD	0.9927	94	3.0610	9.2758
M*e*thoxychlor	1.0000	90	0.0191	0.0579

However, the relatively high LOD and LOQ for compounds like endosulfan and p'p DDE suggest that further method development or pre-concentration steps might be necessary to improve the sensitivity, particularly in environments with low concentrations of these contaminants [10,26].

Given the environmental context, it is important to also consider the potential matrix effects which might contribute to higher LOD and LOQ values for certain compounds [2].

Concentration of Organochlorine Pesticides in Surface Water and Sediment

Table 2 shows results of the level of organochlorine pesticides (OCPs) residues in real surface water and sediment samples collected from Ogun River tributary at Idi Ayunre, near Ibadan. The concentrations are reported for upstream water, downstream water, and sediment at various points, and for specific OCPs, including different isomers of Lindane, Endosulfan, Aldrin, Heptachlor, and others. Alpha Lindane and Dichlorodiphenylchloroethylene (DDMU) compounds were not detected (BDL, Below Detection Limit) in all samples (water and sediment), which suggests they are either absent in the environment or below the detection sensitivity of the analysis. Beta Lindane concentrations are low but detectable, with slightly higher levels in downstream water. Gamma Lindane concentrations were almost the same in both upstream and downstream waters. This suggests minimal variation in Lindane contamination between these two points in the river. Delta Lindane shows higher concentrations (0.13 µg/L) in the upstream water compared to the downstream water (0.10 µg/L), though the difference is small.

Endosulfan showed a very high concentration in the upstream water (10.24 µg/L) compared to downstream water (0.99 µg/L). The high variability in upstream concentrations (indicated by the high standard deviation) suggests sporadic contamination or the presence of a source point of endosulfan in the upstream section [10]. Heptachlor concentrations are also higher in the upstream water (0.04 µg/L) compared to the downstream (0.03 µg/L), but the difference is very small, indicating little variation in contamination levels. Heptachlor epoxide showed no detectable levels in the water and sediment samples, which could either mean these compounds were not present in the environment or they were below the detection thresholds for the study. Endrin is detectable in both upstream (0.04 µg/L) and downstream water (0.02 µg/L), with slightly higher levels in the upstream water. Beta Endosulfan is present in both water samples, with a slight decrease from upstream to downstream (0.06 µg/L to 0.05 µg/L). DDT metabolites (m,p'-DDD, p,p'-DDE). These compounds show consistent concentrations in both upstream and downstream waters, suggesting either widespread contamination or the persistence of Dichlorodiphenyl trichloroethane (DDT) residues in the environment [12,18, 20]. p,p'-DDE was not detected in any sample, suggesting limited occurrence within the study area.

Methoxychlor showed nearly identical concentrations in both water samples (0.07 µg/L in upstream and downstream), indicating steady contamination levels across the river section. The total concentrations of OCPs in the upstream water (12.28 µg/L) were significantly higher compared to downstream water (1.39µg/L) and sediment (0.90 µg/kg). This could suggest a point source pollution or higher pesticide use in the upstream area, which depletes downstream. The standard deviations are quite high, especially in the upstream water, where the value of 2.92 is much larger than the mean (1.12). This indicates significant variability in pesticide levels, which may point to irregular sources of contamination [35]. These findings established that their contamination level was higher than the 0.5 µg/L total maximum residues limit (MRL) by the World Health Organization [10]. The sediment levels were also above the 0.2 µg/kg MRL set by the European Union [2,4,10].

Table 2: Mean Concentration of Organochlorine Pesticides Residues in Surface Water and Sediment Samples

Organochlorine Pesticides	Upstream water (µg L^{-1})	Downstream Water (µg L^{-1})	Sediment (µg kg^{-1})
Alpha Lindane	BDL	BDL	BDL
beta Lindane	0.01 ± 0.00	0.02 ± 0.01	0.02 ± 0.01
gamma Lindane	0.03 ± 0.00	0.03 ± 0.01	0.03 ± 0.01
delta Lindane	0.13 ±0.01	0.10 ± 0.00	0.09 ± 0.00
Endosulfan ether	10.24 ±11.46	0.99 ± 0.80	0.54 ± 0.46
Heptachlor	0.04 ± 0.01	0.03 ± 0.00	0.03 ± 0.00
Aldrin	1.58 ± 2.08	BDL	BDL
Heptachlor epoxide	0.00 ± 0.00	0.00 ±0.00	0.00 ± 0.00
DDMU	BDL	BDL	BDL
Alpha Endosulfan	BDL	BDL	BDL
p,p ' – DDE	BDL	BDL	BDL
Dieldrin	BDL	BDL	BDL
Endrin	0.04 ± 0.02	0.02 ± 0.01	0.02 ± 0.01
Beta Endosulfan	0.06 ± 0.03	0.05 ± 0.03	0.03 ± 0.01
m,p'-DDD	0.02 ± 0.01	0.02 ± 0.01	0.02 ± 0.01
m,p'-DDT	0.06 ± 0.04	0.06 ± 0.01	0.06 ± 0.04
Methoxychlor	0.07 ± 0.03	0.07 ± 0.01	0.06 ± 0.03
Total	12.28	1.39	0.90
Mean	1.12	0.14	0.09
SD	2.92	0.28	0.15

OCPs = Organochlorine Pesticides; SD = Standard Deviation; BDL= Below Detection Limit 0.0001

Note: Standard deviation was provided as the measure of dispersion in this; however, precision assessment using %RSD was not appropriate due to values falling near or below the limit of quantification. The mean values are of triplicate analyses.

This study is in agreement with the study by Adehuga *et al*., [23] on determination of organochlorine pesticide residues and trace metal concentration in well and stream waters from agricultural farm settlement in Ile-Oluji, Ondo State, where upstream water showed elevated pesticide levels due to the proximity of agricultural activities. This was particularly evident with endosulfan and methoxychlor, both of which showed concentrations as high as 20 µg/L in upstream waters and much lower levels downstream. Moreso, the study findings were also in agreement with the study on by Fosu -Mensah [36] where total pesticide concentrations were higher in regions with intensive agricultural runoff, with similar trends of elevated upstream contamination due to direct pesticide discharge into the waterway [30,33].

However, this study differs in findings from the study by Akoto *et al.*, [35] in a study of pesticide residues in water, sediment and fish from Tono Reservoir and their health risks implications*;* where total OCP concentrations were significantly higher in agricultural regions, ranging from 20-40 µg/L in water samples. This contrasts with the findings from this study, where the total pesticide concentration in upstream water was much higher (12.28 µg/L). The difference could be attributed to the use of larger quantities of OCPs in agriculture in the Ogun River, compared to the Tono Reservoir, which may have less intense agricultural pesticide use or more effective environmental controls.

Therefore, it is worthy to note that these contaminants were still present in the environment despite their global banning [37]. This could be as a result of their persistence or their being sold under different trade names especially in developing countries. The general trend of the distribution of these pollutants in upstream samples followed by downstream water and sediment is (upstream > downstream > sediment) (Figure 3) while Figure 4 gives the representative chromatographic peaks of the investigated matrices.

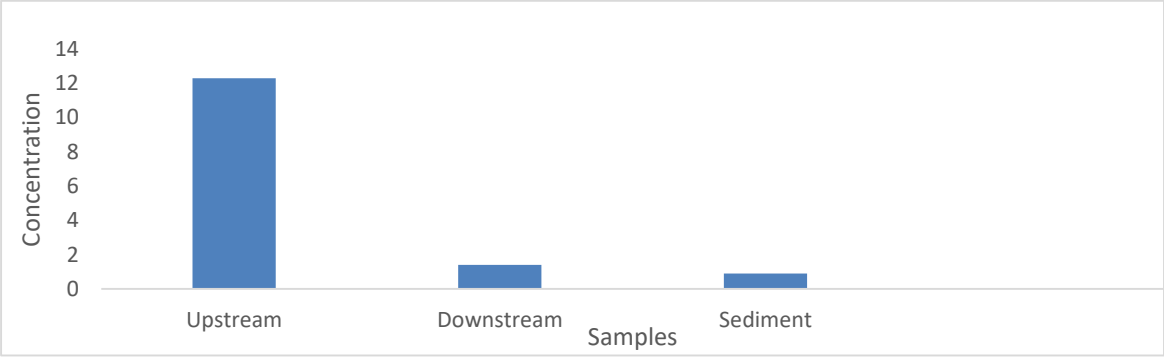

Figure 3: Total OCP compounds in upstream, downstream and sediments of Ogun River Tributary

Note: The upstream and downstream water samples concentration levels were expressed in µg/L while the sediment samples were expressed in µg/kg.

Statistical Analysis

One-way analysis of variance (ANOVA) was used to evaluate differences in organochlorine pesticide (OCP) concentrations among upstream water, downstream water, and sediment samples. The analysis showed that the mean concentrations of OCPs did not differ significantly among the three sampling locations. Although the total OCP concentration was highest in upstream water (12.28 µg L^{-1}) compared with downstream water (1.39 µg L^{-1}) and sediment (0.90 µg kg^{-1}), the observed variation was largely influenced by the high concentration of endosulfan ether in the upstream samples and was not statistically significant at the 95% confidence level. Statistical significance was set at $p < 0.05$.

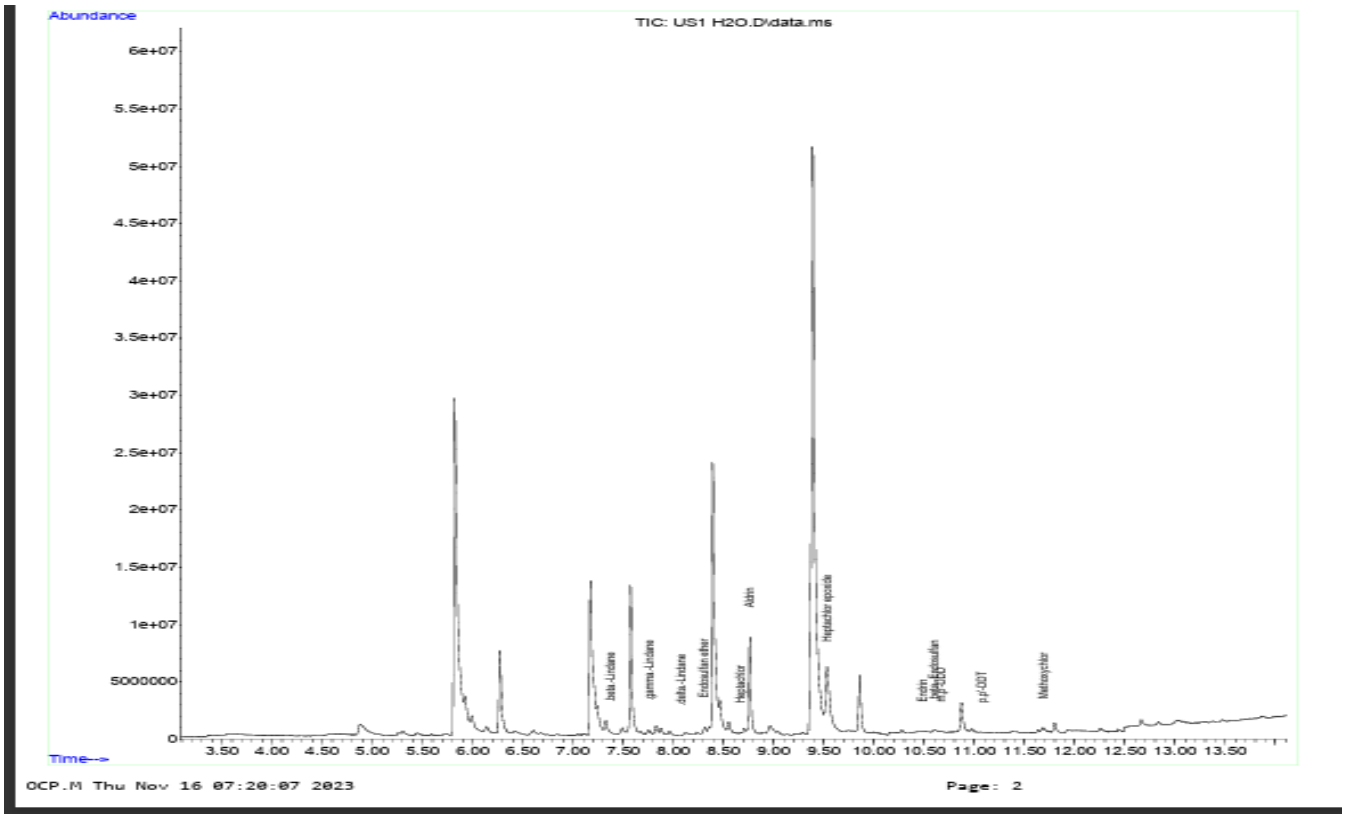

Figure 4**:** Representative Chromatographic peaks of the investigated matrices

Conclusion

The validated analytical method demonstrated satisfactory linearity, accuracy, and sensitivity for the determination of organochlorine pesticide residues in environmental samples. Detectable concentrations of several OCPs were found in water and sediment samples from Odo Ona River, with upstream locations exhibiting higher contamination levels. The concentrations exceeded recommended guideline values, indicating potential environmental and public health concerns.

Recommendations

The findings highlight the need for continuous monitoring and strengthened regulatory measures to minimize organochlorine pesticides (OCPs) contamination and protect aquatic ecosystems and public health. Regular water quality assessments, public awareness initiatives, and the adoption of sustainable agricultural practices should be encouraged to reduce their input and prevent further environmental contamination.

Acknowledgement

We express our sincere gratitude to the technologists of the Department of Chemistry, University of Ibadan for their invaluable support and technical expertise, which greatly contributed to the successful completion of this study. We are also deeply grateful to the residents of Idi Ayunre, administrative headquarters of Oluyole Local Government Area, Oyo State, Nigeria for their cooperation and assistance during the field sampling exercises. Their local knowledge and willingness to facilitate access to sampling sites were crucial to the smooth execution of the study. Additionally, we acknowledge any colleagues, mentors, and institutions whose guidance and encouragement helped in the planning, analysis, and interpretation of the research findings.

References

1. Abaineh, A., Ejigu, D., Atlabachew, M. & Dejen, E. (2024). Risks of pesticides on aquatic ecosystems and pesticide management effectiveness in Ethiopia: review. *International Journal of Environmental Science and Technology*, 21, 8833-8848.
2. Adegun, A. O., Akinnifesi, T. A., Ololade, I. A., Busquets, R., Hooda, P. S., Cheung, P. C. W., Aseperi, K. A. & Barker, J. (2020). Quantification of neonicotinoid pesticides in six cultivable fish species from the River Owena in Nigeria and a template for food safety assessment. *Water,* 12 (9), 2422.
3. Forns, J., Lertxundi, N., Aranbarri, A., Murcia, M., Gascon, M., Martinez, D., Grellier, J., Lertxundi, A., Julvez, J., Fano, E., Goñi, F., Grimalt, J. O., Ballester, F., Sunyer, J. & Ibarluzea, J. (2012). Prenatal exposure to organochlorine compounds and neuro psychological development up to two years of life. *Environment International, 45*, 72-77.
4. Adegun, A. O., Akinnifesi, T. A., Ololade, I. A., Olanisakin, A. A. & Omodara, B. N. (2020). Cabamates and pyrethroid pesticide residues in fish from Owena River, Ondo State, Nigeria and their health risk evaluation. *Scholars International Journal of Chemistry and Material Sciences, 3*(4), 41- 47.

5. Steenland, K., Mora, A. M., Barr, D. B., Juncos, J., Roman, N. & Wesseling, C. (2014). Organochlorine chemicals and neurodegeneration among elderly subjects in Costa Rica. *Environmental Research,* 134, 205-209.
6. Adegun, A. O., Ajibola, A., Hashimi, M. A., Adegoke, A. S., Amao, L. G. & Adewuyi, G. O. (2025). Occurrence and distribution of polycyclic aromatic hydrocarbons in raw foods from selected markets in Ibadan, Nigeria. *Chem Class Journal*, *9*(1), 1254-1272.
7. Njoku, K., Ezeh, C., Obidi, F. & Akinola, M. (2017). Assessment of pesticide residue levels in vegetables sold in some markets in Lagos State, Nigeria. *Nigerian Journal of Biotechnology, 32,* 53-60.
8. Olisah, C., Okoh, O. O. & Okoh, A. I. (2019). Global evolution of organochlorine pesticides research in biological and environmental matrices from 1992 to 2018: a bibliometric approach. *Emerging Contaminants, 5*, 157-167.
9. Olutona, G. O., Ayano, S. A. & Obayomi Davies, O. (2014). Organochlorine pesticide in water and bottom sediment from Aiba Reservoir (Southwestern Nigeria). *Chemistry and Ecology, 30*(6), 513-531.
10. Adegun, A. O., Akinnifesi, T. A., Adewunmi, A. A., Alaba, G. A., Odeniran, A. O., Agboola, O. O., Adeagbo, D. A., Adebayo, A., Aseperi, K. A. & Barker, J. (2024). Organochlorine and organophosphate pesticides in cocoa producing soil, sediment, surface and treated water from Owena River Basin, Nigeria. *Nigerian Research Journal of Chemical Sciences, 12* (2), 418-447.
11. Keswani, C., Dilnashin, H., Birla, H., Roy, P., Tyagi, R. K., Singh, D., Rajput, V. D., Minkina, T. & Singh, S. P. (2022). Global footprints of organochlorine pesticides: a pan global survey. *Environmental Geochemistry and Health, 44*, 149-177.
12. Chukwudi, O. & Annabel, O. (2021). Organochlorine pesticides determination of Okumesi River Umukwata for pen aquaculture in schools: a tool for wealth creation in Nigeria. *ATBU Journal of Science, Technology and Education, 9*(1), 336-345.
13. Zhou, W., Li, M. & Achal, V. (2024). A comprehensive review on environmental and human health impacts of chemical pesticide usage. *Environmental Challenges*, 100410.
14. Ezequiel, P., Miguel, G., Gustavo, H. M. & Nora, M. (2015). Toxicology of organochlorine: implications of presence in breast milk. *Journal of Applied Life Sciences International, 2,* 49-64.

15. Soleman, S. R., Lee, H. J. & Kim, Y. S. (2024). Effect of pesticide exposure on stunting incidence: implications for child neurodevelopment. *Clinical and Experimental Pediatrics*, *67*, 1123-1132.
16. Patel, S. & Sangeeta, S. (2019). Pesticides as the drivers of neuropsychotic diseases, cancers, and teratogenicity among agro workers as well as general public. *Environmental Science and Pollution Research*, *26*, 91-100.
17. Adegun, A. O., Akinnifesi, T. A. & Ololade, I. A. (2020). A critical review of persistent organic pollutants and their health effects. *Scholars International Journal of Chemistry and Material Sciences, 3*(5), 48–57.
18. Kumar, J., Lind, L., Salihovic, S., van Bavel, B., Ingelsson, E. & Lind, P. M. (2014). Persistent organic pollutants and liver dysfunction biomarkers in a population based human sample of men and women. *Environmental Research*, *134*, 251-256.
19. Oyeyiola, A. O., Fatunsin, O. T., Akanbi, L. M., Fadahunsi, D. E. & Moshood, M. O. (2017). Human health risk of organochlorine pesticides in foods grown in Nigeria. *Journal of Health and Pollution, 7*, 63–70.
20. Akinnifesi, T. A., Borokini, F. B., Adegun, A. O., Mafinuyomi, E., Olonisakin, A., Omodara, N. B. & Oladoja, N. A. (2024). Polycyclic aromatic hydrocarbon in sediment, ground and surface water resources in coastal areas of Ondo State, Nigeria. *Journal of Chemical Society of Nigeria, 49*(2), 261-282.
21. Shinggu, D., Yamta, O. M. & Jeffrey, T. B. (2015). Level of organochlorine pesticides residue in fish, water and sediment in Biu Dam (Reservoir), Borno State, Nigeria. *International Research Journal of Pure and Applied Chemistry, 5*, 150–159.
22. Witczak, A., Pohoryło, A. & Mituniewicz Małek, A. (2016). Assessment of health risk from organochlorine xenobiotics in goat milk for consumers in Poland. *Chemosphere, 148*, 395-402.
23. Adehuga, O. S., Sheriff, U. I., Ugochukwu, U. & Ayooluwa, A.I. (2022). Determination of organochlorine pesticide residues and trace metal concentration in well and stream waters from agricultural farm settlement in Ile Oluji, Ondo State. *Chemical Science International Journal, 31*(1), 1-8.
24. Klinčić, D., Herceg Romanić, S., Matek Sarić, M., Grzunov, J. & Dukić, B. (2014). Polychlorinated biphenyls and organochlorine pesticides in human milk samples from two regions in Croatia. *Environmental Toxicology and Pharmacology, 37* (2), 543-552.

25. Jayaraj, R., Megha, P. & Sreedev, P. (2016). Organochlorine pesticides, their toxic effects on living organisms and their fate in the environment. *Interdisciplinary Toxicology, 9*(3-4), 90-100.
26. Ishaq, Z. & Nawaz, M. (2018). Analysis of contaminated milk with organochlorine pesticide residues using gas chromatography. *International Journal of Food Properties, 21*(1), 879-891.
27. Ismail, M., Sayed, M., Khan, H. M. & Cooper, W. J. (2014). Analysis of pesticides in water samples and removal of monocrotophos by γ irradiation. *Journal of Analytical & Bioanalytical Techniques*, 5, 181-187.
28. Adegun, A. O., Akinnifesi, T. A., Eluyeba, O. J., Agboola, O. O., Adeagbo, D. A., Aseperi, K. A. & Barker, J. (2024). Seasonal variation in bioaccumulation of organochlorines and organophosphate pesticides in fish from River Owena, Nigeria and their health risk appraisal. *Nigerian Research Journal of Chemical Sciences, 12*(1), 51–74.).
29. Saunders, L., Kadhel, P., Costet, N., Rouget, F., Monfort, C., Thome, J. P., Guldner, L., Cordier, S. & Multigner, L. (2014). Hypertensive disorders of pregnancy and gestational diabetes mellitus among French Caribbean women chronically exposed to chlordecone. *Environment International, 68*, 171-176.
30. Al Antary, T. M., Alawi, M. A., Estityah, H., Haddad, N. & Al Antary, E. T. (2015). Chlorinated pesticide residues in human breast milk collected from Southern Jordan in 2012/2013. *Toxin Reviews, 34*(4), 190–194.
31. Cheung, P. C. W., Busquets, R., Hooda, P. S., Aseperi, A. K., Barker, J., Adegun, A. O., Akinnifesi, T. A., Ololade, I. & Abosede, S. (2023). Characterisation of neonicotinoid insecticides in the cocoa producing Owena River Basin of Nigeria by a QuEChERS method coupled to liquid chromatography tandem mass spectrometry. *The Open Environmental Research Journal, 16*, e259027762301300.
32. Shaker, E. M. & Elsharkawy, E. E. (2015). Organochlorine and organophosphorus pesticide residues in raw buffalo milk from agro industrial areas in Assiut, Egypt. *Environmental Toxicology and Pharmacology, 39*(1), 433-440.
33. Zhang, G., Pan, Z. & Bai, A. (2014). Distribution and bioaccumulation of organochlorine pesticides in the food web of Nansin Lake, China. *Environmental Monitoring and Assessment, 186*(4), 2039-2051.

34. Adir Cembranel, S. E., Frigo, P., Silvio, C. & Sampaio, E. M. (2017). Residue analysis of organochlorine and organophosphorus pesticides in urban lake and sediments. *Engenharia Agrícola*, *37*(6), 1254-1267.
35. Akoto, O., Azuure, A. & Adoteyi, A. (2016). Pesticide residues in water, sediment and fish from Tono Reservoir and their health risk implications. *SpringerPlus*, *5*, 1849–1860.
36. Fosu Mensah, B., Elvis, D. O., Darko, G. & Gordon, C. (2016). Assessment of organochlorine pesticide residues in soils and drinking water sources from cocoa farms in Ghana. *SpringerPlus*, *5*(1), 869.
37. Afzala, W., Habib, S. S., Ujanc, J. A., Mohanye, M. & Bibib, H. (2024). Assessment of pesticide residues: a comprehensive analysis of seasonal trends and health implications. *Journal of Environmental Science and Health, Part B, 59*(10), 642-653.