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ORGANOCHLORINE PESTICIDE RESIDUES IN FISH AND DAM WATER FROM OGBOMOSO, SOUTHWESTERN NIGERIA: SPATIAL DISTRIBUTION AND MATRIX-DEPENDENT CONTAMINATION

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Abstract

This study assessed pesticide residues in water and Nile tilapia (*Oreochromis niloticus*) from Ogbomoso Waterworks and Papa Abede Waterworks dams in Ogbomoso, Oyo State, Nigeria, focusing on their occurrence, concentrations, spatial distribution and compliance with applicable residue limits. Water samples were randomly collected from different sections of the waterbodies, while sixteen fish specimens were obtained, comprising six from each study dam and two from each of two privately managed control water bodies. Fish muscle tissues were extracted using the QuEChERS method, and pesticide residues were identified and quantified using gas chromatography–mass spectrometry (GC–MS). Data were analysed using descriptive statistics and analysis of variance, with Duncan's Multiple Range Test applied at a 5% significance level. The results revealed organochlorine, organophosphate, carbamate and pyrethroid pesticide residues in the sampled water and fish. Among the organochlorines, p,p′-DDT recorded 3.60 mg/l in Ogbomoso Waterworks water and 0.64 mg/l in Papa Abede Waterworks water, while concentrations in fish were 0.73 and 0.75 mg/kg, respectively. The concentration of p,p′-DDD in water reached 2.34 mg/l. Among the organophosphates, dimethoate recorded 2.74 mg/l in water, while dichlorvos reached 2.61 mg/l in Ogbomoso Waterworks water and 0.86 mg/kg in fish from the same location. Carbaryl recorded 1.59 mg/l in water, whereas permethrin reached 0.69 mg/l in Ogbomoso Waterworks water. Overall, Ogbomoso Waterworks generally exhibited higher pesticide-residue concentrations than Papa Abede Waterworks, while the

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control locations recorded comparatively lower concentrations for many compounds. Statistical comparisons showed variations among compounds, locations and sample types, although not all differences were statistically significant. Comparison with the applicable benchmarks adopted in the study indicated that several residues exceeded the relevant limits, suggesting contamination of the investigated aquatic environments and non-compliance with applicable maximum residue limits in affected fish samples. The study recommends routine pesticide-residue monitoring, improved pesticide application practices and measures to minimise agricultural runoff into water bodies. The findings provide baseline information for environmental management and food-safety monitoring in Ogbomoso, Nigeria.

Introduction

Pesticide contamination of aquatic ecosystems is an important environmental concern because pesticides used in agricultural landscapes can reach surface waters through runoff, drainage, erosion, leaching and spray drift (AbuQamar *et al.,* 2024; Abaineh *et al.,* 2024). Once pesticides enter aquatic systems, their environmental fate is influenced by properties such as solubility, persistence, hydrophobicity, adsorption and degradation, which determine their distribution among water, suspended particles, sediments and aquatic organisms (AbuQamar *et al.,* 2024; Benson *et al.,* 2023).

Organochlorine pesticides (OCPs) are of particular concern because many compounds are persistent and may remain detectable in environmental and biological matrices long after their application has ceased (Olisah *et al.,* 2020; AbuQamar *et al.,* 2024). Although the use of several OCPs has been restricted or prohibited in many countries, residues of compounds such as aldrin, dieldrin, endrin, heptachlor and DDT-related chemicals continue to be reported in African aquatic environments and biota (Olisah *et al.,* 2020; Benson *et al.,* 2023).

Fish are important matrices for pesticide monitoring because they may accumulate contaminants from water, food and sediments, thereby providing evidence of biological exposure within aquatic food webs (Jonsson & de Queiroz, 2023; AbuQamar *et al.,* 2024). The relevance of fish monitoring is also strengthened by the fact that fish are widely consumed as food, making pesticide residues in edible tissues important from a food-safety perspective (Jonsson & de Queiroz, 2023).

Recent research in Ibadan, southwestern Nigeria, detected OCP residues in freshwater fish from Dandaru River and Eleyele Lake and demonstrated that contamination levels and associated dietary risks varied among fish species and locations (Olanrewaju *et al.,* 2025). Similarly, recent investigation of fish from the Osun River detected multiple OCP congeners and reported spatial variation in residue concentrations, further demonstrating the continuing occurrence of persistent pesticide residues in aquatic food resources in southwestern Nigeria (Oladimeji *et al.,* 2026).

Evidence from Chanchaga River in Nigeria has also demonstrated the occurrence of multiple OCP residues in surface water, with concentrations reported above selected international drinking-water standards at some sampling stations (Ikayaja *et al.,* 2024). Spatial differences in pesticide residues can arise from variation in surrounding land use, runoff pathways, hydrological conditions,

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sediment characteristics and contaminant retention, making location-specific assessment important when evaluating aquatic contamination (Benson *et al.,* 2023; Oladimeji *et al.,* 2026).

Despite increasing evidence of OCP contamination in Nigerian rivers, lagoons and lakes, information remains comparatively limited for smaller inland Waterbodies that support local fisheries and may receive pesticide inputs from surrounding agricultural activities (Olisah *et al.,* 2020; Olanrewaju *et al.,* 2025). The present study therefore investigated the occurrence and distribution of selected OCP residues in water and Nile tilapia (Oreochromis niloticus) from Ogbomoso Waterworks and Papa Abede Waterworks Waterbodies in Ogbomoso, southwestern Nigeria, with emphasis on differences between fish and water, spatial variation among sampling locations, and the interaction between sampling location and sample type (Jonsson & de Queiroz, 2023; Oladimeji *et al.,* 2026).

Study Area

The study was carried out at LUTECH Ogbomoso, Oyo State, Nigeria, with laboratory work conducted in the Department and Laboratory of Crop and Environmental Protection, Ladoke Akintola University of Technology (LAUTECH). Ogbomoso is located approximately at latitude 8.1333°N and longitude 4.2356°E, with a mean annual temperature of about 26.1°C.

Experimental Sites

Two Waterbodies were selected as the experimental sites and two control sites were also selected: (1) Waterworks Dam, Areago Area, along Ogbomoso Waterworks, Ogbomoso, located approximately at latitude 8.20544°N and longitude 4.19615°E; and (2) Waterworks Dam, Papa Abede Esaanu Aje Area, behind Papa Abede Waterworks High School Ogbomoso, located approximately at latitude 8.13373°N and longitude 4.24014°E. Also these two Waterbodies constitute the study sites for collection of water and fish samples and the control site for Waterworks Dam, Areago, Ogbmoso

Water Sample collection

Water samples were collected from the two selected water bodies. Each water body was divided into four regions, and a sampling line (transect) was established across each region. Samples were collected at random points along the transect, including approximately 3 m (±1 m) from the riverbank and at the centre of each region, to account for spatial variations in flow dynamics. Water samples were collected from a depth of 0–15 cm using clean 5-L containers. The containers were washed with detergent, thoroughly rinsed with deionised water, and rinsed three times with water from the sampling site before sample collection. Each sample container was properly labelled with the sampling location, date and season, placed in an ice-packed cooler, and transported to the laboratory for further analysis. And the control was collected 75 m upstream of each water body

In the laboratory, samples from each location were thoroughly mixed, and 50 ml aliquots was measured in four (4) places from each location. The aliquots were properly labelled and stored under refrigeration until analysis.

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Fish Sample collection

A simple random sampling technique was used to collect the fish samples from the two selected waterbodies. A total of sixteen (16) specimens of Nile tilapia (*Oreochromis niloticus*) were collected for pesticide-residue analysis. Six (6) specimens were randomly collected from each of the two selected waterbodies, Ogbomoso Waterworks and Papa Abede Waterworks dams, giving twelve (12) specimens from the main study locations. An additional two (2) specimens were collected from each of two privately managed fish waterbodies, which were purposively selected to serve as control locations, giving a total of sixteen (16) specimens. The sampling intensity was considered appropriate for the contaminant-monitoring objectives of the study and was consistent with recommendations for fish contaminant sampling and previous pesticide-residue research (U.S. EPA, 2022; Unyimadu *et al.,* 2018). The fish samples were placed in clean, labelled polyethylene bags and transported in an insulated container containing ice packs to minimise spoilage and preserve sample integrity prior to laboratory analysis.

Water Sample Preparation.

Before analyzing samples for pesticide residues, the water sample will undergo filtration using 0.45 µm fibre glass filters (manufactured by Whatman) to eliminate any suspended particles.

The water samples were stored in chilled cooler boxes at a temperature of 4 ± 2 ◦C and transported to the Pesticide Residue Laboratory. This laboratory was certified and specializes in conducting quantitative multi-residue pesticide analysis. The extraction of pesticide residues from water samples was conducted using US EPA Method 3510 which is specifically designed for the analysis of semi-volatile and non-volatile organics in aqueous matrices.

Preparation of fish samples and extraction

The extraction and cleanup processes was carried out using the original QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) technique. 10 grams of the homogenized fish sample was placed into centrifuge tubes and replicate four times from the mixed fish samples. After adding 1.6 g of anhydrous $MgSO_4$, 0.4 g of NaCl, and 4 mL of ACN (containing 1% acetic acid), the mixture was stirred together for one minute in a vortex mixer or a bowl. To separate the phases, the mixture was centrifuged for three minutes at 3000 rpm (liquid-liquid partition). The organic solvent ACN-corresponding supernatant was put into a tube with 150 mg of $MgSO_4$, 70 mg of C18and 70 mg of adsorbent PSA. After a 30-second handshake, the tube will have centrifuged for one minute at 4000 rpm. To eliminate the extra coloring components, the supernatant was filtered using a syringe filter (0.45µm). The filtered supernatant was kept at 4 °C after being evaporated at 45 °C under a nitrogen stream. Under ideal circumstances, the solutions were moved to an auto sampler vial and injected into the GC-ECD.

Extraction and Clean up Procedures

AOAC Pouch Qsep (QuEChERS Sample Extraction Pouches) sachets containing magnesium sulfate and sodium acetate was obtained from Agilent Technologies (Santa Clara, USA). HPLC-grade acetonitrile was sourced from Romil (Cambridge, UK), while glacial acetic acid, primary and secondary amine (PSA), deionised water, acetone and HPLC-grade pesticide standard solutions

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was procured from Sigma-Aldrich, (Germany). Pesticide residues was analyse using a multi-residues extraction and cleanup procedure based on the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method, following AOAC official method 2007.01 with slight modification to accommodate the spiking of multiple analytes.

The samples were analyzed using Gas Chromatography-Mass Spectrometry GC-MS) and was conducted at Afe Babalola University's Chemical Engineering Laboratory in Ado Ekiti, Nigeria.

Statistical analysis

Data were entered and analyzed using IBM SPSS Statistics. Descriptive statistics, including mean, standard deviation, and standard error, will summarize pesticide residue concentrations. Analysis of variance (ANOVA) was used to compare mean residue levels between the two Waterbodies, and Duncan's Multiple Range Test was applied for mean separation at a 5% significance level where appropriate.

General comparison of organochlorine residues in fish and water

The general comparison showed no significant difference in OCP concentrations between fish and water samples ($P \leq 0.05$) although water generally recorded higher concentrations. Aldrin occurred at 0.30 mg/kg in fish and 0.62 mg/l in water, while Dieldrin was 0.49 mg/kg and 0.51 mg/l, respectively. Similar patterns were observed for Endrin, Heptachlor and Heptachlor oxide. The higher concentrations in water may reflect differences in environmental partitioning, recent inputs and physicochemical behaviour, whereas residues in fish represent uptake and subsequent biological processing. Pesticides can partition among water, sediments and aquatic organisms according to their persistence and physicochemical properties (Benson *et al.,* 2023; AbuQamar *et al.,* 2024).

Endosulfan and Endosulfan II showed relatively small differences between matrices, whereas the DDT-related compounds exhibited greater differences. Notably, p,p′-DDT reached 1.14 mg/l in water compared with 0.41 mg/kg in fish. The detection of DDT and its metabolites is environmentally important because these compounds are persistent and may remain in aquatic systems for extended periods. Recent studies have also reported OCP residues in Nigerian freshwater fish and surface waters, including systems in Ibadan and the Osun River (Olanrewaju *et al.,* 2025; Oladimeji *et al.,* 2026).

Although the absence of a significant matrix effect indicates that the overall fish–water differences were not statistically significant, the higher concentrations observed in water demonstrate the importance of assessing both sampling locations and sample types. The occurrence of OCP residues in fish further indicates exposure within the aquatic food web and supports the need for continued monitoring of water, sediment and edible fish. (Benson *et al.,* 2023; Olanrewaju *et al.,* 2025; Oladimeji *et al.,* 2026).

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Tab. 1: General Comparison of Organochlorine Residue Concentrations in Fish and Water Samples

Pesticide detected	Fish Conc. (mg/kg)	Fish MRL (mg/kg)	Water Conc. (mg/l)	Water Quality benchmark (mg/l)
Aldrin	0.30a	0.10	0.62a	0.00003
Dielrin	0.49a	0.10	0.51a	0.00003
Endrin	0.36a	0.01	0.46a	0.00002
Heptachlor	0.30a	0.01	0.49a	0.00003
Heptachlor Oxide	0.26a	0.01	0.62a	0.00003
Endosulfan	0.27a	0.05	0.28a	0.01
Endosulfan II	0.27a	0.05	0.36a	0.01
P'P' DDT	0.41a	0.05	1.14a	0.001
P'P DDE	0.31a	0.05	0.45a	0.001
P'P' DDD	0.20a	0.05	0.75a	0.001

Note: Value with the same alphabet along the row are not significant different.
MRL represents the Maximum Residue Limit (mg/kg).
WQB represents the Water Quality Benchmark (mg/l)
The MRL values were adopted from the Codex Alimentarius Commission (CAC/FAO/WHO, 2023) and the European Commission Regulation (EC)

Spatial distribution of organochlorine residues across the sampling location

Sampling location significantly influenced the concentrations of OCP residues ($P \leq 0.05$), with Ogbomoso Waterworks generally recording the highest concentrations, followed by Papa Abede Waterworks, while the control locations had lower residues. Aldrin, Dieldrin, Endrin, Heptachlor and Heptachlor oxide reached 1.10, 0.97, 0.97, 1.00 and 1.20 mg/kg, respectively, at Ogbomoso Waterworks. The DDT-related compounds were also prominent, with p,p′-DDT recording the highest concentration (2.17 mg/kg). Most residues at Ogbomoso Waterworks and Papa Abede Waterworks exceeded their respective MRLs.

The higher concentrations at the impacted locations agree with findings from other Nigerian aquatic systems. Olanrewaju *et al.* (2025) detected OCP residues in fish from Dandaru River and Eleyele Lake in Ibadan, including *Oreochromis niloticus*, and reported differences in residue occurrence among fish species and water bodies. Similarly, Oladimeji *et al.* (2026) reported total OCP concentrations of 0.219–2.555 mg/kg in fish from eight locations along the Osun River, demonstrating spatial variation in OCP contamination across southwestern Nigeria.

The predominance of DDT-related compounds in the present study is also consistent with Benson *et al.* (2023), who identified DDT-related compounds and cyclodienes among the major OCP groups in Lagos Lagoon water and sediments and suggested contributions from current as well as historical inputs. The consistently higher residues at Ogbomoso Waterworks may therefore reflect greater local pesticide inputs and environmental retention than at the control locations. Overall, the spatial pattern observed in this study is consistent with previous Nigerian studies showing that OCP contamination varies among aquatic locations according to local activities and environmental conditions.

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Tab.2.: Comparative Distribution of Organochlorine Pesticide Residues across the Sampling Locations.

Pesticide Detected	Ogbomoso Waterworks	Papa Abede Waterworks	Ogbomoso Waterworks Control	Papa Abede Waterworks Control	MRL (mg/kg)
Aldrin	1.10a	0.51ab	0.10b	0.12b	0.01
Diedrin	0.97a	0.49ab	0.27b	0.28b	0.01
Endrin	0.97a	0.45ab	0.08b	0.13b	0.01
Heptachlor	1.00a	0.51ab	0.01b	0.07b	0.03
Heptachlor Oxide	1.20a	0.54ab	0.00b	0.03b	0.03
Endosulfan	0.63a	0.48a	0.00b	0.01b	0.05
Endosulfan II	0.84a	0.40ab	0.02b	0.01b	0.01
P'DDT	2.17a	0.70b	0.06b	0.17b	0.006
P'DDE	0.98a	0.36b	0.10b	0.15b	0.2
P'DDD	1.32a	0.40b	0.09b	0.10b	0.2

Note: Value with the same alphabet along the row are not significant different.
MRL represents the Maximum Residue Limit (mg/kg).
WQB represents the Water Quality Benchmark (mg/l)
The MRL values were adopted from the Codex Alimentarius Commission (CAC/FAO/WHO, 2023) and the European Commission Regulation (EC)

Interaction between Sample Type and Sampling Location

The interaction between sample type and sampling location significantly influenced the distribution of OCP residues ($P \leq 0.05$), indicating that contamination patterns differed according to both the environmental matrix and sampling site. For example, Aldrin concentrations in fish ranged from 0.10 to 0.52 mg/kg across the locations, while water concentrations ranged from 0.10 to 1.68 mg/l, with the highest concentration occurring at Ogbomoso Waterworks. Similar matrix-dependent patterns were observed for Dieldrin, Endrin, Heptachlor and Heptachlor oxide, with water generally recording higher concentrations at the impacted locations.

The pronounced difference between Ogbomoso Waterworks and the control locations suggests stronger pesticide influence at Ogbomoso Waterworks. This agrees with Olanrewaju *et al.* (2025), who reported OCP residues in fish from Dandaru River and Eleyele Lake in Ibadan and observed variation in residue occurrence among aquatic locations. Oladimeji *et al.* (2026) likewise reported spatial variation in OCP concentrations in fish collected from different locations along the Osun River, demonstrating that contamination levels can vary considerably within the same aquatic system.

The interaction pattern was particularly evident for the DDT-related compounds. p,p′-DDT reached 3.60 mg/l in Ogbomoso Waterworks water, while p,p′-DDE and p,p′-DDD also showed higher concentrations at Ogbomoso Waterworks than at the other locations. This predominance of DDT-related residues is comparable with the findings of Benson *et al.* (2023) in Lagos Lagoon, where DDT-related compounds were detected in water and sediments. The higher residues observed at

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Ogbomoso Waterworks may therefore reflect differences in local pesticide inputs, environmental persistence and transport.

The findings demonstrate that assessment of OCP contamination using only one sample or sampling location may underestimate the contamination status of an aquatic system. The combined assessment of water and fish therefore provides a more informative picture of environmental exposure, as also demonstrated in recent Nigerian aquatic studies (Benson *et al.*, 2023; Olanrewaju *et al.*, 2025; Oladimeji *et al.*, 2026).

Tab.3.: Comparative Analysis of Organochlorine Residues among the Sample Types, Sampling locations and Permissible MRL Standards (mg/kg)

Pesticide Detected	Ogbomoso Waterwork	Papa Abede Waterwork	Ogbomoso Waterwork Control	Abede Waterwork Control	Fish MRL (mg/kg)	Water Quality Benchmark (mg/l)
Aldrin	1.10a	0.51ab	0.10b	0.12b	0.10	0.00003
Diedrin	0.97a	0.49ab	0.27b	0.28b	0.10	0.00003
Endrin	0.97a	0.45ab	0.08b	0.13b	0.01	0.00002
Heptachlor	1.00a	0.51ab	0.01b	0.07b	0.01	0.00003
Heptachlor Oxide	1.20a	0.54ab	0.00b	0.03b	0.01	0.00003
Endosulfan	0.63a	0.48a	0.00b	0.01b	0.05	0.01
Endosulfan II	0.84a	0.40ab	0.02b	0.01b	0.05	0.01
P'DDT	2.17a	0.70b	0.06b	0.17b	0.05	0.001
P'DDE	0.98a	0.36b	0.10b	0.15b	0.05	0.001
P'DDD	1.32a	0.40b	0.09b	0.10b	0.05	0.001

Note: Value with the same alphabet along the row are not significant different.

MRL represents the Maximum Residue Limit (mg/kg).

WQB represents the Water Quality Benchmark (mg/l)

The MRL values were adopted from the Codex Alimentarius Commission (CAC/FAO/WHO, 2023) and the European Commission Regulation (EC)

Conclusion

The study established the occurrence of multiple organochlorine pesticide (OCP) residues in water and *Oreochromis niloticus* from Ogbomoso Waterworks and Papa Abede Waterworks Dams in Ogbomoso, southwestern Nigeria. Ten OCP residues were detected, indicating contamination by a mixture of persistent pesticide compounds and DDT-related residues.

Fish and water concentrations did not differ significantly ($P \leq 0.05$), although water generally recorded higher concentrations. OCP contamination varied spatially, with Ogbomoso Waterworks consistently showing the highest concentrations and the control locations generally recording lower levels. The significant sample type × location interaction further demonstrated that residue distribution depended on both the sampling matrix and location.

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Several residues exceeded the applicable food-residue limits in fish and water-quality benchmarks, indicating food-residue non-compliance and environmental contamination at affected sites. However, these findings do not constitute a quantitative human-health risk assessment, which requires site-specific exposure and consumption data. The results contribute to growing evidence of OCP contamination in Nigerian freshwater ecosystems and edible fish (Benson *et al.,* 2023; Olanrewaju *et al.,* 2025; Oladimeji *et al.,* 2026).

Regular monitoring of water, sediment and edible fish is recommended, particularly at sites with elevated residues. Further studies should include seasonal sampling, sediment assessment, source-apportionment investigations and quantitative dietary risk assessment based on local fish-consumption patterns. Improved pesticide stewardship, integrated pest management and vegetated buffer zones around aquatic systems should also be promoted to reduce pesticide entry and protect aquatic ecosystems and food safety.

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